



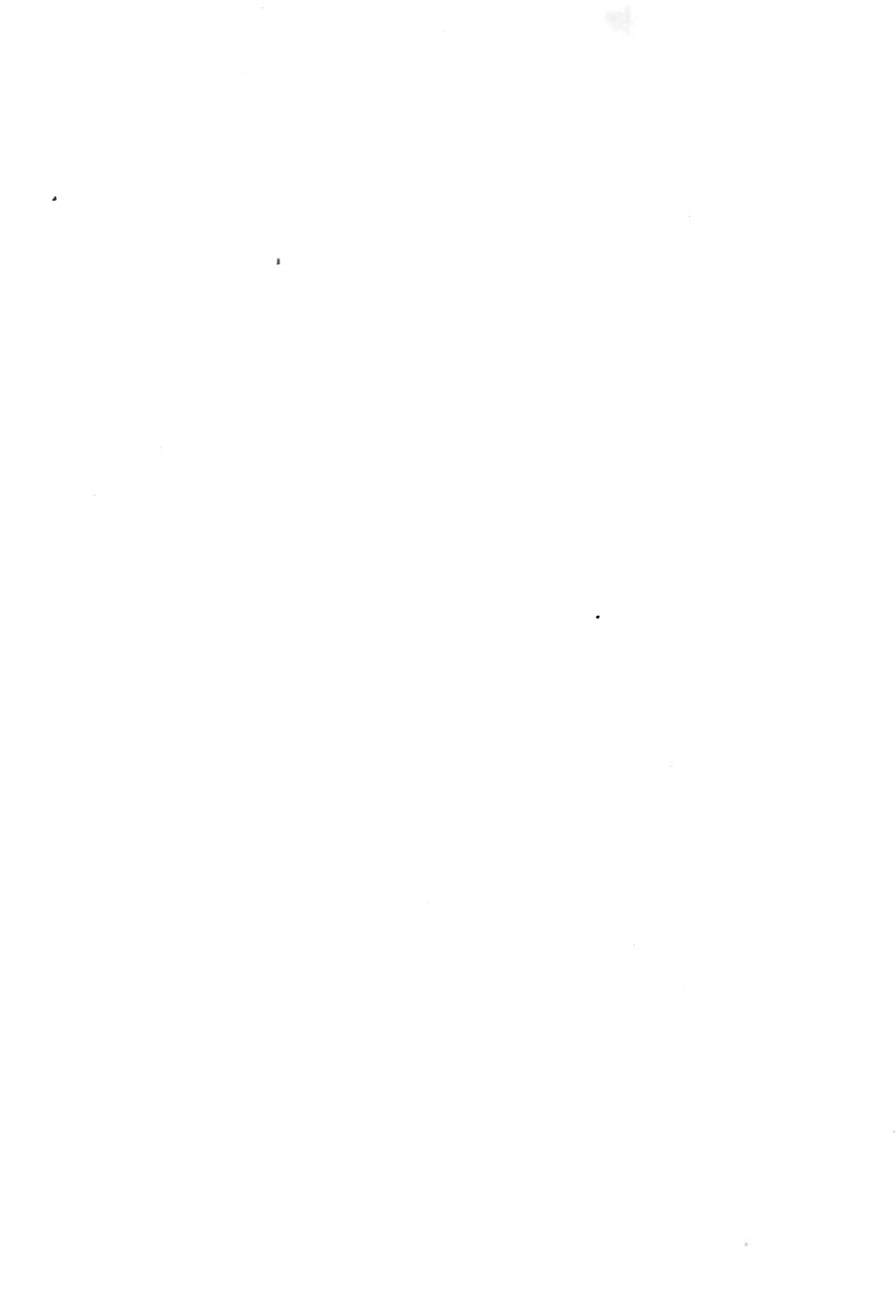


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The Secretary is instructed to negotiate for the purchase of copies of the Society's Journal for January 1883, and January, February, and April 1886. Members possessing odd copies of these numbers are particularly requested to communicate at once, stating price required, with Mr. Cresswell. The stock of all other numbers is at present sufficient for the Council's requirements.

#### LIST OF MEMBERS ELECTED, 23rd JANUARY, 1890.

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Barrett, Arthur A., 46, Mount Pleasant, Liverpool.

Bayay, Aug. de, Victoria Brewery, Melbourne, Australia, brewer.

Bott, Dr. Wm., Raffles Institution, Singapore, science lecturer.

Brindley, G. F., Silverdale, Douglas Road, Handsworth, Birmingham, chemical engineer.

Brown, Edw., Russian Steam Oil Mills, 22, Kourlandsky Street, St. Petersburg, analytical chemist.

Brown, R. J., M.Sc., 3, Oakfield Terrace, Davenport, near Stockport, chemical teacher.

Carr, Isaac, Gas Works, Widnes, engineer and manager.

Chapman, Alf. C., 23, Easton Buildings, Gower Street Station, N.W., analytical chemist.

Church, Elihu D., jun., 124, Milton Street, Brooklyn, N.Y., U.S.A., mining engineer.

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Grevel, Hermann, 33, King Street, Covent Garden, W.C., publisher.

Hagen-Schow, J. A., 4, Stanley Villas, Rutland Road, Hammersmith, W., brewing chemist.

Lacey, E. C., 126A, Bermondsey Street, London, S.E., manufacturing chemist.

McKillop, Jno., Puloh Brani, Singapore, metallurgist.

Mellon, E. L., Glenside, Clayton Bridge, near Manchester, chemist.

Merek, E., Darmstadt, Germany, manufacturing chemist.

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Morgan, Jno. Jas., 6, Tredegar Road, Ebbw Vale, Mon., chemist and assayer.

Peers, T. H. P., 184, Old Kent Road, London, S.E., druggist.

Pomroy, Dr. Chas. T., 266, Halsey Street, Newark, N.J., U.S.A., ink manufacturer.

Rosell, Claude A. O., United States Patent Office, Washington, D.C., U.S.A., chemist.

Rothband, W. S., 61, Elizabeth Street, Cheetham, Manchester, chemical manufacturer.

Sewell, Parker, 11, Alice Street, South Shields, analytical chemist.

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Smith, Harry E., 133, 26th Street, Milwaukee, Wis., U.S.A., assistant chemist.

Sowerby, Thos. H., Sherwell, Dartmouth Place, Blackheath, S.E., soap manufacturer.

Stevenson, J. Shannan, Jarrow Chemical Works, South Shields, chemical manufacturer.

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Watson, Eric E., 13, Kingsmead Road, Oxtou, Birkenhead, chemical student.

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Wilson, W. W., Chirnside Villa, Droylsden, Manchester, chemical assistant.

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Clift, Jno., 1/o Kemp Field House; Tar Works, Knottingley, Yorks.

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Doidge, H., 1/o Alberta Terrace; 25, Beech Avenue, Sherwood Rise, Nottingham.

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Gracey, Robt., 1/o Liverpool; 2, Bystock Terrace, Exeter.

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Hill, J. Kay, 1/o Waterside; Fullarton Cottage, Irvine, N.B.

Holt, G. Crompton, 1/o Liverpool; Green Bank, near Kidsgrove, Stoke-on-Trent.

Hooton, E., 1/o Burton Joyce; Fairfield, Hamilton Drive, Nottingham.

Ikuta, Dr. M., Journals to Tokyo, Japan.

Jackson, Jno., 1/o 260; 98, Dobbie's Loan, Glasgow.

James, J. H., Journals to J. H. Cordner-James, 162, New Bond Street, W., until February 15; then Johannesburg, South African Republic.

Johnson, A. E., Journals to 1, Eagle Villas, Penn Fields, Wolverhampton.

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Lewis, G. T., 1/o South Front Street; 4th and Walnut Streets, Philadelphia, Pa., U.S.A.

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 McKeehnie, D. M., 1/o St. Helens; The Hollies, Eccleston Park, Prescott.

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Neil, W., 1/o 126; 165, Turner's Road, Burdett Road, E. Owen, Thos., 1/o Cotham Park; Westbury-on-Trym, Bristol.

Parkinson, Dr. R., 1/o Bradford; Yewbarrow House, Grange-over-Sands, Lancashire.

Paterson, Alex., 1/o Cape Town; King William's Town, Cape Colony.

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Smith, J. W., 1/o Syracuse; Massachusetts Institute of Technology, Boston, Mass., U.S.A.

Smith, Miles H., 1/o Wandsworth; 3, Great Tichfield Street, London, W.C.

Sykes, Dr. B. C., 1/o St. John's House; Brook House, Cleckheaton, Yorks.

Tatlock, Jno., 1/o Sauchiehall Street; 40, Renfrew Street, Glasgow.

Taylor, R. H., 1/o Marjorie Street; 2, Ross Walk, Leicester.

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Thomson, G. Carruthers, 1/o Glasgow; 39, Rolland Street, Dunfermline, N.B.

Tyzaek, S., 1/o Montgomery Road; Beckford Lodge, Williamson Road, Sheffield.

Waterfall, W. B., 1/o Redland Grove; Thirlmere, Clavering Road, Redland, Bristol.

Weldon, E., 1/o Sandon Street; 64, Noel Street, Nottingham.

Welsh, W., 1/o Ashton-on-Mersey; Holt Town, Manchester.

White, S. J., 1/o Philadelphia; Welsbach Incandescent Gas Light Co., Gloucester City, N.J., U.S.A.

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Wood, Milton R., 1/o Ash Street; 33, Oakland Avenue, Brooklyn, N.Y., U.S.A.

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Green, Herbert, 1/o Tovil House, Maidstone.

Manlove, E. A., 1/o Holtham Road, London, N.W.

#### Deaths.

Austin, E., 126A, Bermondsey Street, S.E.

Bocklisch, J. E., Rohrite Explosive Co., Gathurst, Wigan.

Grafton, F. W., M.P., 91, Portland Street, Manchester.

Jekyll, H. H., 7, Carholme Road, Lincoln.

## London Section.

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#### SESSION 1889-90.

1890.

Feb. 3rd:—

Messrs A. H. Allen and W. W. Staveley. "On the Properties and Applications of Metallic Compounds of the Phenols."

Mr. Watson Smith. "A New Method of Hardening Steel for Cannon and Projectiles."

Prof. S. M. Losanitsch. "On Bromanil."

March 3rd:—

Dr. H. Schlichter. "Analysis of Textile Fibres and Fabrics."

Dr. L. T. Thorne. "A Review of the Methods of producing Oxygen commercially."

March 31st (instead of April 6th—Bank Holiday):—

Triennial Election of Committee and Officers of Section. Nominations to be sent to the Honorary Secretary on or before Monday, March 17th.

Dr. W. S. Squire. "Note on a Machine for a Methodical Separation of Solids and Liquids."

Dr. S. Rideal. "Note on the present Monetary Value of Available Oxygen."

May 5th:—Mr. Wm. Webster. "The Electrical Treatment of Sewage."

June 2nd:—Dr. W. S. Squire. "The Artificial Production of Cold theoretically and practically considered."

July:—Annual General Meeting in Nottingham.

Meeting held Monday, 6th January, 1890.

MR. DAVID HOWARD IN THE CHAIR.

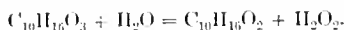
### PEROXIDE OF HYDROGEN: ITS PRESERVATION AND COMMERCIAL USES.

BY C. T. KINGZETT, F.I.C., F.C.S.

PEROXIDE of hydrogen is at once an extraordinary and a most interesting compound, extraordinary as regards the modes of its preparation and interesting on account of the facility with which it loses oxygen. It is this facility of decomposition which renders it so valuable as an oxidising and bleaching agent, but at the same time has in the past militated against its more extensive adoption for such purposes. For a long time it has been recognised that if means could be devised of preserving this substance from spontaneous decomposition to any considerable extent, its market value would enormously increase, for while it is without rivals as an antiseptic oxidising agent, it is also of a perfectly innocuous character and free from all objections. Peroxide of hydrogen has been with me the subject of much study, and as is well known, I have for many years been engaged in the manufacture of compounds containing it or capable of yielding it in association with other substances under definite circumstances.

In connection with this part of my subject it may be mentioned by way of a summary statement that, when terpenes are subjected to oxidation by air, oxygen, or ozone, there is formed an active organic compound of the nature of a peroxide which remains dissolved in the hydrocarbons used for producing it, and when the product is placed in

contact with water, peroxide of hydrogen is formed and passes into aqueous solution by some reaction of which the following equation may be accepted as giving, if not the actual reaction, at least an approximately correct representation:—



The compound  $C_{10}H_{16}O_2$  (soluble camphor) is uncrystallisable and accompanies the peroxide of hydrogen in the aqueous solution.

There is a great similarity between this chemical change and that by which the peroxide of hydrogen is made by the other well-known process, viz., by the action of dilute acids upon peroxide of barium. Taking sulphuric acid as representative of all acids, it may be said that when peroxide of barium is exposed to the presence of sulphuric acid and water, the following change results:—



It will be understood of course that I have here resorted to the use of somewhat abnormal formulæ in order to clearly represent what I believe to be the nature of the chemical change, and I have intentionally expressed peroxide of hydrogen by the formula  $H_2O$  for reasons to which I am about to make reference.

The constitution of peroxide of hydrogen is generally graphically represented as follows:—

Fig. 1.



which would signify that each atom of oxygen is diatomic, and is directly combined with one atom of hydrogen. But the character of the substance and its facility of decomposition are altogether opposed to such a view, and in a short paper which was communicated to the British Association some years ago\* I gave expression to my opinion that in a strictly chemical sense it is rather to be regarded as oxygenated water or as an oxide of water. That is to say, oxygen is to be regarded as either a triad or a tetrad in valency, and the one atom is combined with the other which alone is directly combined with both atoms of hydrogen, thus:—

Fig. 2.

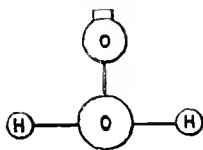
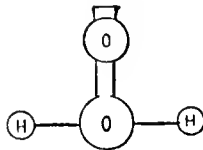
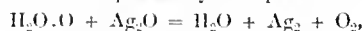


Fig. 3.



This view accords with the general sense of chemical opinion that when in a compound an atom of one substance is directly combined with another such atom, the combination is weak, and there is consequently facility of decomposition. This view is also supported by certain observations which were made by M. Berthelot, showing that when oxide of silver is placed in contact with peroxide of hydrogen, the amount of oxygen set free is only equal to that contained in the peroxide of hydrogen employed, the residual matter containing the whole of the silver and oxygen originally present in the oxide of silver, but redistributed, so that there results a mechanical mixture containing one-third of the silver in the reduced metallic state, and the oxygen, in combination with the balance, in the form of sesquioxide of silver. To put the matter in another way, the chemical change is not to be expressed by the equation—



but rather by this one—



Further confirmation is lent to my views by the very existence of ozone which may be viewed as a form of triatomic oxygen.

To pass on now to the chief part of my subject, it should be first stated that, while peroxide of hydrogen is undoubtedly very prone to suffer decomposition, the statement which is made in most text-books to the effect that dilute solutions are immediately decomposed on ebullition, is radically wrong. It is, therefore, all the more to be regretted that this method has been advocated for estimating the strength of the liquid. As a matter of fact, dilute solutions may be concentrated by evaporation, although, of course, there is an attendant loss of oxygen.

Mr. G. E. Davis at one time† published some figures illustrating the rate of decomposition of a solution of peroxide of hydrogen, but before reproducing these it may be as well to explain the meaning of a description, which is common enough in commerce; I refer to the strength of such solutions as expressed in volumes. A 10-volume solution of peroxide means a solution which is capable of yielding of itself, by perfect decomposition, 10 times its own volume of gaseous oxygen at the ordinary temperature and pressure.

Now, Mr. Davis worked with a 10-volume solution, so that 100 cc. yielded 1,000 cc. of oxygen when fully decomposed; in other words, the solution contained 3.04 per cent. by weight of real peroxide of hydrogen, and was capable of yielding 1.43 per cent. by weight of oxygen gas.

On August 2, 1878, the strength of the solution was 9 volumes.

"	"	31	"	"	"	"	8.4	"
"	"	24	"	"	"	"	8.0	"
"	Sept.	1	"	"	"	"	7.9	"
"	"	24	"	"	"	"	7.8	"
"	Oct.	24	"	"	"	"	7.2	"

I shall presently furnish other figures from my own experience, but may at once state that the rate of decomposition is by no means constant. It varies with the temperature, exposure to light, and the impurity of the solution. Sometimes a solution will be found to suffer even less change than that observed by Mr. Davis, while at others it undergoes complete decomposition in course of a few weeks, and more or less suddenly. The observations I have made will serve to explain to some extent the reasons for this capricious character.

To those engaged in the manufacture of peroxide of hydrogen it is well known that the presence of a little ether restrains the decomposition, and, indeed, in his experiment, Mr. Davis found that by adding a little ether to the solution on October 24th it had not deteriorated in strength two months later in the year. The action of the ether is not, however, always to be depended upon, and its presence is apt to lead to danger, particularly if added to solutions which are likely to be exported to hot climates, while it is sometimes objectionable on other grounds also. I have suggested that the restraining influence of ether is to be found in the fact that it also generates peroxide of hydrogen by atmospheric oxidation. It may also act by reason of its volatility, thus generating a vapour which exercises a gentle restraining pressure over the solution.

Dr. G. Gore has shown, in a paper recently communicated by him to the Royal Society, that when chlorine water is exposed to light, the chemical changes that occur in one distinct period result in the production of hydrochloric, hypochlorous, and chloric acids, and that at a later stage a further formation of hydrochloric acid takes place, while at the same time peroxide of hydrogen is formed in solution. Under the influence of prolonged sunlight, the whole of the oxygen of the hypochlorous and chloric acids united with water to form the peroxide, and that substance subsequently entered into combination with the whole of the hydrochloric acid and formed a definite "solution compound" which is represented by the formula  $2 HCl, H_2O_2$ . This is the more interesting because it is well known that, as a bleaching agent, chlorine acts intermediately by the decomposition of

\* See Chem. News, Vol. XLVI., 141.

† Chem. News, Vol. XXXIX., p. 221.

water, and it is nascent oxygen that is credited by chemists with the bleaching effects thus attainable. It is, after all, not impossible that the effects are due to the formation of peroxide of hydrogen, a substance which is, as a bleaching agent for woollen and some other goods, rapidly displacing bleaching powder in public estimation.

I now propose to call attention to the decomposition and preservation of peroxide of hydrogen as influenced by the presence in it of other substances in a state of solution.

I may premise that I was led to this investigation by my desire to put upon the market a series of disinfectant solutions for use by surgeons and others which should be characterised, in common with "Sanitas" disinfecting fluid, on the one hand by the presence of a definite quantity of peroxide of hydrogen, and on the other hand by containing a definite amount of some one or other of a series of reliable antiseptic and germicidal substances. Some medical men prefer one antiseptic, and others exercise other selections, but I insist upon the necessity of using any such germicides, in association with a potent oxidising agent capable of chemically destroying the toxic products which result from the growth of bacteria and other micro-organisms in suitable media. It is these toxic products that are more to be dreaded than the micro-organisms themselves, and no system of disinfection is perfect, from a chemical point of view, which overlooks the necessity for their chemical change or destruction.

After surmounting many difficulties, into the details of which I need not enter here, I was faced with what proved to be the greatest obstacle of all, viz., the liability of such solutions when prepared, to undergo decomposition, and that sometimes with almost explosive violence.

As I felt there was no royal road to success, and no scientific clue to a means of overcoming the trouble, I determined to study the matter in a constructive sense, and to ascertain, over long periods of time, the preservative or destructive influences which might be exercised by dissolving, in solutions of peroxide of hydrogen, definite percentages of various substances, and the results which have been tabulated for convenience this evening represent some of the results.

TABLE I.

Name and Amount of Substance added.	Original Strength of the $H_2O_2$ Solution in Terms of cc. Hypo. per 100 cc. on Nov. 30th, 1887.	After 20 Days.	After 62 Days.	After 176 Days.	Loss.
5 Per Cent. Standard ...		730	706	586	Per Cent. 20
$K_2SO_4$ .....		710	690	562	23
$Na_2SO_4$ .....		720	706	592	19
$(NH_4)_2SO_4$ ..		720	704	586	20
$MgSO_4$ .....		726	508	146	80
$FeSO_4$ .....		46	42	26	96
$Al_2(SO_4)_3$ ...		688	592	274	62
$HgSO_4$ .....		660	442	94	87
$CuSO_4$ .....	730 cc.	158	74	52	93
$ZnSO_4$ .....		704	576	292	60
$MnSO_4$ .....		574	338	54	92
$KNO_3$ .....		730	706	618	15
$NaNO_3$ .....		730	718	616	15
$NH_4NO_3$ ....		730	708	594	19
$Ba(NO_3)_2$ ...		724	712	610	16
$Sr(NO_3)_2$ ...		736	724	630	14
$Ca(NO_3)_2$ ...		724	716	600	18

TABLE I.—continued.

Name and Amount of Substance added.	Original Strength of the $H_2O_2$ Solution in Terms of cc. Hypo. per 100 cc. on Nov. 30th, 1887.	After 20 Days.	After 62 Days.	After 176 Days.	Loss.
$Mg(NO_3)_2$ ..		734	716	608	Per Cent. 17
$Fe(NO_3)_2$ ...		52	36	20	97
$Cu(NO_3)_2$ ...		226	82	54	93
$Zn(NO_3)_2$ ...		726	60	38	95
$Pb(NO_3)_2$ ...		714	570	254	65
$Hg(NO_3)_2$ ..		726	670	46	94
KCl .....		710	702	606	17
NaCl .....		722	668	392	46
$NH_4Cl$ .....		724	712	618	16
$BaCl_2$ .....		712	720	630	14
$SrCl_2$ .....	730 cc.	724	714	610	16
$CaCl_2$ .....		710	724	620	15
$MgCl_2$ .....		722	710	620	15
$Al_2Cl_6$ .....		524	228	44	94
$Fe_2Cl_6$ .....		68	54	52	93
$CuCl_2$ .....		82	74	76	90
$PbCl_2$ .....		602	484	84	88
$SuCl_2$ .....		648	642	562	23
$ZnCl_2$ .....		720	718	544	25
$HgCl_2$ .....		724	712	606	17

In this series of observations, 50 cc. of peroxide of hydrogen solution was used in each experiment, the solution being of such strength that 100 cc. was capable of liberating from an acidified solution of potassic iodide such an amount of iodine as required 730 cc.  $\frac{N}{10}$  hypo. solution ( $Na_2S_2O_3$ ) to decolourise it.

0.5 gram. of the several substances indicated in the first column was added to as many quantities of the peroxide. The mixtures were placed in well-stoppered bottles freely exposed to the light of the laboratory, and the strength of the solutions, in terms of cc.  $\frac{N}{10}$  hypo. was determined as above explained from time to time.

The experiments were started on 30th November 1887.

From a study of the results it will be seen that none of the substances which were tested materially promoted the stability of the peroxide, although several nitrates and chlorides exercised a limited restraining influence. It will be further seen that very many of the added substances materially facilitated the decomposition of the peroxide.

In the somewhat disjointed series of observations next to be described and the results of which are expressed in Table II., the substances indicated in the first and second columns were added to so many solutions of 100 cc. each, except in the case of chloroform, in which 50 cc. peroxide was employed together with the amount of absolute alcohol as indicated for the purpose of dissolving the added chloroform.

The original testings of the solution are expressed in the table. The solutions containing the added substances were placed in stoppered bottles and gently shaken from time to time.

It will be seen that one standard solution did not suffer any decomposition in course of 31 days, while in another instance a standard solution suffered change to the extent of nearly 16 per cent. in 29 days; a third standard solution

lost 74 per cent. in 22 days and the whole of its  $\text{H}_2\text{O}_2$  within 84 days; in the fourth series of observations the standard solution lost 95 per cent. in 36 days. The rule was that the greater the warmth of the weather the greater was the loss in a given time.

It will also be observed that alcohol, glycerin, chloroform, absolute phenol, ether, acetic acid, and potassic bisulphate exercised a restraining influence.

The next series of observations which are tabulated in Table III. were based upon the results of the preceding experiments, and were at once more systematic and much more extended in point of time, lasting over 405 days so far as the results now communicated are concerned, while some of the solutions are even yet in course of observation.

One per cent. by weight in volume of the added substances was employed, and the observations were commenced on August 28th, 1888. Originally 100 cc. of the  $\text{H}_2\text{O}_2$  which was employed, required 1,296 cc.  $\text{N}_{10}$  hypo. solution.

The testings were made by taking out 5 cc. of the solutions, making up to 50 cc. with added water and using 5 cc. of the diluted solution for the purpose of decomposing acidified KI solution, after which the iodine set free was determined in the usual manner.

The phenol, beta-naphthol, and thymol mixtures all became discoloured (brown), the last-named one being least discoloured.

The original solution of peroxide was exactly neutral, but gave a precipitate with solution of barium chloride.

The following substances were imperfectly dissolved:  $\beta$ -naphthol, thymol, and salutar.

The results speak for themselves, and it is only necessary to point out the very distinctive value of the following substances in restraining the decomposition of the peroxide, viz., alcohol and ether.

Many other of the added substances were more or less effectual, in common with which it is seen that sulphuric acid was of some value.

On April 14th, 1889, a fresh series of observations were made, using a solution of peroxide of hydrogen which was slightly acid to test paper.

With sulphuric acid it gave a slight precipitate of sulphate of barium.

With barium chloride it did not yield a precipitate. Ammonia caused in it a flocculent precipitate which was to a large extent soluble in ammonium chloride. With hydric-disodic phosphate it gave a slight precipitate. By way of strength 100 cc. liberated such an amount of iodine from acidified potassic iodide as required 1,540 cc.  $\text{N}_{10}$  hyposulphite of sodium for complete reaction.

Quantities of 100 cc. each were taken, and to them was severally added 1 gm. of the substances indicated in Table IV.

Again, it will be observed that alcohol and ether each materially preserved the solutions of peroxide of hydrogen from decomposition. So also did camphoric acid, camphor and menthol, while thymol, the influence of which is very comparable with that exhibited in the previous series of experiments, comes out better in comparison with all these substances.

The observations which are still in hand have not extended however over such a protracted period as those which are expressed in Table III.

Viewing the whole results of the third and fourth series of observations it is perhaps to be inferred that the comparative success attained in the third series was due to the employment of an *exactly neutral* solution of peroxide, for although sulphuric acid exercises a restraining influence on its own account, it facilitates decomposition in comparison with the repressive action of ether, alcohol, thymol, camphoric acid, camphor and other substances.

It was upon the basis of these and other results of a like character, and having regard to the mode of preparation of the crude article, that I have been enabled to devise satisfactory means for preserving peroxide of hydrogen as presented in commerce from any appreciable loss of strength over reasonable periods of time.

It remains for me to state that these means have been patented, and none of the substances which I have indicated

as being useful for repressing the natural decomposition of peroxide or for preserving it from such change, can be employed by others than the Sanitas Company, Limited (to whom I have assigned my rights), or their licensees.\*

In those cases in which it has been ascertained that certain substances facilitated the decomposition of peroxide of hydrogen there is a great range of activity, and in the operations of bleaching these facts admit of practical utilisation. As a rule, some alkaline compound such as ammonia or carbonate of sodium is used to promote the operations, but they act too violently and cause much loss of oxygen, thus adding to the expense of the process. For many such purposes therefore, these alkaline compounds may in future be well replaced by some of the compounds referred to in my tables, and of which borax affords a striking example.

Apart from the previously well-known oxidising properties of peroxide of hydrogen, it was proved for the first time by some experiments, of which an account was presented by me to the British Association in 1876, that this substance exhibits striking antiseptic effects and is capable, even when present in very small proportions, of entirely arresting processes of fermentation which are originated by living organisms. The antiseptic properties of the substance were subsequently confirmed by the observation of Guttman and Fränkel in Germany. Later still, some similar observations were made by M. Baldy, who concluded that peroxide of hydrogen is in no respect inferior to carbolic acid.

M. Paul Bert and M. Regnard confirmed my own earlier experiments, they showing (Comp. Rend. 94, 1383—1386) that an extremely dilute solution of peroxide of hydrogen prevents the fermentation of glucose by the agency of yeast, prevents mycdermic growth in red wines, lactic fermentation in milk, and the putrefaction of albumen, &c. On the other hand they found that it was incapable of arresting the changes which are induced by soluble non-organised ferments, such as diastase. This distinction is of importance, because if peroxide of hydrogen be employed for internal administration, it affords a guarantee that the presence of that substance in the human system will not arrest nor interfere with the many assimilative and other processes which are carried on by means of ferments of the pancreatic type.

It was therefore to be expected that this compound would be found of peculiar benefit for the treatment of wounds and for general use in the practice of antiseptic surgery, and that anticipation has been amply realised in the practice of M. Baldy, M. Regnard, and particularly that of M. Péan in the hospital of St. Louis. It has been demonstrated beyond question that all wounds which are treated with peroxide of hydrogen progress well, healing by first intention, all discharges being healthy in character and free from odour. Chronic ulcers are similarly treated with the same successful results, so also cases of gangrene, fetid suppurations, and bad ozonic discharges.

M. Péan performed many important operations in an atmosphere impregnated with peroxide of hydrogen instead of carbolic acid, and M. Baldy employed gauze and wool which had been soaked in peroxide, for placing in contact with wounds by means of bandages where prolonged contact was desirable. He also found that it might, with safety, be used as an injection for washing out cavities.

Dr. A. W. Harlan, in a paper read before the American Dental Society (Dental Record, November 1882), strongly recommended the use of peroxide in the treatment of blind alveolar abscesses and pyorrhea alveolaris, because the rapid evolution of oxygen gas effects a thorough evacuation of the pus without creating any undesirable after effects.

M. Landolt, of Paris, and Dr. J. E. Adams, of the London Hospital and Royal London Ophthalmic Hospital, have both used peroxide of hydrogen with great success in cases of purulent discharge from the conjunctiva (see British Medical Journal, December 9th, 1882).

Since October 1885, Professor Hofmooke has employed peroxide with the greatest advantage in the treatment of diphtheria, while for syphilitic and cancerous swellings and wounds generally, it is now in everyday use by many of the most eminent surgeons at home and abroad.

\* See this Journal, 1889, p. 1,000; and 1890, p. 101.

In a recent paper (read before the King's County Medical Association, February 5th, 1889, during a discussion on diphtheria: see "Gaillard's Medical Journal" for March 1889) Dr. E. R. Squibb, of Brooklyn, says:—"If diphtheria be at first a local disease and be auto-infections—that is, if it be propagated to the general organism by a contagious virus located about the tonsils, and if this virus be, as it really is, an albuminoid substance, it may and will be destroyed by this agent upon a sufficient and sufficiently repeated contact."

Peroxide of hydrogen never fails to thoroughly cleanse all mucous surfaces. It not merely exhibits the power of destroying pus and other such products by oxidation, but it is admitted by the most reliable bacteriologists to be an exceedingly powerful germicide, ranking perhaps in this respect next to biniodide of mercury, while, as distinct from that substance, it is quite non-poisonous.

As all air-breathing animals depend upon atmospheric oxygen for their very life, it stands to reason that for all diseases that are in any way dependent upon defective oxidation, peroxide of hydrogen is the best natural remedy, being a solution of oxygen in chemical combination with the elements of water.

Valuable as is peroxide of hydrogen alone, it becomes still more valuable in its properties when associated with other reliable antiseptics and germicides as presented in the series of bactericides to which I have made general reference in an earlier part of this paper.

With respect to the uses of peroxide of hydrogen for bleaching, a summary statement will suffice. It has been used and is being used with success for bleaching wool and woollen goods, silk (particularly tussah), ostrich feathers, living and dead human hair, sponges, ivory, bone, wash leather, horn, gut, wooden articles, and so forth. It may also be employed for bleaching cotton, fine yarns, and textiles, but not so successfully as for woollen goods. For such bleaching purposes tanks or vessels of wood, glass, or earthenware are employed. Metallic vessels have to be avoided. The general method of procedure consists in freeing the articles from dirt and grease by washing them with ammonia water or with dilute soda or carbonate of soda, and after having thoroughly rinsed them out they are immersed in the solution of peroxide of hydrogen, sometimes used of 10-vol. strength and at other times of only 2-vol. strength; frequently ammonia or some other alkali is added to the solution of peroxide in order to facilitate the bleaching.

The operation of bleaching is generally carried out at a temperature of about 30°–50° C., heat being employed in the form of steam carried through a leaden coil immersed in the bath.

As I have previously pointed out, the addition of an alkali in order to hasten the effect is made at the expense of using an extra amount of peroxide, and in my opinion borax would replace it to advantage where it is necessary to employ any such agent for facilitating the operation.

TABLE II.

Name of Substance introduced.	Percentage of same.	Orig. Test-ings.	On 29th Day.	On 63rd Day.	Percentage of Loss.	—	—	—
Standard .....	..	870	732	674	22	..	..	..
Alcohol (proof spirit).	5 cc. = 2½ per cent. real alcohol.	822	716	756	8	..	..	..
Glycerin.....	2½ per cent.	856	730	746	13	..	..	..
Standard .....	..	738	686	..	7	..	..	..
Borax .....	5 per cent.	710	80	..	89	..	..	..
Hydrochloric acid	15 cc. solution (containing 33·8 per cent. real HCl).	650	156	..	76	..	..	..
Chloral .....	5 per cent.	718	626	..	13	..	..	..
Oxalic acid.....	5 "	714	278	..	61	..	..	..
Standard .....	..	724	On 31st Day. 732	..	0	..	..	..
Sulpho-phenic acid	5 per cent.	628	686	..	0	..	..	..
Boric acid .....	5 "	712	572	..	20	..	..	..
Standard .....	..	794	On 22nd Day. 206	On 53rd Day. 20	On 84th Day. 0	On 182nd Day. ..	Percentage of Loss. 100	..
Sodium benzoate..	5 per cent.	614	34	Discontinued.	..	..	100	..
Chloroform and alcohol.	5 cc. chloroform and 50cc. abs. alcohol.	742	742	734	694	660	11	..
Borax neutralised with boric acid..	5 per cent.	724	32	Discontinued.	..	..	100	..
Absolute phenol ..	5 "	816	752	630	558	480	41	..
Standard .....	..	786	On 36th Day. 22	Percentage of Loss. 27	On 67th Day. 0	Percentage of Loss. 100	On 116th Day. 0	On 165th Day. ..
Potassic chlorate..	5 per cent.	846	66	22	50	24	0	..
Ether.....	5 cc.	798	688	14	666	16·5	700	610
Acetic acid (glacial)	5 cc.	794	746	6	698	12	660	620
Caustic potash....	5 per cent.	782	32	96	14	98·2	0	..
Potassic bisulphate	5 "	796	778	2	738	7·2	740	680

Started April 15, 1888.

Started April 17, 1888.

Started June 5, 1888.

Started June 22, 1888.



TABLE III.

Name of Substance added. (1 per Cent. in each Case.)	cc. Hypo. required per 100 cc. 28th Day = Loss.	Hypo. required on 4th Day = Loss.	Hypo. required on 28th Day = Loss.	Hypo. required on 202nd Day = Loss.
	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Standard A. No addition .....	1,160 .. 10	1,140 .. 12	940 .. 27.4	790 .. 39.0
Standard B. H <sub>2</sub> SO <sub>4</sub> on 16th day .....	1,180 .. 9	1,140 .. 12	1,000 .. 22.8	938 .. 27.6
Ethylie alcohol .....	1,280 .. 1	1,300 .. 0	1,200 .. 7.4	1,198 .. 7.5
„ ether, sp. gr. 720 .....	1,340 .. 0	1,520 .. 0	1,210 .. 4.3	1,254 .. 2.4
Methylated chloroform, sp. gr. 1.498 .....	1,210 .. 1	1,210 .. 4	1,120 .. 13.5	1,058 .. 18.3
Acetic acid (congeals at 56°) .....	1,300 .. 0	1,340 .. 0	1,180 .. 8.9	1,148 .. 11.4
Glycerin, sp. gr. 1.260 .....	1,260 .. 3	1,260 .. 3	1,140 .. 12.0	1,064 .. 17.9
Phenol, pure cryst. P.B. ....	1,180 .. 9	1,200 .. 7	1,050 .. 18.2	1,035 .. 20.1
Beta-naphthol, cryst. ....	1,140 .. 12	1,160 .. 10	980 .. 24.3	894 .. 31.0
Chlorol, P.B., pure cryst. ....	1,280 .. 1	1,340 .. 0	1,120 .. 13.5	1,030 .. 20.5
Thymol .....	1,220 .. 6	1,240 .. 4	1,120 .. 13.5	1,085 .. 16.2
Phenylacetic acid .....	1,300 .. 0	1,350 .. 0	1,180 .. 8.9	1,170 .. 9.7
Sulpho-phenic acid, cryst. ....	1,260 .. 3	1,260 .. 3	1,200 .. 7.4	1,170 .. 9.7
Boric acid .....	1,060 .. 18	980 .. 24	800 .. 38.2	590 .. 54.4
KHSO <sub>4</sub> .....	1,240 .. 4	1,260 .. 3	1,080 .. 16.6	1,120 .. 13.5
KCl .....	1,100 .. 15	1,060 .. 18	800 .. 38.2	690 .. 46.7
Na <sub>2</sub> SO <sub>4</sub> .....	1,140 .. 12	1,120 .. 13	900 .. 30.5	800 .. 38.2
NaNO <sub>3</sub> .....	1,340 .. 0	1,120 .. 13	880 .. 32.1	790 .. 39
NaC <sub>2</sub> H <sub>3</sub> O <sub>2</sub> .....	100 .. 92	60 .. 95	Discontinued.	
BaCl <sub>2</sub> .....	1,080 .. 16	1,040 .. 19	800 .. 38.2	710 .. 45.2
NaCl .....	1,160 .. 10	1,120 .. 13	880 .. 32.1	785 .. 39.4
HgCl <sub>2</sub> .....	1,200 .. 7	1,200 .. 7	900 .. 30.5	725 .. 44.0
Sulphur .....	1,220 .. 6	1,180 .. 9	920 .. 29.0	742 .. 42.7

TABLE III.—continued.

Name of Substance added. (1 Per Cent. in each Case.)	Hypo. required on 300th Day = Loss.		Hypo. required on 325th Day = Loss.		Hypo. required on 405th Day = Loss.		Hypo. required on 490th Day = Loss.*	
	Per Cent.		Per Cent.		Per Cent.		Per Cent.	
Standard A. No addition.....	520	.. 59.8	340	.. 73.7	220	.. 83.0	140	.. 89.2
Standard B. H <sub>2</sub> SO <sub>4</sub> on 16th day.....	800	.. 38.2	680	.. 47.5	513	.. 60.4	410	.. 68.3
Ethylie alcohol .....	1,260	.. 2.7	1,140	.. 12.0	1,020	.. 21.2	1,000	.. 22.8
„ ether, sp. gr. 720.....	1,320	.. 0*	1,200	.. 7.4	1,100	.. 15.1	1,000	.. 15.9
Methylated chloroform, sp. gr. 1.498 ....	940	.. 27.4	760	.. 41.3	568	.. 56.1	Discontinued.	
Acetic acid (congeals at 59°) .....	1,060	.. 18.2	900	.. 30.5	642	.. 50.4		
Glycerin, sp. gr. 1.260 .....	820	.. 36.7	580	.. 55.2	366	.. 71.7		
Phenol, pure cryst. P.B.....	920	.. 29.0	860	.. 33.6	715	.. 44.8		
Beta-naphthol, cryst.....	Discontinued.							
Chloral P.B., pure cryst.....	760	.. 41.3	520	.. 59.8	366	.. 71.7		
Thymol.....	900	.. 30.5	840	.. 35.1	696	.. 46.3		
Phenylacetic acid.....	980	.. 24.3	940	.. 27.4	770	.. 40.5		
Sulpho-phenic acid, cryst.....	960	.. 25.8	860	.. 33.6	752	.. 41.9		
Boric acid .....	Discontinued.							
KHSO <sub>4</sub> .....	800	.. 38.2	680	.. 47.5	532	.. 58.9		

\* The results given in the two last columns have been added since the paper was read.—Author.

TABLE IV.

Solution. (1 Per Cent. of added Substances.)	Testings after 43 Days, viz., on May 27, 1889.	Percentage of Loss.	Testings after 97 Days, viz., on July 20, 1889.	Percentage of Total Loss.	Testings after 176 Days, viz., on Oct. 7, 1889.	Percentage of Total Loss.	Testings after 270 Days, viz., on Jan. 9, 1890.*	Percentage of Total Loss.
H <sub>2</sub> O <sub>2</sub> only (white bottle).....	1,151	25	700	54	435	71.7	370	75.9
Large ditto (blue bottle) .....	1,377	10	890	42	610	60.4	530	65.5
Methylated ether, sp. gr. 720 .....	1,143	6	1,410	8	1,330	13.6	1,280	16.8
Chloroform, sp. gr. 1.498 .....	1,302	15	850	44	565	63.3	Discontinued.	
Ethylie alcohol, P.B.....	1,490	3	1,370	11	1,225	20.4	1,180	23.3
Glacial acetic acid .....	1,358	11	1,120	27	930	39.6		
Glycerin, sp. gr. 1.260 .....	1,424	7	1,020	33	595	61.3		
HNO <sub>3</sub> .....	1,292	16	740	51	415	73.0		
H <sub>2</sub> SO <sub>4</sub> .....	1,406	8	990	35	690	55.2	Discontinued.	
Sulpho-phenic acid.....	1,207	21	1,020	33	855	44.4		
Phenylacetic " .....	1,415	8	1,170	24	942	58.8		
Salicylic " .....	1,245	19	1,010	34	810	47.4		
Camphoric " .....	1,538	0	1,420	8	1,255	18.5	1,180	23.3
Thymol.....	1,538	0	1,460	5	1,355	12.0	1,300	15.5
Camphor .....	1,500	2	1,410	8	1,260	18.1	1,260	18.1
Chloral hyd. P.B. ....	1,330	13	850	44	475	69.1	Discontinued.	
Menthol .....	1,538	0	1,400	9	1,275	17.2	1,250	18.8
Nicotine.....	1,085	29	840	45	705	54.2		
NaHSO <sub>4</sub> .....	1,349	12	810	47	500	67.5		
KHSO <sub>4</sub> .....	1,330	13	830	46	540	64.9	Discontinued.	
Cresote.....	1,330	13	1,200	22	1,105	28.2		

\* The results in these two last columns have been added since the paper was read.—*Author*.

## DISCUSSION.

The CHAIRMAN said that the practical application of peroxide of hydrogen depended in very many cases on the possibility of retaining the solution at approximately the same strength for a considerable period. Every chemist knew, however, that under ordinary circumstances it was a most unstable body. The restraining action on the decomposition of hydrogen peroxide exhibited by the various substances mentioned by Mr. Kingzett was extremely curious and surprising. He would have expected that the more unstable the associated body the more rapid would be the decomposition of the peroxide of hydrogen; but this had proved to be by no means the case. The useful adaptation of oxygen depended, broadly speaking, on the careful study of the conditions of its activity, and everything which threw light upon that important subject was of the highest scientific and industrial value.

Professor CLOWES said that the paper had suggested one or two points of theoretical importance upon which he would be glad to have some further information. It was well understood that the valency attributed to the atom of a chemical element was disputable, and therefore he was not surprised to find suggestions for altering the valency of oxygen. But Mr. Kingzett had assumed, apparently, that it might be either triad or tetrad instead of dyad. Probably he had already assigned his reasons for that assumption, but if he could now give them again, members of the Section would probably be glad to hear them: he (Dr. Clowes) did not understand how the oxygen atom could be represented as triad in the molecule of ozone. In the table written on the blackboard, too, Mr. Kingzett indicated the change occurring between hydrogen peroxide and silver oxide, and introduced the formula AgO<sub>3</sub>. He would be glad to know whether Mr. Kingzett had any definite grounds for assuming the existence of that unfamiliar substance.

Dr. SQUIRE had at one time had a great deal to do with hydrogen peroxide, and found that, provided that it were properly made to begin with, ether preserved it very well, in fact, left nothing to be desired. But there was peroxide and peroxide, and these remarks applied only to a tolerably pure article. The amount of ether required was small, about one ounce to the gallon being sufficient to preserve the peroxide for six months at least. At all events, peroxide so preserved was sold under the name of Robare's Aureoline in thin flat glass bottles, which would burst with the slightest internal pressure, and in this form remained for months in hairdressers' shops with perfect safety. In what way the ether acted, he did not know; certainly not, as Mr. Kingzett suggested, in consequence of its vapour exercising a certain pressure. If a slight pressure were all that is required, this would be supplied by the partial decomposition of unprotected peroxide in closed vessels. The pressure thus produced ought to stop further action, but this was not found to be the case. In reference to the author's remarks as to the strength of the solution, he would point out that it was not only in America that a five-volume solution of hydrogen peroxide was sold as 10 volumes. By treatment with permanganate or bichromate of potassium 10 volumes were obtained, of course, but half of it came from the decomposing agent and the other half from the peroxide itself. He believed that by the universal custom of the trade the label 10 volumes meant evolving, not containing, 10 volumes.

Dr. STEINHART wished to know whether Mr. Kingzett had tested his solutions to which alcohol had been added, and whether the latter, in the presence of so powerful an oxidising agent as peroxide of hydrogen, had not been oxidised to aldehyde, and finally to acetic acid. It was well known that an acid solution was more stable, and it occurred to him, therefore, that the stability of the author's hydrogen peroxide in this case might be due to the presence of acetic acid and not to the alcohol.

Dr. S. RIDEAL thought it would be interesting to be able to compare the different monetary values of oxygen, free and combined. He would therefore ask the author to give the meeting, if he could, some data as to the monetary value of oxygen as an oxidising agent. They would thus be able to judge whether bleaching as performed by hydrogen peroxide would be cheaper or dearer than when due to bleaching-powder or other oxidising agent.

Mr. KINGZETT, in reply, said that the reason he had represented oxygen as either triatomic or tetraatomic was that it could not possibly be diatomic. If it were diatomic the two oxygen atoms in the peroxide of hydrogen molecule would be equally associated with each atom of hydrogen, and consequently of equal stability, which was not the case, seeing that one atom only was so easily dissociated. Chemical common sense clearly showed that the two atoms could not have the same combined value in peroxide as the one atom of oxygen had in a molecule of water. It followed, therefore, that it must be either triatomic or tetraatomic. He inclined to the view that it was triatomic, because it undoubtedly was so in ozone. As to the chemical equation on the slate which had been referred to in course of the discussion, he wished to disclaim all responsibility for it. It was due to the French chemist, Berthelot, and represented the analytical results of his investigation as communicated to the *Académie des Sciences*.

Dr. Squire's remarks were especially valuable, because of his large experience in the manufacture of peroxide of hydrogen; and he had hit the right nail on the head when he said that the action of the ether depended largely upon how the hydrogen peroxide itself had been prepared. Therein laid the crux of the matter. If it were carelessly prepared, if it were either acid or alkaline in character, or if it contained certain impurities, no agent would prevent it from decomposing within certain limits; but if it were properly prepared, as it could be by those who understood the subject, then the substances which he had mentioned were capable of preserving it from material loss of strength for 12 months or more. With respect to the strength of hydrogen peroxide solutions he knew that there were manufacturers here, dishonest enough—he used the term advisedly, for it was dishonesty—to represent that their five-volume solutions were 10-volume solutions, relying on the quantity of oxygen as obtained by the so-called permanganate test. In America they always did so; but of course that could be got over by testing the solution with acidified iodide of potassium, and estimating the iodine thus set free in the usual manner.

Dr. Steinhart had inquired as to the specific chemical action of alcohol, and had suggested that the stability of the peroxide, as preserved by that substance, was due to the formation of acetic acid and the production thereby of an acid reaction. That could not be, for if Dr. Steinhart would refer to Table 3 he would find that in the first two experiments (which were made with standard solutions) he had added 1 per cent. of sulphuric acid to one of them on the 46th day, and it would be seen that the restraining influence of the acid reaction was expressed by the fact that on the 405th day the unprotected solution had lost 83 per cent., whereas the protected solution (which for 46 days was maintained under the same conditions) had, owing to the addition of the sulphuric acid, lost only 60 per cent. Therefore the restraining action of the sulphuric acid was about 23 per cent. It would also be seen from the same table (experiment No. 6) that acetic acid certainly exercised an even greater restraining influence than sulphuric acid; so much so, that on the 405th day the loss was only 50 per cent. Therefore it had a greater effect *per se*. Whether the effects of the sulphuric and acetic acids were to be regarded as due merely to "acid reaction," or were to be viewed in their individual characters, there could be no question but that alcohol certainly restrained very much more than did either the one acid or the other, as was revealed by the figures submitted. It was therefore absolutely incredible that the preservative effect of alcohol is due to the generation of acetic acid therefrom.

Lastly, as to the monetary value of oxygen in its various forms, he was sorry not to be able to give any exact data.

Air was cheap enough, but it was not capable of producing those decolourising and oxidising effects which had to be obtained in the commercial bleaching of silk, cotton, paper, bone, hair, &c. Atmospheric oxygen, therefore, had no value in relation to such applications, nor was pure oxygen any more valuable, so far as he knew. Hydrogen peroxide, and that alone, had a distinct value for such purposes. Common air and oxygen were of no utility, either, for antiseptic purposes, whereas peroxide of hydrogen had a most pronounced value in that respect. With regard to bleaching-powder he had stated in his paper that peroxide of hydrogen was, so far as Germany and France were concerned, believed to be rapidly replacing bleaching-powder for bleaching woollen, silk, and other goods, partly in consequence of the destructive effect of chloride of lime on the tissues of such materials, and also because peroxide of hydrogen gave better results. He believed that the adoption of peroxide would follow more largely in England in time; and as to the comparative cost, the results which had been published justified him in saying that peroxide of hydrogen was not more expensive than bleaching-powder for such purposes, while its production was constantly being cheapened and its effective action increased.

## ON A NEW METHOD OF COLOUR ANALYSIS BY MEANS OF THE TINTOMETER.

BY J. W. LOVIBOND.

I HAVE to-night to describe and illustrate by a series of experiments a new instrument for the analysis and registration of colour.

I do not now propose to discuss the theoretical laws which govern the question of colour, having endeavoured as far as possible to keep myself free in my experiments from any undue bias to any particular theory.

The instrument and method to which I refer is a new means of impartially judging the various colours; for recording the factors of colour which combine to form any given colour; and a system for registering the same, even in the most complicated combinations, and the most delicate shades as well as in the deepest tints which exist in nature.

The illustrations which I propose to submit will represent work already done, and those cases where the application is commercial, have already become routine in the industries to which they apply.

*Description of Apparatus.*—The apparatus which is before you now on the table may be divided into two essential parts.

First, an optical instrument which gives two fields of view under equal optical conditions free from errors arising from unequal side lights, from the influence of adjacent bodies, and from any difference of colour-perception, which may exist between the right and left eye of the observer. The grave extent of error arising from the last cause will be seen on reference to the first set of columns in Table No. 3 on "Neutral Gray," where out of nine observers, only No. 1 has the colour-perception practically equal for both eyes; No. 2 equal for red and blue, but varies in yellow as 10·0 to 7·5; No. 3 varies in red as 10·0 to 9·0; in yellow, 10·0 to 7·5; in blue, 10·0 to 14·0; whilst No. 6 pronounced six observations as tinged with green; these are equally divided between the yellow and blue columns.

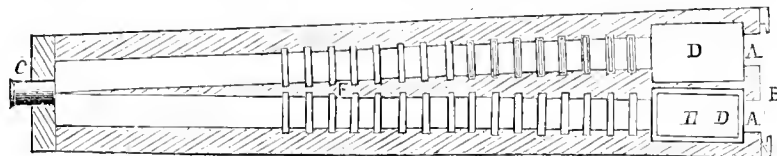
The second part is the standard scale, of which I shall speak presently.

The instrument consists of two tubes, A, A, side by side, divided by a central partition B, terminating at the centre of the eye-piece C in a knife edge, which, being inside the range of vision, is not noticed, so that light, entering the openings D D at the opposite end, passes in equal quantities

up each tube to the eye of the observer, giving a clear view of any opaque bodies which may be placed outside, or of transparent bodies inside the tubes.

The sides and central partition are grooved in order to hide the edges of the measuring glasses, and of the gauged

vessels for containing fluids. Stops are placed at convenient distances to cut off light reflected from the sides, and the size and shape of the apertures D can be altered by means of diaphragms to suit small or irregularly shaped samples.



**The Standard Scale.**—The second essential part is the standard scale, which consists of sets of coloured glass slips, all the glasses of each set being of the same colour, but each glass differing in depth of colour, the difference being in degrees of equal value throughout the scale.

In all comparisons of a single colour, the starting point has been taken as a pure white. The standard white used as a background for the glasses is pure lime sulphate for small surfaces, and for large surfaces, as for the reflectors, the smooth side of Messrs. Chance's opal glass, but whichever is used should be quoted.

I may here say that a comparison of pressed lime sulphate against snow shows no free colour in the lime sulphate, which is only .25 of a degree of neutral gray less white than the snow itself.

**Colour Scale.**—The relative positions of two-tenths, one degree, ten degrees, and twenty degrees are marked on the colour scale of primaries before you.

The equality of the scale can be tested in the lantern by placing a single glass of 20 degrees value on each side, and then making 20 degrees by any combinations from other parts of the scale as  $12 + 7 + 1$  equals  $3 + 8 + 9$ .

**The Unit.**—In the early days of the instrument, before the extent of its application for analytical purposes was fully realised, the necessity of dividing the colour scale into degrees of equal value before reliable work could be done, was evident. The starting point and value of these degrees were questions of the greatest importance and anxiety.

It was decided to fix upon as a starting point a definite degree of colour, the addition of which should be perceptible in the deeper shades, and dividing this degree into tenths and hundredths for the lighter shades, where the perception is keener, thus building up the scale by successive additions of equal value.

It will be seen that the starting point for each colour was arbitrary, that the value of the degree in each scale was equal throughout its entire length; but the impure colour in the glass then available made the fixing of a unit of common value for each primary colour almost hopeless.

A better quality glass, and more recent work, has, however, developed a possible basis for equalising the value of the unit for the three primary colours, red, yellow, and blue, and which are alone found necessary for most analytical work, and even holds out a hope of constructing a colour nomenclature with some degree of scientific accuracy.

I am aware that the colour in my primary standards, red, yellow, and blue, are not pure, but as they are the purest yet obtainable, and as by their means most of the colours in nature are measurable, I will ask that, for the purpose of illustration only, they may be considered as the three primary colours in glass.

**The Neutral Gray.**—The new departure is founded on the fact that neutral gray under certain known conditions is always made by a combination of 1 red, 1.2 yellow, and 2.4 blue in the original scales, and these proportions hold good at all depths. Then, by assuming these combining values of red, yellow, and blue to be the units of their respective colours, a simple relationship of equality between these three primaries is established, at least so far as the production of neutral gray is concerned.

Having established this common equivalent as a unit of colour in reference to neutral gray, by removing the yellow

equivalent a normal purple is left, made up of one equivalent of red and one of blue. By removing the blue equivalent a normal orange is left, made up of one equivalent of red and one of yellow. By removing the red equivalent a normal green is left, made up of one equivalent of yellow and one equivalent of blue.

Further, by placing 10 normal orange on each side of the screen, and adding a single degree of red to one side, and a single degree of yellow to the other, we have a 10 normal orange + 1° red, and a 10 normal orange + 1° yellow. Test this further with higher differences, as 5 on each side, and also with normal purple and normal green.

If binary colour is taken to be the sum of any two colours composing it, then any departure from the normal towards either primary can be accurately measured and described.

When a colour is composed of the three primaries, the units of neutral gray may be deducted as such, and the balance looked upon as free colour. Let us see how this works out by matching a sombre colour, such as brown paper, on the screen, by, say, 5 red, 6.3 yellow, and 4.4 blue. The equation may be written thus:—

$$\begin{array}{rcccl} & \text{R.} & \text{Y.} & \text{B.} & \text{N. G.} & \text{R.} & \text{Y.} \\ \text{Brown paper} & 5 & + 6.3 & + 4.4 = & 4.4 & + 0.6 & + 1.9; \end{array}$$

the latter part of the equation, 4.4° neutral gray, .6° of free red, and 1.9° free yellow, is an accurate description, and conveys an intelligible idea and record of the colour itself in this particular brown paper. Test this further by separating the free colour from the neutral gray. The method is available for the measurement of all sad-coloured bodies.

It also follows that, by the constant addition of neutral gray tints, a scale of equal degrees, ranging from white to black, may be established, and available for measuring the penetrating power of light.

Some precautions are necessary in selecting the proportion of primary colours for the composition of a true neutral gray; for instance, a person whose sight is abnormally strong or weak, even for a single primary, would never agree with an average selection, and this difficulty is increased when the abnormal condition applies to one eye only, as then each eye gives a distinctly different reading. The colour of the light must also be considered, as that from a blue sky, a dull sky, or a white cloud gives different results. The angle at which the daylight is taken has also an influence, the light gaining in red as the instrument or reflector declines from the perpendicular to the horizontal.

The proportions of primary colours for the neutral gray standard I have been dealing with is the result of observations taken in the open air, under a dull gray sky, with light as nearly perpendicular as possible, reflected from Messrs. Chance's opal glass, and is the average of 324 observations made by nine persons of my own staff, none of whom are colour-blind in the ordinary sense of the word, but all have more or less abnormal perceptions for one or more primaries in one of their eyes. I have not, however, removed any of the observations from the table of averages on this account.

A reference to the Table No. 3 will show that of the 324 observations, 130 were pronounced neutral gray, 91 slightly tinged with yellow, 80 with blue, and 23 with red. In no case had the observer a knowledge of what he was judging, and the judgments were frequently checked up and down



TABLE OF COLOUR MEASUREMENTS OF COMMERCIAL SUBSTANCES.

	200 Red Series.		510 Yellow Series.		1180 Blue Series.		Neutral Gray.		Free Red.		Free Yellow.		Free Blue.
Annatto paste A .....	13.6	+	11.8	+	5.	=	5.	+	8.6	+	6.8	..	
„ B .....	15.75	+	7.25	+	2.25	=	2.25	+	13.5	+	5.	..	
PAPER:													
Brown paper .....	6.6	+	8.3	+	5.4	=	5.1	+	1.2	+	2.9	..	
No. 1 writing paper .....	0.27	+	0.45	+	0.31	=	0.27	+	0.04		..	+	0.04
No. 2 „ .....	0.33	+	0.51	+	0.5		0.33	+	0.18		..	+	0.17
FABRICS:													
Red sateen .....	28.8	+	6.1	+	5.8	=	5.8	+	23.0		..	+	0.3
Purple velvet .....	36.0	+	3.3	+	9.6	=	3.3	+	32.7		..	+	6.3
Light blue satin .....	..	+	1.2	+	4.1								
PAINTS:													
Chrome yellow on paper .....	2.7	+	12.0										
Orange chrome „ .....	9.8	+	4.5										
FLOUR:													
Hungarian E.O.P. No 0, Lofty ground .....					Amber, Series 52. 2.0				Brown, Series 50. ..				Gray, Series 30. ..
„ „ Fine „ .....					1.75				..				..
Spring wheat flour .....					..			4.75	+		0.25		
English super .....					0.5	+		2.75			..		
CANDLES:													
Patent No. 1 .....					Series 52. 1.75			+			Series 500. 0.375		
Patent No. 3 .....					3.5			+			.75		

**Conclusion.**—In submitting this apparatus and method to your judgment, I am fully conscious that there must be many questions which time and more exhaustive work will elucidate, but I am not conscious of having hidden a weakness or exaggerated a strength, and feel sure that when the weaknesses are pointed out, they will act as a spur to further investigation. And in conclusion I wish gratefully to acknowledge the assistance received by me from many quarters; it will be impossible for me to enumerate all, indeed, in some instances I have no knowledge even of the names of gentlemen who have made valuable suggestions.

I am, however, specially indebted to Mr. H. Le Neve Foster, who worked out the method of quantitative estimation of carbon in steel; to Mr. T. Jobson, jun., of the Stocksbridge Works, near Sheffield, who overcame a difficulty in steel solutions of a reddish tinge; to Professor Hummel, of the Yorkshire College, Leeds, who suggested a method of bringing surfaces uneven in texture or colour to a suitable condition for comparison by throwing them out of focus with a lens at the eye-piece; to Dr. Knecht, of the Technical College, Bradford, for some comparisons in Turkey reds; to Mr. Boverton Redwood for co-operation in working out standards of colour for petroleum and cocoa-nut oils; to Dr. Munro, of the Downton College of Agriculture, for investigation into the method of making ammonia estimations, and for some special work on the colour of waters. The fine divisions in the standard scale is mainly the work of one of my daughters.

The working out of the neutral gray equation is the direct result of conversations with Professor Crookes, Professor Church, and Captain Abney, greatly aided by the brilliant lectures of the latter at the Society of Arts last session.

#### DISCUSSION.

The CHAIRMAN said that the subject of the paper was one of very wide interest, and capable of most important practical application. All chemists had to do with the

judging of colours, and all had a profound conviction of the perfection of their individual colour sense. Everyone present knew how delicate and difficult a matter it was to hint to a friend that his eyesight was not quite what it should be. With regard to the sense of sight, everyone considered himself perfect. He happened to have a number of relatives so far colour-blind as to be capable of imagining emeralds to be rubies, and *vice versa*. In all his experience in this direction, however, he had never met with a colour-blind woman, and did not believe that there was one. He would appeal to Mr. Lovibond on that point.

Mr. LOVIBOND said that he could agree with the Chairman's experience to this extent, that the only absolutely non-colour-blind person he had ever met was a woman.

The CHAIRMAN, continuing, said it was certain that as a rule women's eyes were far more accurate than men's. We ought to recognise the fact that a great many of us were more or less colour-blind with respect to certain tints; and if chemists in particular did so they would save a great deal of inaccurate work. He believed that it was in that room that Dr. Tidy had related his experiences in testing the average eye for accuracy. He could not recall the exact figures, but he remembered that a very small percentage of those tried could, for instance, sort out twenty shades of brown with an approximation to accuracy. Nevertheless, it was possible by careful training to greatly develop the power of the eye in this respect. To do so one must have accurate instruments and methods; and in Mr. Lovibond's apparatus and his method of gauging the different depths of colour, there seemed to him to be the means of training the eye to the work which the eyes of chemists were called upon to do. The matter was one well worthy the study not only of those to whom it was a subject of special importance, as to those engaged in dyeing, but of every chemist. They had to thank the Royal Institution for the loan of the lantern, and Mr. Heath for attending to exhibit the various colours on the screen.

Mr. BOVERTON REDWOOD was glad to have an opportunity of bearing testimony to the value for certain technical purposes of the instrument which Mr. Lovibond had taken so much trouble to exhibit and explain to the meeting. In connexion with the class of products with which he had to deal, it had been his lot for 20 years past to be almost daily engaged in chromometrical determinations, and consequently when Mr. Lovibond brought the tintometer under his notice some two years ago, he examined it with great interest. He had already had the opportunity of explaining, in that room, the principles of construction of the instrument with which, in the petroleum trade, they were in the habit of determining the colours of some of their products. That instrument had certain defects; and he had never found it well adapted for the registration of the colour of such oils as were employed for lubricating purposes. He had suggested, therefore, that there was an opening in that direction for such an instrument as the tintometer, and had gladly concurred in Mr. Lovibond's proposal that they should together endeavour to arrange a scale of standards suited for commercial use in the lubricating oil trade. He might say that in his laboratory for nearly two years past they had been in the habit of testing with the tintometer and recording the colour of a two-inch stratum of every sample of lubricating oil submitted to them, and there had thus been established a numerical scale of colour which already possessed some significance. They had of course experienced difficulties at times in consequence of the variable character of the daylight available—difficulties which all who were in the habit of working in London would appreciate. He did not refer to such exceptional cases as the darkness which had recently enveloped the city; but apart from such cases as that, Londoners had to work under atmospheric conditions which would afford Mr. Lovibond food for serious study after the more transparent atmosphere of Salisbury. He had reason to hope that at no distant date some artificial light equivalent to ordinary daylight, or capable of being employed alternatively, with some definite correction, might be provided. In that case the tintometer would come more largely into use than under present conditions. The experience they had had of the use of artificial light that evening was not encouraging; and, as one who had had some experience in the use of the instrument by daylight, he was bound to say that the impressions to be derived from the demonstrations just given were less favourable than those which would result from a short use of the instrument by ordinary daylight.

Mr. W. THORP felt grateful to Mr. Boverton Redwood for the encouragement conveyed by his concluding remarks. He had often to experience the difficulties due to the great peculiarities of what we were pleased to call daylight in London. It was no uncommon thing for a part of his work to be set aside for days, or even weeks, in consequence. Referring to the Chairman's remarks, he thought that any who had a short experience of a colour works would soon have any ideas of the perfection of their own eyesight knocked out of them. There were certain men at the works with which he was connected who had especially good eyes for certain colours, and when an examination of those particular colours was required it was done by those men. He himself had, he thought, a good eye for some colours, but there were others that he would not dream of judging. Under these circumstances the instrument before them ought to prove valuable. It would certainly be useful in giving a numerical value for depth of colour. He was not so clear as to its value for giving an exact representation of tone or tint, especially as it seemed to him, judging from the experiments just made under artificial light, that the tints produced by the mixtures of glasses were not what could be called *pure* colours. He believed, indeed, that Mr. Lovibond had referred to that point in his paper, and had said that they were not pure. It was often a matter of great importance to decide whether a colour was rich or poor, and how the instrument would deal with such a point as that he could not say. But while looking at the pieces of glass exhibited, a horrible doubt occurred to him as to their permanence, and he would be glad to know from the inventor whether he had considered that. People were very apt to think that coloured glass was permanent, yet probably most of those present had seen specimens of glass which had

begun their existence as colourless, and which before long had acquired an appreciable colour. He remembered seeing some glass which in 18 months had attained an intensity which would be represented on the scale before them by at least 2 degrees. If changes increasing or diminishing in depth, or varying, were to be feared, it would be a great impediment to the application of such instruments. How such a difficulty was to be met he did not see, but it was certainly a point worthy of careful consideration.

Dr. ALDER WRIGHT said that there were one or two points upon which he would be glad to have further information. In the first place, he did not understand what was meant by one, two, or three degrees. Did it mean that the glass was, for example, tinted with one, two, or three grains respectively of some colouring material per pound? Secondly, how far were the colours which were transmitted through the glasses mono-chromatic? In other words, if the glasses were viewed through a spectroscope, would one get a single line as the absorption spectrum, or a large proportion of bands at other parts of the spectrum? If there were any considerable proportion of other colours, it would be difficult to ensure that any combinations of glasses would give even approximately pure colours. He supposed it would be almost improper to inquire what the materials were with which the glasses were tinted. But if the author could give any information which would tend to show that a scale could be reproduced with accuracy at a future period, it would be of considerable interest to all possible users of the instrument.

Mr. FRIESE-GREENE wished to ask the author whether he had taken into consideration the effect due to the surfaces of the glasses used. It was obvious that as he increased the number of glasses in building up his colours, he would get more surfaces and, consequently, more reflection due to the breaking up of light all along the different media. His own experience in photography led him to think that this was a point which might be of considerable importance in the case of the instrument under notice. Again, it was well known that the eye in going from one colour to another always carried with it as it were the tone of the first colour; and not only so, but there was a kind of opposition set up by the retina endeavouring to neutralise the colour. Consequently, if the eye passed quickly from one colour to another, there was produced on the retina a mixture of the first colour or tint with the second. That was a matter which ought to be taken into consideration. It might be met by the observer going into the dark until the tone was neutralised, and then looking at the colour.

Dr. L. THORNE said that he had had one of Mr. Lovibond's tintometers in practical use for some time, and could fully corroborate Mr. Redwood's remarks as to the advantages to be derived from it. He had used it for estimating colour in oils, and also with regard to the colours of more or less bleached natural products, and had found it valuable in either case. Referring to what he might call the optical part of the instrument, he considered it a great advance on the apparatus hitherto used. Getting the one absolute field split up as it were by means of the wedge-shaped division, so that with the same eye one could get at the same focus the two fields in equal physical condition, enabled one to obtain an estimation of colour much more accurately than by any other process. As to the permanency of the standard colours, no doubt Mr. Lovibond would have something to say. But assuming that point to be satisfactory, he could strongly corroborate what had been said as to their value for future work. A colour having once been matched, the standards required for it could be noted, and the colour could be reproduced with absolute accuracy at any future time. He had not had a large range of colours to work with, but those which he had tested he had found to be matchable with ease; occasionally a very bright tint would be a little awkward to match, but not often. To him the point of special novelty in the paper was the portion relating to the production of neutral grays. It was a matter of the greatest interest from the theoretical point of view that the grays could be made up from those three standard colours, and that different depths of gray would be shown by exactly



the same proportions of the three colours. This led one to suppose that there was a possibility of getting what might be taken as natural standard mints of tone in the different standard colours in place of the purely arbitrary units at present employed. Referring to Dr. Wright's remarks, he believed it had been mentioned that at present the scales of colours were essentially arbitrary ones. One standard—a pale colour—was taken as a unit in each tint, and the other shades were built up from that by actual trial with the glasses. So far as his own experience had gone he had found the tintometer capable of doing very good service in dealing with both liquids and solids.

Mr. J. J. EASTICK said that he had had one of Mr. Lovibond's instruments in use for the last three years, and had found it simply invaluable. He had, however, experienced one or two difficulties. One was, that some of the colours of the solutions which he tested were brighter and nearer the primary colours than the glasses themselves. Another difficulty—or rather he might say a warning—was that if an observer was looking through an inch of colour in certain liquids, and the colour was, for example, 20, if he then looked through two inches of the same liquid the tint would not be 40, as might be expected. He did not know whether that was the case with all colours, but it certainly was so with several. He had heard with great pleasure the hint thrown out by Mr. Boverton Redwood as to the possibility of overcoming the difficulties of artificial light. Even if the promised light was not so perfect as the reflection from a white cloud, it would still be a boon if it was an improvement upon the present means of carrying on operations during a fog or night.

Dr. MUNRO desired to add a few words as to the use of the tintometer for measuring the colour of water, as he thought it likely to be especially useful to chemists in that direction. In Table 6 he had given, as an illustration, some measurements of a number of waters from one neighbourhood—all within half a mile of a common centre. It would be seen from the figures in the last column that these waters ranged from seven-tenths of a degree of colour in a 2 ft. tube to 53 degrees, which was a most extraordinary variation. He had found the instrument very useful in following certain changes in the Lisdoonvarna sulphur spas. The twin sulphur spas, as shown in the table, always had the same composition; but near it was the Gowlaun spa, from the same formation, which was subject to extraordinary variations in strength. It was to be noted that there was a certain variation in colour also; and so there was probably some connexion between the two things. That enabled them to form some idea of what went on under the ground, for the variation in strength was perhaps due to the percolation of water; and they could in that case ascertain by the variation in colour what particular water was getting in. He had had an opportunity of seeing Mr. Lovibond's neutral gray by daylight, and as far as he could judge it was free from any positive tint.

Mr. LOVIBOND, in reply, said that in reference to Mr. Boverton Redwood's remarks as to the bad quality of London light, it had been found a great difficulty at first, as the small surfaces commanded by the two apertures did not admit sufficient light to illuminate the object. But he had recently overcome it to a great extent by including a reflector at a low angle, so as to collect the light from a large surface and throw it into the comparatively small space. An illustration of this would be seen upon the table in the form which his assistants called the Eiffel tower. He had not as yet given much attention to the use of the instrument with artificial light, but he had no hesitation in saying that, given the said light had permanent characteristics, there would be no difficulty in establishing its relative value compared with daylight, and thus making it available with a certain constant correction. With reference to Mr. Thorp's doubt as to the exact representation of tint—that was the strong point of the instrument, and half an hour's work by daylight would completely satisfy the most sceptical on that point. As to the purity of the colours, he was very much at fault. He had only a certain material to deal with, and he made the most he could of it. He had a blue, for instance—he

called it a blue, but it was really a purple; for it was a cobalt put on white glass. To seek for absolute purity of colour in glass, taking the spectrum colours as his standard, would be futile. But for ordinary colour measurement, all that was needed was to employ so much more or less red, blue, or yellow to compensate for the departure from purity in the standards. This was no real detriment to the value of the instrument for measuring commercial colours; but it would be fatal to the measurement of pure colours *directly*—though they could be measured *indirectly* by toning the pure colours down by a series of neutral grays. As to the permanence of the colours, he had the assurance of the makers, the red being oxide of gold, the yellow silver, and the blue cobalt. Under these circumstances he could not guarantee their permanence; but if any chemist would establish the value of a unit for any coloured chemical compound, he would have the power of testing for himself its permanence. As to the value of a degree on his scale of tints, that was purely arbitrary, and it was fixed so that the difference of a degree up to 30 might be just perceptible. It started at 1; then the 2 was equal to two 1's. If he had to deal with two glasses against one, he added a white glass to the one, so as to equalise the number of surfaces on each side. As to the spectrum value of the colours, he had already said that the two were not comparable. If he had to compare his colours with the spectrum colours, he should tone the latter down to his colours and get the indices on the spectrum side. He said this to show the elasticity of the instrument to meet any question asked of it. The accuracy of the scale of colours was in the hands of the chemist; and if anyone found a scale inexact he hoped they would bring it to his notice. No real difficulty existed in grading the colours beyond the necessary labour, which was excessively trying. As to reproducing the scales, he had deposited standards at the colleges of Liverpool, Leeds, and Bradford, on the condition that anyone using the tintometer should be at liberty to take his glasses to the college and test them with the standards. With reference to Mr. Friese-Greene's remarks as to the interference of complementary colour sensations, that was a serious difficulty in judging the value of any one colour; but when judging the difference between two colours placed side by side, as in the tintometer, it counted for nothing. In matching two colours in the instrument with one eye, any interference equally affected both sides, and whatever colour impressions might be conveyed to the eye by the interference, the measuring glasses required to make a match were the same. That was proved by the diagrams which dealt with the judging of certain colours under different conditions; in which it would be seen that although the lights varied, the same results were obtained by a considerable number of persons, thus showing that the question of outside influences (arising from adjacent colours) did not apply. The observer was simply judging of two things before him; and if one observer called it a green, while another said it was a red, that made no difference when the two sides were equal. The glasses were the indices of the difference; and as the glasses were always the same, they reduced these hitherto unmeasurable differences to a matter of common understanding. He had not as yet met with any difficulty as to the reproduction of his scale. If difficulties did arise, all he could say was that they would have to be overcome. Solutions brighter than the glasses were at one time a source of difficulty which was only overcome by the discovery of the theory of the neutral grays. If the observer had a solution brighter than the glass, he would first add an equal number of glasses, and if that were not sufficient, it was evident that he was dealing with a purer colour than the glasses themselves. He would then add glasses to the pure side and bring that down to the impure. In either case the glasses required would be the indices of the difference.

## Manchester Section.

Chairman: Ivan Levinstein.  
Vice-Chairman: Edw. Schunck.  
Committee:

J. Angell.	J. Grossmann.
G. H. Bailey.	P. Hart.
R. F. Carpenter.	A. Liebmann.
G. E. Davis.	Sir H. E. Roscoe, M.P.
H. Grimshaw.	C. Truby.
Harold B. Dixon.	D. Watson.

Hon. Local Secretary:

J. Carter Bell, Bankfield, The Cliff, Higher Broughton Manchester.

SESSION 1890.

Notices of Papers and Communications for the Meetings to be sent to the Local Secretary.

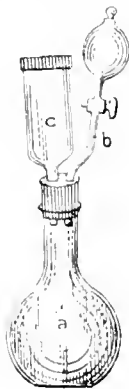
Meeting held at Victoria Hotel, Manchester, on  
Tuesday, 7th January, 1890.

MR. PETER HART IN THE CHAIR.

### WIBORGH'S METHOD FOR THE ANALYSIS OF SULPHUR IN IRON AND STEEL.

J. B. COHEN, PH.D.

THIS method, which has recently been published in detail in Professor Ledebur's new volume on iron and steel analysis (*Leitfaden für Eisenhütten-Laboratorien*: Vieweg und Sohn), is described as rapid and accurate. The apparatus is shown in the figure.



It consists of a wide-necked flask, *a*, of 250–300 cc. capacity, to which is fitted a double-bored cork. Through one hole passes a tap-funnel, *b*, which terminates just below the cork, and through the other a cylindrical glass tube, *c*, about 21 cm. long, open at both ends. The upper end is about 6 cm. in diameter and the lower end drawn out so as to pass through the second hole in the cork. The amount of sulphur is determined by the depth of the yellow stain produced by  $H_2S$  on the surface of a piece of calico previously soaked in a 5 per cent. cadmium acetate solution and dried, the calico being stretched across the wide open end of the cylindrical tube.

The calico may be stretched by means of two stout india-rubber rings  $\frac{1}{2}$  inch in width, which fit tightly round the upper part of the wide tube. One ring is allowed to project above the tube so as to form a horizontal edge, and over

this the calico is stretched by means of the second ring. Ledebur employs a wooden ring and two clamps, but the above method answers perfectly well.

The flask is first half filled with water and boiled on a sand-bath to expel air, the sample of iron or steel, .1 to .8 gm., according to the amount of sulphur, is quickly introduced, and the water again boiled for a few minutes; dilute sulphuric acid (1:5) is introduced from the tap-funnel gradually until the iron is dissolved without interrupting the heating.

Sulphuretted hydrogen is evolved and the calico is stained. To obtain an even stain it is necessary that the cylindrical tube should have a vertical position. When the iron is completely dissolved, the boiling is continued for 8 to 10 minutes. The calico is now removed, well rinsed, and dried, and the stain compared with a set of standard colours. The set of standards (six shades are sufficient) is prepared by taking a sample of iron with a known quantity of sulphur and obtaining various shades with different weights of the sample by the method described above. These should be mounted on white cardboard and kept for comparison. Having made an analysis and fixed upon the standard shade which comes nearest to that of the sample, the weight of sulphur is determined by the following formula:—

If *w* is the weight of the standard sample corresponding to the standard shade containing *s* of sulphur, and *w*<sub>1</sub> is the weight of the sample taken, then the weight of sulphur *s*<sub>1</sub> in the sample is  $s_1 = \frac{w s}{w_1}$ .

The same standard shade can therefore be used for a variety of samples when different weights are employed.

### A PRODUCT OF THE DISTILLATION OF ROSIN.

BY F. BROWN-SWORD, B.S.C., AND J. B. COHEN, PH.D.

A SAMPLE of the aqueous distillate from a rosin still was sent us for examination. As we have been unable to find any mention of the constituents of this distillate, we thought a short notice of our results would be of interest to the Society.

The proportion of this aqueous distillate amounts to about one-third per cent. of the charge. It has a strongly acid reaction and smells of rosin oil, of which there is always a small quantity floating in oily drops on the surface. The acid, which was found to be acetic acid, was present partly as free acid, partly as ferrous salt. The acid, which in one case was collected in wooden barrels, had evidently attacked the iron condenser or pipe leading from the still. We received samples of two distillations. The quantity of acid was determined as follows: To 1,000 cc. of the distillate ammonia was added until alkaline and  $H_2S$  passed into the solution to precipitate the iron. The filtrate was concentrated and boiled with excess of baryta and the excess precipitated with  $CO_2$ . The solution was filtered and evaporated to dryness. In the litre 23.8 grms. of barium acetate were found. In a second quantity, 500 cc. yielded 12 grms. barium acetate. This is equivalent to 12 grms. acetic acid in the litre, or rather more than 1 per cent.

In the second distillate the barium salt was obtained as before, and converted into the sodium salt, which was fused and weighed. One litre gave 170 grms. of fused salt equivalent to 120 grms. of acetic acid, or about 12 per cent. The sodium salt was heated with concentrated sulphuric acid, and the acetic acid which distilled was again distilled over  $P_2O_5$ , when the product froze on cooling to 0°. The lead salt prepared from this acid gave the following result on analysis. .5035 gm. gave .403  $PbSO_4$ .

Calculated.	Found.
54.61	54.57 per cent.

## A COMPARISON OF THE METHODS IN USE FOR ESTIMATING ORGANIC NITROGEN.

BY R. W. ODDY, A.I.C., AND J. B. COHEN, PH.D.

THERE is no analytical process which has had more demands made upon it in recent years than a rapid and reliable method for estimating organic nitrogen. The new process of Kjeldahl appears to have supplied these requirements. That of Wanklyn is obviously inadequate, especially as it has been shown that ammonia is decomposed by alkaline permanganate on long boiling. Dumas' method has stood the test of time.

It appeared to us interesting to make a comparison of these three methods, and to determine how nearly they agreed.

Colourless isinglass was selected as a typical albuminoid substance. It was powdered and dried and analysed by the three processes.

The analyses by Dumas' method, which needs no description, gave the following results:—

No. of Analysis.	Weight taken.	Ce. of Nitrogen.	Temp.	Pressure.	Per Cent. of N.
1	Grm. '1292	17.2	20	Mm. 746.5	15.28
2	'1595	20.6	19.5	760	15.2
3	'201	27.6	20	755	15.93
4	'1771	23.3	21	755	15.22
5	'1335	17.8	17	760.1	15.74

As this method always gives too high a result due to the difficulty of completely expelling all air from the tube, we may take 15.2 per cent. as a close approximation to the truth.

A portion of the same sample of isinglass was dissolved in water, and the amount of the albuminoid ammonia determined exactly as described by Wanklyn in his volume on Water Analysis. It should be mentioned that a *very small* quantity of the sample remained undissolved and in suspension, and the analysis is to that extent unreliable. On the other hand we are not limited in Wanklyn's process to dissolved albuminoids, and, moreover, the result agrees exactly with Wanklyn's analyses of the same substance (*vide* p. 163, sixth edition, p. 183, seventh edition).

We found 12.75 per cent. of ammonia, Wanklyn 12.7 per cent. of ammonia, equivalent to 10.5 per cent. of nitrogen.

As might have been anticipated, the percentage is much too low, being nearly two-thirds of the actual amount.

The method of Kjeldahl was carried out as described in Dittmar's book on Quantitative Analysis, p. 83. The sulphuric acid used was distilled, as it was found that the commercial acid contained nitrites and nitrates. In distilling the sulphuric, the first portion passing over was rejected.

The following are the results:—

No.	Per Cent. N found.	
1	13.50	With pure sulphuric acid.
2	13.50	
3	13.59	
4	14.12	With a sample of commercial H <sub>2</sub> SO <sub>4</sub> .
5	14.25	
6	14.14	
7	15.8	With another sample of commercial H <sub>2</sub> SO <sub>4</sub> .
8	15.2	

With the exception of No. 2, in which litmus was used as an indicator, the titration was done with methyl orange.

Taking 13.5 per cent. as nearest to the truth, we see that it is very much too low in comparison with Dumas' process.

Although the majority of observers have obtained satisfactory results by Kjeldahl's process, and it is undoubtedly to be recommended in the case of the more readily decomposed organic compounds, as we have proved in the case of acetanilide, we have no doubt that in the case of the less easily decomposed bodies the results have a tendency to be much too low.

## THE STABILITY OF PERMANGANATE SOLUTIONS.

BY R. W. ODDY, A.I.C., AND J. B. COHEN, PH.D.

It is known that solutions of permanganate of potassium lose in strength on keeping, but the exact amount of this loss has never been ascertained. The object of the present paper has been to determine this amount.

A solution of permanganate was prepared in the usual way by dissolving about 5 grms. of the crystallised salt in about a litre of distilled water, which was then boiled for two to three hours, and, when cold, diluted to one litre. The strength of this solution was determined by titration with iron wire dissolved in sulphuric acid, and also by solution of ferrous ammonium sulphate of ascertained purity. The results of the two determinations agreed exactly. The solution of the permanganate was now divided between two well-stoppered bottles, one of which was kept in the dark, the other exposed to the light during a period of about four months, and the strength ascertained at intervals.

The testing of the permanganate was carried out in the same manner as already described. On each occasion fresh solutions of the same sample of iron wire in sulphuric acid and of ferrous ammonium sulphate were prepared.

The results show that light has no influence in the change, but that regular though slight decrease in strength occurs, as shown by the tabulated results.

Strength of 1 cc. KMnO <sub>4</sub> in Grms. of Fe.			Strength of 1 cc. KMnO <sub>4</sub> in Grms. of Fe.		
Date.	Exposed to Light.		Date.	Kept in Dark.	
	Iron Wire.	Double Salt.		Iron Wire.	Double Salt.
1887. Nov. 14	'00968	..	1887. Nov. 14	'00968	..
Dec. 1	'00964	'00963	Dec. 1	'00964	'00963
" 10	'00960	'00960	" 10	'00963	'00960
" 20	'00961	'00959	" 20	'00959	'00962
1888. Jan. 10	'00955	'00959	1888. Jan. 10	'00959	'00959
March 8	'00940	'00941	March 8	'00948	'00952

By the method of testing, a single drop of permanganate might produce an experimental error equivalent to about  $\frac{1}{2}$  per cent. of iron in each cc.

The table, therefore, is only reliable to .5 in the fifth decimal place. There is, however, a difference during the four months of a full 2 per cent., allowing for experimental error, though the probability is it amounts to 3 per cent.

The decrease is apparently regular. During the first two months, November 14—January 10, there is a loss equivalent to 1 per cent. of iron, and during the last two months nearly the same. This loss translated into percentages will of course vary with the amount of iron in the sample. In the case of a sample with, say, 25 per cent. of iron, this amounts to  $\frac{1}{2}$  to  $\frac{3}{4}$  per cent.

It therefore appears that permanganate solutions which have been previously well boiled should be tested once a fortnight to ascertain the amount of change, and that the amount of change during this period will not exceed that of experimental error and may be neglected.

MR. J. CARTER BELL said that he had kept solutions of permanganate for many months, and had found the change to be very slight. Nearly 30 years ago Dr. Percy had experimented upon this very subject, and in March 1862 he prepared a large quantity of permanganate, and on that date he obtained as a mean of four experiments a standard of 9.854 (1,000 grains of solution = 9.854 grains Fe).

The solution was again standardised in March 1863, and the mean of two experiments gave a standard of 9.797 (10,000 grains of solution = 9.797 grains Fe), or an alteration in 12 months of only .057 grain.

There was no necessity to boil the solution of permanganate if pure distilled water were used.

## THE RATE OF DISTILLATION OF ACETIC ACID.

BY J. A. WILSON.

THE results of the experiments contained in this paper were obtained some short time ago, in an attempt to apply the principle of researches made by M. Duclaux, on the rate of distillation of various fatty acids, to the valuation of commercial acetates. Duclaux found on distilling solution containing various fatty acids, that each acid possessed a rate of vaporisation peculiar to itself, moreover, the least volatile acids, distilled in the portions of distillate, coming first, whilst acetic and formic acid behave in the opposite manner. In the case of acetic acid, Duclaux found that when 110 cc. of a liquid containing acetic acid were distilled in a retort of 250 to 300 cc., that by the time 100 cc. of the liquid had passed over or 10/11, it contained 79.8 per cent. of the total quantity present. Formic acid does not distil so easily, whilst on the other hand propionic acid is more volatile, and butyric acid again more than the latter. When two or more acids are present in a liquid, each maintains its own special characteristic rate of distillation. Tables showing these special characteristics may be found in most of the works on organic analysis (Allen's Commercial Organic Analysis, vol. I, p. 408; Wynter Blyth, Food, p. 488), and I may say that I have not been able to find any other researches on the same subject other than those under discussion. The experiments made were mostly from a technical point of view, and, as stated above, mainly with the intention of applying the principle to the commercial assay of acetates, acetic acid, and vinegars. In most of the text-books on analysis tables are given showing the rate at which each acid distils, but no mention is made of the strengths of the solutions the author experimented with. However, as the process was mainly intended for the detection of volatile acids of wine, we may reasonably consider they would be dilute. In my experiments a 250 cc. retort was employed attached to a condenser 22 in. long and diameter of  $\frac{1}{2}$  in., and the neck of the retort was not surrounded by a dome, or wrapped with flannel, or any other of the devices to confine the heat and thus prevent condensation.

Pure semi-normal alkali was used for the titration, with phenol phthalin as indicator.

In Table 1, 110 cc. of the acid of strength mentioned in column 1 were pipetted into a 250 retort and distilled at the usual rate till 100 cc. had passed into a graduated receiver, the amount of acid in the distillate being then found by heating with slight excess of alkali, cooling, and then adding semi-normal hydric chloride till pink colour discharged. That signified under total was obtained by direct titration.

TABLE 1.

$C_2H_3O_2$ per Cent. Total.	$C_2H_3O_2$ in 10/11 Distillate.	Per Cent. of $C_2H_3O_2$ in 10/11 Distillate of that in Retort.
1.015	0.928	83.15
2.287	1.896	82.90
4.18	3.81	83.52
10.45	9.49	82.56
26.19	23.73	82.37

The above experiments were carried out most carefully, and in duplicate, and I was somewhat disappointed in them. I ought here to mention that there being danger in distilling such as the No. 5 strength of acid, an aliquot portion was taken, so as to bring it approximately to No. 4, and made up to 110 cc. and distilled to 10/11 as before.

Table 2 shows the results obtained from three commercial samples of vinegar, with the difference that the figures under total are obtained by successive distillations with addition of water.

TABLE 2.

Acetic Acid per Cent., Total.	Acetic Acid in 10/11 Distillate.	Per Cent. of Acetic Acid in 10/11 Distillate to that in Retort.
4.406	3.705	82.96
4.530	3.78	83.44
4.75	3.97	83.58

The above samples of vinegars gave precipitates with silver nitrate and barium chloride; on account of the former a little silver sulphate was added. Commercial vinegars as a rule contain from 2.5 to 3.7 per cent. solid matter, but they do not appear to have affected the distillation much.

Table 3 shows the influence of neutral salts, sodic chloride, and sulphate, on the speed of distillation, No. 3 being accounted for on the pressure of approximately 56 per cent. of hydrated water.

TABLE 3.

$C_2H_3O_2$ Per Cent. Total.	Amount of Neutral Salt per 100 cc.	$C_2H_3O_2$ in 10/11 Distillate.	Per Cent. in 10/11 Distillate to that in Retort.
10.26	5 grms. pure NaCl.	10.47	92.77
"	10 " " "	10.92	96.76
"	5 grms. $Na_2SO_4$ 10 aq.	9.75	86.39

The influence of phosphoric acid was the subject of two more experiments, the solution being prepared by dissolving 220 grms. of glacial phosphoric acid in 200 cc. of water. The purity of the acid was proved by treating 20 cc. on the retort and distilling to dryness, no volatile acid being obtainable.

TABLE 4.

100 cc. Acid Solution, 10 cc. Phosphoric Acid, $C_2H_3O_2$ Total.	$C_2H_3O_2$ in 10/11 Distillate.	Per Cent. of $C_2H_3O_2$ in 10/11 to that in Retort.
10.05	9.90	98.51
4.25	4.16	97.88

The experiments I next made with the commercial acetates were conducted in the usual manner, in the same retort, viz., that of 250 cc. capacity, which is considerably larger than that usually employed, which is about from 125 to 170 cc. The same phosphoric acid was employed, and

the quantity of substance taken was arranged so as to give as near as possible the same amount of volatile acid. The standard was that of potassium acetate, which was made by dissolving 2.5 grms. in 250 cc. of water. 100 cc. was then taken by an accurate pipette in the retort, and 10 cc. phosphoric acid added, distilled to 10/11, and the quantity of acid in the distillate found by titration. Water was added to the retort after cooling, and distilled to about 5 cc. successively till any acid came over. Results in Table 5:—

TABLE 5.

Commercial Salt.	C <sub>2</sub> H <sub>3</sub> O <sub>2</sub> Per Cent. Total.	C <sub>2</sub> H <sub>3</sub> O <sub>2</sub> in 10/11 Distillate.	Per Cent. of Total in 10/11 Distillate.
Acetate potassium .....	60.22	57.00	94.65
Acetate soda.....	44.23	43.22	97.74
„ chrome (liquid) ..	10.605	10.20	96.18
„ „ (solid) ...	50.61	48.50	95.83
„ „ (2 spec.) .	48.66	46.30	95.15
„ copper (pure)....	60.36	59.70	98.90
Acetate lime liquid 24° T...	13.24	12.68	95.77
„ „ „ „	12.88	12.38	96.11
Sulpho acetate alumina: Tin red liquid 16° T...	7.32	6.97	95.22
„ „ „ „	6.79	6.48	95.43
Sulpho acetate alumina: Scarlet liquor 16° T...	5.90	5.60	94.91

The lowest of the above results is 94.61 in the case of acetate of potassium, whilst the highest, that of cupric acetate, is 98.9; the mean result is 95.99 per cent. I have constructed Table 6 on this mean result, and it shows the results in conjunction with those actually obtained.

TABLE 6.

Name of Salt.	C <sub>2</sub> H <sub>3</sub> O <sub>2</sub> from 95.99 per Cent. Factor.	C <sub>2</sub> H <sub>3</sub> O <sub>2</sub> per Cent. Actual from Column 2, Table 5.	Error.
Acetate potassium.....	59.38	60.22	+ 0.84
„ soda.....	45.03	44.23	+ 0.80
„ chrome .....	10.62	10.605	+ 0.015
„ „ (solid)	50.52	50.61	- 0.09
„ „ „	48.23	48.66	- 0.43
„ copper.....	62.19	60.36	+ 1.83
„ lime, 24° T....	13.21	13.24	- 0.03
„ „ „	12.59	12.88	+ 0.01
Sulpho acetate alumina: Tin red liquor.....	7.26	7.32	..
„ „ 2.....	6.75	6.79	- 0.04
Sulpho acetate alumina: Tin red liquor.....	5.83	5.90	- 0.07

The results above are seriously in error in two or three cases, and whether the mean factor 95.99 will be used, each one will decide himself. In the majority of cases the results come very near those calculated.

## ON A METHOD FOR DETERMINING ALKALIS IN PRESENCE OF SULPHITES.

BY JAS. GRANT AND J. B. COHEN, PH.D.

It is impossible to estimate the alkalis in presence of sulphites by ordinary titration with acid, as a certain quantity of the acid is taken up by the sulphite, SO<sub>2</sub> being evolved. Moreover, oxidising agents, such as bromine or nitric acid, to convert sulphite to sulphate, cannot, for obvious reasons, be employed.

Hydrogen peroxide, however, answers the purpose perfectly well. It has no action on the alkalis, and completely oxidises sulphites to sulphates. The method then is to add the requisite quantity of the peroxide, boil up, cool, and titrate the alkalis with acid in the usual way, using methyl orange, which is not affected by the peroxide, as indicator. Since beginning this research we have found that this method of estimation is in use in some alkali works, but believe it has never yet been published.

The effect of hydrogen peroxide on various indicators, as methyl orange, litmus, &c., was investigated, and we found that in the cold neither of these two indicators is attacked, but on prolonged boiling both are decolourised.

Secondly, experiments were made to ascertain whether the sulphite was completely oxidised. A solution of alkaline sulphite was boiled up with the requisite quantity of hydrogen peroxide and poured both hot and cold into a mixture of iodine and starch, but in neither case was the faint blue colour in the slightest degree discharged, showing the absence of sulphur dioxide.

The next point to decide was, whether carbonate or caustic are affected by the hydrogen peroxide. Different weighed samples were dissolved in water, H<sub>2</sub>O<sub>2</sub> added, and the amount of alkali determined. The results were compared with those obtained by titrating a known quantity of the same sample, but without the addition of H<sub>2</sub>O<sub>2</sub>, and proved that peroxide does not affect the estimation of carbonate or caustic.

Lastly, we endeavoured to fix, if possible, the quantity of hydrogen peroxide required to convert the whole of the sulphite into sulphate.

The method of analysis adopted is as follows:—A measured volume of hydrogen peroxide is run into a beaker together with three or four drops of methyl orange. As H<sub>2</sub>O<sub>2</sub> is always slightly acid, a small quantity of a very dilute solution ( $\frac{1}{100}$ ) of NaOH is added by means of a 1 cc. pipette till the neutral point is reached. Next the required quantity of alkaline sulphite solution is added, and boiled up at once, but gently. During the latter part of the boiling the methyl orange is bleached. The solution is cooled under the tap, a few more drops of methyl orange added, and the solution is titrated with normal hydrochloric acid solution. Both methyl orange and litmus give the same results, but the former shows the neutral point more distinctly.

The following table gives the results of several analyses:—

Solution I. consisted of 3 grms. of sodium sulphite and 7 grms. of sodium carbonate dissolved in 500 cc. of water, equivalent to .014 gm. of Na<sub>2</sub>CO<sub>3</sub> in each cc.

Solution II. contained 9.5 grms. of sodium carbonate and 0.5 gm. of sodium sulphite dissolved in 500 cc. of water, equivalent to .019 gm. of Na<sub>2</sub>CO<sub>3</sub> in each cc.

Solution III. contained 9.5 grms. of sodium sulphite and 0.5 gm. of sodium carbonate dissolved in 500 cc. of water, equivalent to .001 gm. of Na<sub>2</sub>CO<sub>3</sub> in each cc.

No. of Experi- ment.	H <sub>2</sub> O <sub>2</sub> taken.	10 cc. of Solution contained Na <sub>2</sub> CO <sub>3</sub> .	Na <sub>2</sub> CO <sub>3</sub> found.	
			After standing Half an Hour.	When boiled at once.
I. a.	Cc. 6.0	Grm. 0.14	Grm. 0.0982	Grm. 0.1033
I. b.	9.5	0.14	0.1409	0.1409
I. c.	10.0	0.14	0.1394	0.1370
I. d.	16.0	0.14	0.1405	0.1420

No. of Experiment.	$H_2O_2$ taken.	10 cc. of Solution contained $Na_2CO_3$ .	$Na_2CO_3$ found.	
			After standing Half an Hour.	When boiled at once.
I. a.	5.0	0.19	0.1893	0.1907
I. b.	10.0	0.19	0.1893	0.1900
I. c.	10.0	0.38	0.3706	0.3796
III. a.	10.0	0.01	0.01326	0.01319
III. b.	10.0	0.01	0.01326	0.01326
III. c.	15.0	0.01	0.01172	0.01195
III. d.	20.0	0.01	0.00989	0.01023

From the foregoing experiments it will be seen that—

1st. The quantity of ordinary 10 per cent.  $H_2O_2$  required depends on the percentage of sulphite present in the alkaline sulphite solution. The "caustic salts" of commerce contain about 50 per cent. of sulphite, hence it is sufficient to take 10 cc. of commercial hydrogen peroxide for every .1 gm. of salts solution, although this gives twice the theoretical quantity of oxygen required to oxidise the sulphite to sulphate. For salts containing above 50 per cent. of sulphite it is better to take double the volume of hydrogen peroxide.

2nd. It is unnecessary to let the mixture of alkaline sulphite solution and hydrogen peroxide stand half an hour before boiling up, seeing that the increase in quantity of acid required for neutralisation is almost inappreciable.

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C. H. Ridsdale.  
J. E. Stead.

Hon. Local Secretary and Treasurer:  
Dr. J. T. Dunn, The School, Gateshead.

Notices of Papers and Communications for the Meetings to be sent to the Local Secretary.

Meeting held in the College of Science, Thursday,  
January 9th, 1890.

MR. JOHN PATTINSON IN THE CHAIR.

## DISCUSSION ON MR. STEAD'S PAPER ON THE DETERMINATION OF ALUMINIUM IN IRON AND STEEL. (VOL. VIII., p. 965.)

MR. STEAD said that the method described was applicable in all cases where chromium was absent. If that element was present it would be detected (after dissolving up the sodium hydrate fusion) by the yellow colour of the chromate which would in all probability be produced. In such a case the

yellow solution must be well boiled with a few drops of sulphide of sodium or other alkaline sulphide, by which treatment the chromate is reduced, and an insoluble chromium compound produced. From this point the process may be followed as described in the paper.

Mr. THOS. CROSBY, Warrington, analyst at a works where aluminium steel had been tested, had informed him (Mr. Stead) that he had tried the method and had obtained satisfactory results.

MR. LAIDLER suggested that if the process were carried out so as to get the mixed oxides of aluminium and chromium precipitated and weighed, then the chromium might be oxidised and determined by a colorimetric process, which would be sufficiently accurate, considering the small quantities that had to be dealt with.

MR. JOHN PATTINSON stated that he had lately had to examine samples of German silver for aluminium. He was sure that without the method of Mr. Stead he would have been unable to detect any, but with it he had determined in three cases .02, .02, .03 per cent. of aluminium.

MR. RIDSDALE sent the following communication on the subject:—

A few months ago, feeling that a thoroughly good method for estimating aluminium in iron and steel was very much needed, I decided to investigate the matter, and though I have not had time to go into it as far as I had originally intended, I may perhaps be permitted to give a few results for what they are worth.

My first wish was to obtain figures showing clearly the value, or rather the absence of value, of three or four existing methods for separation of  $Al_2O_3$  from  $Fe_2O_3$ , &c.

The amount of Al present in a sample of pure ammonia alum was accurately determined, and this was added in such quantities to solutions of various weights of steel drillings as to give the equivalent of definite percentages of Al.

*First Method tried.*—Precipitates of  $Fe_2O_3$  and  $Al_2O_3$ , as obtained with ammonia and ammonium acetate from HCl solutions oxidised with  $HNO_3$ , dissolved in HCl and titrated with  $K_2Cr_2O_7$  (standard), Al being estimated by difference.

	Per Cent.	Error Per Cent.
Percentage of Al which should have been found	1.57	
Shown by this method	{ 2.49 2.15 2.26	{ .99 .63 .76

Manifestly such a method would be utterly inapplicable for lower percentages.

*Second Method tried.*—Fusion of the above precipitate with "pure"  $KHO$  or  $NaHO$  (in all cases a blank being done) and precipitating, after acidifying with HCl, by  $NH_3$  and  $NH_4C_2H_3O_2$ .

	Per Cent.	Error Per Cent.
Percentage of Al which should have been found	.1	
Shown by this method	{ .105 .16 .18 .05	{ .005 .06 .08 .05

also—

	Per Cent.	Error Per Cent.
Percentage of Al which should have been found	1.50	
Shown by this method	{ .90 1.18 .78	{ .60 .82 .72

The barium hydrate method I have tried on former occasions and failed to get reliable results, but had not tried it with this set of experiments.

At this juncture Mr. Stead told me of his method, and I at once tried it on steels with various quantities of Al added:—

	I.	II.	III.
	Per Cent.	Per Cent.	Per Cent.
Percentage of Al should be	.05	.25	.50
Actually found, Stead's method	{ .044 .064	.22	.495
Error	.005 to .014	.03	.005

From the above results I feel sure that with a little practice almost absolutely correct results can be obtained, and at any rate incomparably better than the other methods cited, which though they answer fairly well when the amount of iron present is relatively small, are worthless when the amount of iron is from 100 to 1,000 times as great as the Al present.

I should like to point out that I think it a distinct advantage to precipitate the Al as phosphate, because some of the P present in steel and iron is sure to be found in any  $Al_2O_3$  precipitate obtained, and the precipitating of the whole of the Al as phosphate therefore simplifies matters greatly.

#### DISCUSSION ON DR. LUNGE'S PAPER ON THE DETERMINATION OF SULPHUR IN PYRITES. (VOL. VIII., p. 966.)

MR. STEAD suggested that the filtration and washing in Dr. Lunge's method might be avoided if the precipitation by ammonia were done in a measuring flask, the liquid made up to a definite volume, and an aliquot portion of the clear liquid, after settling, used for precipitation by barium chloride. If allowance were made for the volume of the precipitate this might be a perfectly accurate method.

MR. WARNE thought that any of the ordinary methods gave accurate results if performed with sufficient care. He had had considerable experience both with the ordinary method of oxidation by nitric acid, solution in hydrochloric acid, and direct precipitation in the acid solution, and also with Teschemacher and Smith's volumetric method; and he had found that these, carefully carried out, gave results concordant with each other and with those got by other methods.

MR. LAYDLER had also had considerable experience with Teschemacher and Smith's method, and found it gave results agreeing with those got both by the fusion methods and by Dr. Lunge's method. His experience of the fusion method was that it very rapidly destroyed the platinum crucibles used.

MR. JOHN PATTINSON had had tests made on the same sample of pyrites to compare the methods of Teschemacher and Smith, Fresenius, and Lunge. The results were:—

Lunge.	Fresenius.	Teschemacher and Smith.
49.20	49.18	49.25
49.22	49.15	49.30

With a view of testing the absolute accuracy of Lunge's method, he had dissolved and oxidised 5 grains iron wire and added 20 grains sodium sulphate; the mixture was then treated by Lunge's method as though it were the solution of a sample of pyrites. Two determinations yielded sulphur corresponding to 20.05 and 20.00 grains of sodium sulphate respectively, showing the method to be very accurate. In point of convenience there was no comparison between Lunge's method and that of Fresenius, which involved at least two evaporations with hydrochloric acid, a second treatment of the iron oxide with caustic soda, and a passage of carbonic acid gas through the solution to get rid of any lead.

#### ON THE GAS-VOLUMETRIC ANALYSES OF POTASSIUM PERMANGANATE, CHLORIDE OF LIME AND MANGANESE PEROXIDE.

BY G. LUNGE.

SOME years ago a paper by Mr. A. H. Allen drew my attention to a great number of new applications of the apparatus now well known by the name of the "Nitrometer." In a paper published in the "Chemische Industrie" 1885, 161 et seq. (very briefly abstracted in the Journal of the Society of Chemical Industry, 1885, 495), I described the manifold uses to which the nitrometer can be put as an apparatus for gas analysis proper, as an absorptiometer, and especially for gas-volumetric analyses. To fit it for the last-mentioned object, I added to it a flask, provided with an inner tube fused on to its bottom, and suspended from the side tube of the nitrometer, as shown in Fig. 1, which at the same time exhibits the Greiner and Friedrich's patent tap (a similar one is known as the Geissler-Miescher tap, and is

Fig. 1.

Fig. 2.

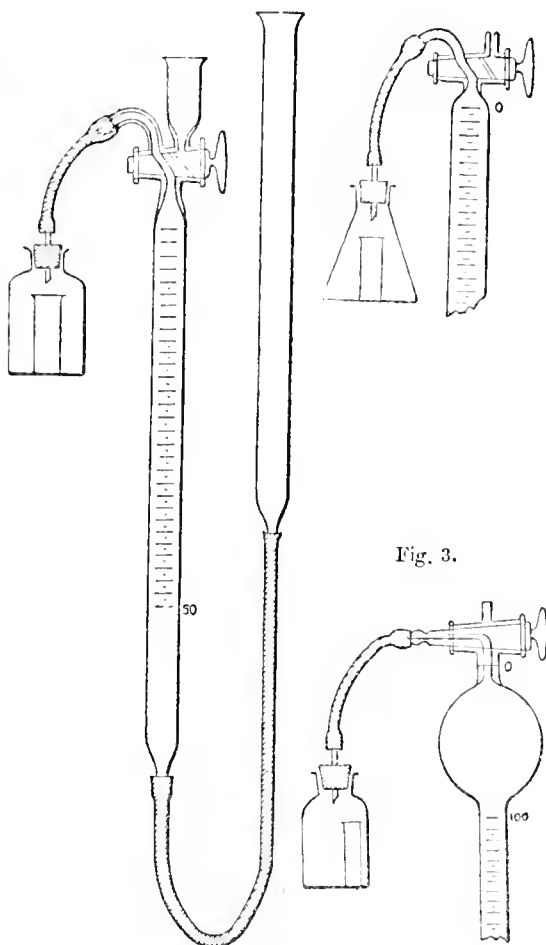


Fig. 3.

applied to the nitrometers supplied by the firm of Dr. H. Geissler at Bonn). This shows how any ordinary nitrometer, such as are now found in most chemical laboratories, can be applied to the afore-mentioned uses. Where, however, the methods concerned are to be employed not merely occasionally, but regularly, it will be preferable to get a nitrometer specially adapted to this use, of which Figs. 2 and 3 show various forms. They have no cup at the



top, which is quite unnecessary for this purpose, but merely a short outlet-tube for air. Fig. 2 shows an instrument provided with one of the new patent taps, which are certainly very handy, and cause a much smaller number of spoiled tests than the ordinary three-way tap, as shown in Fig. 3, which at the same time exhibits the form of nitrometer intended for large quantities of gas, the upper part being widened into a bulb, below which the graduation begins with either 60 or 100 cc., ending at 100 or 140 cc., respectively. There are also various shapes of flasks shown in these instruments, but it is unnecessary to say that these, as well as the bulb arrangements, can be applied to any other form of the instrument. The nitrometers used for gas-volumetric analyses are best graduated in such manner that the zero point is about a centimetre below the tap, whilst ordinary nitrometers have their zero point at the tap itself. I will say at once that for all the cases to be mentioned in the following it is quite unnecessary to employ mercury for filling the instruments, since identical results are obtained with ordinary tap water; but it is decidedly advisable to place this instrument, like any ordinary nitrometer or any other apparatus in which gases are to be measured, in a room where there are as few changes of temperature by cold draughts or gas-burners and so forth as possible.

I shall in this place only treat of those of the manifold uses of the nitrometer, in which *hydrogen peroxide* is used as the principal reagent, and although the methods I shall mention have been previously mentioned by me, both in the above quoted paper and *passim* in the *Berliner Berichte*, &c., I shall now, for the first time, give such data as will amount to a conclusive proof of the correctness of the results to be obtained by this analytical method.

I shall commence with one of the most "elegant" of these methods, and at the same time that which possesses the greatest attainable degree of accuracy, namely the *standardising of potassium permanganate without scales, weights, or substances of accurately known composition*. In this case it is necessary to work with *acid* solutions containing a large excess of sulphuric acid. I had frequently employed this method concurrently with the iron method, but in order to make sure of the degree of accuracy obtainable by this method, I caused Dr. Lengfeld to make a number of specially careful estimations, for which all graduated vessels were calibrated and the materials employed were analysed beforehand. Only the hydrogen peroxide, accidentally, was not tested for its strength, but fortunately this is of no moment, as in this case even a large excess of the reagent can be employed without any difference in the results.

The potassium permanganate employed was made from pure crystals, and was as nearly seminormal as possible, i.e., each cc. corresponding to 0.004 gram oxygen. It was standardised: 1st, with iron wire which was found by analysis to contain 99.9 per cent. of metallic iron; 2nd, with crystallised oxalic acid, purified according to Cl. Winkler's prescription and dried up to constant weight in a Victor Meyer's air-bath at 56°; 3rd, with ordinary commercial peroxide of hydrogen. The results were as follows:—

#### A. Standardising with iron wire.

cc.	Gram. Oxygen.
1 permanganate solution yields.....	0.004002
1 " " " .....	0.003998
1 " " " .....	0.003996
Average .....	0.003999

#### B. Standardising with oxalic acid.

cc.	Gram. Oxygen.
1 permanganate yields .....	0.003998
1 " " " .....	0.003995
Average .....	0.003997

#### C. Standardising with hydrogen peroxide in the nitrometer.

(a.) 10 cc. permanganate + 15 cc. dilute sulphuric acid (1:5) and 15 cc.  $\text{H}_2\text{O}_2$ , shaken up, allowed to stand 10 minutes, shaken again and read off after 15 minutes.

Volume of Oxygen.	Temperature.	Barometer.	The same Corrected.	Volume of Gas at 0° and 760 mm.	1 cc. Permanganate yields, Gram. Oxygen.
Cc. 61.6	°C. 12	Mm. 722	Mm. 711	55.13	0.003951
61.4	13	722	709	54.70	0.003920
61.4	13	730	717	55.28	0.003991
60.2	12	729	718	54.47	0.003904
Average .....					0.003911

(b.) Conditions as before, but 30 cc. sulphuric acid used in lieu of 15 cc.

Cc.	°C.	Mm.	Mm.		
62.3	12	721	712	55.92	0.004007
62.5	13	724	711	55.82	0.004000
62.3	12.5	724	712	55.82	0.004000
Average .....					0.004002

If we exclude series C. (a.) in which, as is evident at once from the considerable variation of the results, the proper conditions had not been attained, the quantity of acid not being large enough, we find as averages of the three methods of standardising the same permanganate solution:—

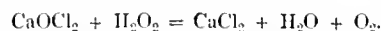
- A. With iron wire 1 cc. = 0.003999 gram oxygen.  
 B.  $\alpha$ -oxalic acid 1 " = 0.003997 " "  
 C. With  $\text{H}_2\text{O}_2$  1 " = 0.004002 " "

Of methods A. and B. the former possesses the greater degree of reliability, since it is extremely difficult to prepare absolutely pure and dry crystallised oxalic acid.

The averages of A. and C. agree better than the single experiments with iron wire do among one another. We cannot but infer that *standardising a solution of potassium permanganate with hydrogen peroxide in the nitrometer, when observing the prescribed precautions, is one of the most accurate known methods for this purpose, and withal possesses the great advantage that it is carried out within an extremely short time, without requiring a fundamental substance of accurately known composition.*

Anybody who has worked in this field will know without special instructions that no gas-analytical operation can yield accurate results unless the graduation of the instruments has been checked, and unless the apparatus is protected against changes of temperature from draughts, gas-burners, and so forth. This of course equally applies to the following methods.

We shall now describe the estimation of *chloride of lime* (bleaching powder or bleach liquor) by means of hydrogen peroxide. The reaction is here—



It is not necessary to know the exact composition of the hydrogen peroxide, but as it is desirable not to employ too large an excess of it in this case, it is best to estimate its percentage by a preliminary test occupying but a few minutes, in which a certain volume of  $\text{H}_2\text{O}_2$  is decomposed by an excess of bleach solution (the inverse of the titration of the latter). This need be done only quite roughly. For the analysis of chloride of lime the hydrogen peroxide must be diluted before use so as not to give out more than 7 cc. of oxygen per cc., and it must be made alkaline by means of caustic soda solution up to the point where a flocculent precipitate appears. The alkaline reaction ought to be quite

distinct, but any *great* excess of alkali should be avoided. It is not necessary to shake much, and the reading ought to be made quickly, say five minutes after mixing the liquids, otherwise the results will be too high owing to the gradual evolution of more oxygen from the alkaline liquid. It might be thought that muddy solutions, such as are regularly employed in testing commercial bleaching powder, would yield less reliable results, the solid matter favouring the evolution of oxygen from  $\text{H}_2\text{O}_2$  otherwise than through the action of  $\text{CaOCl}_2$ ; but this is not so; muddy solutions can be tested by the nitrometer just as well as clear bleach liquors, provided the time of five minutes is not exceeded. As the reaction does not produce a sensible change of temperature, that time will quite suffice, provided that the operator has avoided raising the temperature of the flask in manipulating it, which he can do by handling it always by the neck with his thumb and forefinger only.

It may be as well to give here a general description of the mode of procedure for manipulating gas-volumetric analysis with the nitrometer, common to all analyses according to this method. Fill the nitrometer with water or mercury by raising the level tube till the level of the liquid in the graduated tube is at zero (in the case of instruments bearing the zero-mark a little below the tap, as in Figs. 2 and 3), or at 1.0 cc. (in the case of ordinary nitrometers, beginning their graduation at the tap itself). It is unnecessary to say that in the latter case all readings must be diminished by 1 cc. Close the glass tap. Put the substance to be tested into the outer space of the flask, together with any other reagent apart from the  $\text{H}_2\text{O}_2$  (in the case of bleaching-powder nothing but the bleach liquor, in that of potassium permanganate the 30 cc. of sulphuric acid, &c.). Now put the  $\text{H}_2\text{O}_2$  into the inner tube of the flask, after having, in the case of testing for chlorine, made it alkaline in the previously described way. Put the india-rubber cork, still hanging from the tap, on to the flask, without warming the latter as above described. As this produces a compression of the air within the flask, remove this by taking out the key of the tap in Figs. 1 or 2, or, in Fig. 3, turning it for a moment so as to communicate with the short outlet tube. Now turn the tap back, mix the liquids by inclining the flask, shake up and allow the action to proceed. As the gas passes over into the graduated tube, lower the level tube, so as to produce no undue pressure; at last bring the liquid in both tubes to an exact level and read off.

In our special case all the oxygen of the chloride of lime is given off, together with exactly as much oxygen of the  $\text{H}_2\text{O}_2$ . The total is just equal to the volume of chlorine gas which would be given off by the chloride of lime, and thus immediately represents the French or Gay-Lussac chlorometric degrees, of course after reducing the volume to  $0^\circ$  and 760 mm. pressure. (The reading of the barometer must be corrected by deducting the tension of aqueous vapour for the temperature observed as well as the expansion of mercury, according to the tables found everywhere). These reductions can be easily performed by the tables contained in the "Alkali-Makers' Pocket-book" (pp. 28 to 39), which I had calculated a number of years ago, just in order to facilitate the use of the nitrometer. Other tables are found in the same place, pp. 40 to 42, and in Winkler-Lunge's Handbook of Technical Gas Analyses, p. 114 et seq. All tables can be dispensed with, as well as the reading of the thermometer and barometer, when using the reduction instrument, invented at the same time by Cl. Winkler and myself, of which I have recently published a more convenient modification (Chemiker Zeitung, 1888, 821), an instrument no doubt procurable by every English dealer in chemical apparatus.

In order to find the percentage of available chlorine by weight, that is, the English chlorometrical degrees, it should be borne in mind that every cc. of gas evolved, reduced to  $0^\circ$  and 760 mm., represents 0.003167 grm. of chlorine. Hence, if the quantity of bleach employed is = 1 grm. (for instance, by dissolving 20 grms. in 500 cc. of water, and employing 25 cc. of the solution for each test), each cc. of gas is = 0.3167 per cent. of available chlorine in the bleach. This involves the use of a bulb nitrometer holding 140 cc. If only a 50 cc. instrument is at hand, it will be necessary to take, say, 5 cc. of the first-mentioned bleach solution, in

which case every cc. of gas represents  $5 \times 0.3167 = 1.58$  per cent. of chlorine. The most convenient way is to dissolve 7.917 grms. of bleach in 250 cc. of water, and employing 10 cc. of the solution for each test, when each cc. of oxygen evolved will directly indicate 1 per cent. of available chlorine, and a 50 cc. nitrometer should be used.

The following comparative tests were made by two of my students, Messieurs Schubert and Obregia, on the one hand with the nitrometer, on the other hand with Penot's method (sodium arsenite and potassium iodide as indicator). The work was done as carefully as possible, all instruments being again controlled for the accuracy of their graduation. The results were as follows, all calculated as per cent. available chlorine in 100 solid bleaching powder.

A. Muddy bleach solution. Hydrogen peroxide distinctly alkaline.

(OBREGIA.)

Nitrometer.	Penot.
31.77	31.50
31.73	31.46
31.70	..
31.67	..
Average 31.72	31.48

B. Muddy solution.  $\text{H}_2\text{O}_2$  more strongly alkaline.

(OBREGIA.)

Nitrometer.	Penot.
31.65	31.50
31.67	31.46
Average 31.66	31.48

C. Muddy solution. Hydrogen peroxide made alkaline to the point where a flocculent precipitate begins to form (same treatment as in all following series).

(SCHUBERT.)

Nitrometer.	Penot.
36.67	36.60
36.63	36.72
Average 36.65	36.70

D. Muddy solution. 4 cc. hydrogen peroxide containing 5.4 per cent.  $\text{H}_2\text{O}_2$ .

(SCHUBERT.)

Nitrometer.	Penot.
29.89	29.60
29.87	
29.79	
29.73	
Average 29.82	

E. Muddy solution. 5 cc. of the same  $\text{H}_2\text{O}_2$  diluted with 10 cc. water.

(SCHUBERT.)

Nitrometer.	Penot.
30.45	30.27
30.38	
30.42	
30.46	
Average 30.43	

F. Filtered, clear solution. 5 cc.  $\text{H}_2\text{O}_2$  + 10 cc. water:—

Nitrometer.	Penot.
30.23	30.04
30.19	
Average 30.21	

G. Filtered solution. 7 cc. of hydrogen peroxide (rather weaker) + 10 cc. water (= 1.3 per cent.  $\text{H}_2\text{O}_2$ ).

## (SCHUBERT.)

Nitrometer.	Penot.
29.87	29.60
29.75	
29.79	
29.78	
Average 29.78	

II. Same solution. 10 cc. of same  $\text{H}_2\text{O}_2$  + 15 water (= 1.44 per cent.  $\text{H}_2\text{O}_2$ ).

## (SCHUBERT.)

Nitrometer.	Penot.
29.74	29.60
29.70	
Average 29.72	

I. Influence of different liquids employed for filling the nitrometer, under the same conditions in every other respect.

## (SCHUBERT.)

Mercury.	Water.
29.64	29.64
29.64	29.63
Average 29.65	29.65

N.B.—A large series of similar comparative tests, carried out by other students, yielded precisely the same results, viz., that the estimations are just as accurate when water is used in the nitrometer as when using mercury. The meniscus must, of course, be treated in both cases equally for zero and for the final reading.

The above-quoted results show that the nitrometric method yields quite as concordant results in the testing of bleach as Penot's method, and that both methods are very nearly identical in their results. Still, there is always a little more found in the nitrometric method; but when working in the proper way, with no very large excess of  $\text{H}_2\text{O}_2$  (Series II. proves that a moderate excess does no harm), and not allowing to stand too long, the deviation does not exceed 0.2 per cent., and this amount might be deducted as a constant from the nitrometric results, to bring them into entire concordance with those obtained by Penot's method.

We may, then, conclude that the nitrometer offers an excellent method for checking the results obtained by Penot's method, and for avoiding any errors creeping in by wrong standard solutions. The fact that no standard solution whatever is required in my process makes it specially recommendable for the users of chloride of lime—for instance, bleachers, paper-makers, manufacturers of chloroform—as needs no further explanation.

The analysis of *manganese ore*, or of *Weldon mud*, by means of the nitrometer, offers the great advantage of a saving of time as well as of all standard solutions. In this case the reaction must take place in an *acid* medium. The finely-powdered ores or the Weldon mud are put into the outside space of the flask, and are first treated with dilute sulphuric acid, in order to decompose any carbonates present. Then the hydrogen peroxide is put into the inner tube, the flask is attached to the nitrometer, the liquids are mixed and shaken up, till the colour of the residue shows that the decomposition is complete. With Weldon mud it is instantaneous.

The reaction is—



Half the oxygen evolved comes from the manganese peroxide, hence each cc. of gas corresponds to 0.003897 grm. of  $\text{MnO}_2$ . If 0.3897 grm. manganese ore have been weighed out for each test, the number of cc. of gas evolved indicates immediately the percentage of  $\text{MnO}_2$  in the ore. Comparative tests between this and the ordinary iron-sulphate process, made by Mr. Biedermann, showed—

Nitrometer.	Iron Process.
62.2	62.1 per cent.
62.4	62.3 "
Average 62.3	62.2 "

The following tests were made by Mr. Ruhoff with a very inferior description of ore, unfit for chlorine making, but employed in the manufacture of steel: 0.5 grm. of ore was employed for each test.

Nitrometer filled with Mercury.	Nitrometer filled with Water.
Cc.	Cc.
36.6 oxygen.	36.4
36.6	36.6
36.9	36.6
36.7	..
37.0	..
Average 36.76	36.53

$$= 28.65 \text{ per cent. MnO}_2 = 28.47 \text{ per cent. MnO}_2.$$

The iron process yielded 28.60 per cent.  $\text{MnO}_2$ . For commercial purposes the iron process will, of course, retain its supremacy, but the nitrometric method is a good check upon the former, and as it takes much less time, it is far more convenient for quick estimations in every-day work.

## Nottingham Section.

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SESSION 1889-90.

Notices of Papers and Communications for the Meetings to be sent to the Local Secretary.

Meeting held in the Chemical Lecture Theatre, University College, on November 6th, 1889.

SIR JOHN TURNEY IN THE CHAIR.

THE CHAIRMAN, in opening the proceedings, said it was not a very uncommon thing for men to find themselves in places which they were totally unfitted to fill, and that was the case with himself that evening. When he was asked to become their Chairman he told Dr. Clowes that he knew little or nothing of theoretical and practical chemistry. Unfortunately it had not been his lot to study this important science, and therefore on that occasion he could do little more than preside. As a large manufacturer in the town he had often felt the want of chemical knowledge, and his firm had been stupid enough, like many other people very likely, to go on for nearly 20 years making various articles by rule of thumb. But some eight or ten years ago he did turn his attention to chemistry, to the extent of getting around him men who theoretically and practically understood the subject, and the firm had derived considerable advantage from this step. If this nation desired to maintain its position against foreign competitors we should have to make considerable advances in education. Those advances had begun, and if they were maintained and increased, for there was much to be done yet, the benefit to the country would be certain and great.

# THE DETERMINATION OF SULPHUR IN IRON AND STEEL.

BY L. ARCHBUTT, F.I.C.

THE process of oxidising iron or steel by aqua regia, evaporating to dryness to separate silica, redissolving in hydrochloric acid, filtering, and precipitating with barium chloride, is, when properly carried out, if not the most rapid, certainly one of the most accurate methods of determining the sulphur. The object of this paper is to call attention to certain important fallacies which have crept into the published descriptions of the method, and to describe the simplest and best way of working according to my own experiments.

In a paper communicated to the Chemical News in 1888 (Vol. 58, p. 41), Messrs. Arnold and Hardy stated that the excess of hydrochloric acid used in redissolving after separation of silica is evaporated off "until only sufficient to retain the iron in clear solution is present. The concentrated liquid is then diluted, and the insoluble residue is removed by filtration. The filtrate is next largely diluted and the sulphur is precipitated by means of barium chloride." They then proceeded to bring five objections against the process, of which two are of no importance, and the remaining three may be summarised thus:—1. That excess of acid causes low results; 2. That about two days are needed for an estimation, 24 hours being usually allowed for the precipitation alone; and 3. That the barium sulphate is sometimes difficult to filter, and that it is occasionally impure with iron, the removal of which either involves loss of precipitate or a serious loss of time—all of which objections I will show to be misleading or exaggerated.

Morgan (loc. cit. p. 63), in discussing the above paper, admits that the results are too low if the solution contain excess of acid, and states that the solution when freed from this excess should be diluted to a large volume before precipitation. He also admits that the process takes a long time, but he points out that the results obtained are unsurpassed for accuracy, and that accuracy ought not to be sacrificed for a saving of time.

Blair (The Chemical Analysis of Iron, 1888) says, "redissolve in as little HCl as possible, dilute and filter. Heat the filtrate to boiling and precipitate with BaCl<sub>2</sub>." How far to dilute is not stated, and to heat to boiling would be a risky proceeding unless there were plenty of free acid present. To this free acid there would be no objection, provided the solution be not diluted, but if it be diluted it is of the greatest importance that it should be highly neutral.

These quotations confirm my belief that it is a very general practice with those who use the aqua regia process to make the ferric solution as neutral as possible, and to dilute it considerably before adding the barium chloride. A steel was once sent to me which when tested by this method appeared to contain no sulphur. The solution was so neutral as to be quite dark in colour, but barium chloride produced no precipitate even after standing several days, although the liquid was agitated and passed through a filter paper in order to disturb the equilibrium; yet it was evident there must be some error, because the hydrochloric acid used contained traces of SO<sub>3</sub>. The analysis was therefore repeated in order to try the effect of adding BaCl<sub>2</sub> to the solution without diluting. In this experiment the solution was prepared from 5 grms. of steel and did not exceed 70 cc. in volume; the result was a precipitate of 0.0160 gm. BaSO<sub>4</sub>, which, after deducting 0.0055 gm. due to impurity in the acid, was equivalent to 0.029 per cent. of sulphur in the steel. In addition to increasing the accuracy of the results the reduction in volume of the solution effected a great saving of time, since precipitation was much more rapidly effected and there was less liquid to filter. Accordingly, ever since this experiment was made, several years ago, I have ceased to dilute the solution. In order, however, to obtain some further figures for comparison, the following experiments were recently made. A steel was taken which contained 1.25 per cent. of sulphur, and a highly neutral solution was prepared containing 20 grms. of the sample. This solution was divided, by weighing, into four equal portions, and one portion was undiluted, while the others were diluted respectively to 250 cc., 500 cc., and 750 cc. To each

solution there was added 5 cc. of a 10 per cent. solution of BaCl<sub>2</sub>.2OH<sub>2</sub>. The solutions were all precipitated at 4.30 p.m. and the two most concentrated were filtered next morning; but it was found necessary to allow the others to stand much longer, as precipitation was so slow. The filtrates gave no further precipitate after standing several days.

Volume of Solution.	Weight of BaSO <sub>4</sub> obtained.	= Per Cent. of Sulphur in the Steel.
Cc. 63	Grm. 0.0155	1.25
250	0.0115	1.14
500	0.0330	0.91
750	0.0270	0.74

It will be observed that the quantity of barium salt added was more than ten times the quantity theoretically required, and no more was added to the filtrates. As it has since appeared to be of interest to ascertain whether by adding a still greater relative mass of barium salt complete precipitation could be effected even in a highly diluted solution, another series of four experiments was made, using however a different steel as the supply of the other was exhausted. The steel used this time contained 1.14 per cent. of sulphur, and solutions of it were prepared as before. It is unnecessary to record the details of these experiments, suffice it to say that it was found by adding a sufficient excess of barium salt, and allowing long enough for precipitation, that very nearly all the BaSO<sub>4</sub> could be caused to precipitate even in a highly dilute solution. In each case the final filtrates remained bright and clear after standing several days with an additional 10 cc. of barium chloride solution.

Volume of Solution.	Weight of BaSO <sub>4</sub> obtained.	= Per Cent. of Sulphur in the Steel.
Cc. 70	Grm. 0.0116	1.14
250	0.0386	1.06
500	0.0391	1.18
750	0.0578	1.04

But the experiments clearly prove that there is no necessity for diluting the solution, and that dilution leads to a loss both of accuracy and of time.

Precipitation being therefore effected in a concentrated solution, it became of interest to determine whether free acid is prejudicial. Seven different steels were made the subject of these experiments. Five grms. of each having been dissolved and evaporated as usual to render silica insoluble, the dry residues were dissolved in HCl, and the excess of acid was evaporated off as completely as possible. After adding a little water and filtering, the filtrates were precipitated by BaCl<sub>2</sub>. Side by side with these, duplicate quantities of the same steels were treated in exactly the same way, except that before adding the BaCl<sub>2</sub> each concentrated solution was mixed with 5 cc. of hydrochloric acid (1.10). The results obtained are shown in the following table:—

	Neutral Solution. (60—70 cc.)		Acid Solution. (60—70 cc.)	
	BaSO <sub>4</sub> obtained.	Sulphur.	BaSO <sub>4</sub> obtained.	Sulphur.
		Per Cent.		Per Cent.
1	0.0530	1.46	0.0515	1.42
2	0.0414	1.11	0.0413	1.11
3	0.0415	1.11	0.0415	1.14
4	0.0337	0.93	0.0330	0.91
5	0.0298	0.82	0.0315	0.87
6	0.0245	0.67	0.0245	0.67
7	0.0215	0.59	0.0205	0.55

All the filtrates were tested with more barium solution, but no more precipitate came down. It appears from these experiments that the presence of free hydrochloric acid is not prejudicial to accuracy in a concentrated solution. It is in fact advantageous, since the solution often filters better when acid, precipitation appears to be rather more rapid, and the precipitate when ignited is a purer white. The results obtained agree with, and are an extension of, those recorded many years ago by Noad (Journ. Chem. Soc. 1857, 15), who showed that in decomposing potassium sulphate with barium chloride in a liquid the total volume of which did not exceed 65 cc., the theoretical weight of  $\text{BaSO}_4$  was obtained, whatever the proportion of free hydrochloric acid (1.10) present. These experiments of Noad seem to have been almost forgotten, and it is curious that Fresenius makes no reference to them.

Having thus shown that precipitation should be effected in a concentrated solution of the iron, and that the presence of free hydrochloric acid is beneficial, it remained to determine the length of time necessary for complete precipitation. For this purpose the steel numbered 1 in the last series of experiments was taken, and two separate quantities of 5 grm. were treated as already described, the solution being made acid by the addition of 5 cc. of  $\text{HCl}$  (1.10). Both solutions were heated to about  $200^\circ \text{F}$ ., and to each 5 cc. of the 10 per cent. barium chloride solution were added. One was filtered after standing two hours, and the other after standing three hours, and these were the results:—

	Acid Solution. (60–70 cc.)	
	$\text{BaSO}_4$ obtained.	Sulphur.
Stood two hours .....	*0535	Per Cent. *117
Stood three hours.....	*0519	*113

The filtrates were treated with an additional 5 cc. of the barium chloride solution, and were allowed to stand for 24 hours; only a mere trace of precipitate came down in either filtrate. Precipitation is therefore practically complete in two hours, and possibly in less, but I have not thought it worth while to pursue the subject further, because I generally find it convenient to add the  $\text{BaCl}_2$  over-night and filter next morning.

The following is a description of the method for determining silicon and sulphur:—5 grms. of steel or iron are added to a previously heated mixture of 40 cc. hydrochloric acid (1.10) and 20 cc. nitric acid (1.42) contained in a large lipped beaker. After effervescence has ceased a few crystals of  $\text{KClO}_3$  are added, and the solution is evaporated to complete dryness and heated on the hot plate for at least half an hour, turning the beaker round occasionally so as to dry every part equally. After cooling, the residue is heated with 20 cc. more hydrochloric acid, and is again thoroughly dried. Forty cc. of  $\text{HCl}$  are then added and heated until a clear solution is obtained. The liquid is then cautiously evaporated until a skin begins to form, when the beaker is removed from the plate, 5 cc. of  $\text{HCl}$  are added, and boiling hot water is blown in from a wash-bottle until the hot liquid is about doubled in volume. It is then at once poured on to a 10 cm. filter, which it just about fills, the beaker is rinsed two or three times with a very little more hot water, and the filter is then covered up and allowed to drain, which does not take many minutes. The filter is washed about twice with cold water and removed to another glass, where the rest of the silica remaining in the beaker is rinsed on to it and washed for the silicon estimation. The dark coloured filtrate containing the  $\text{SO}_3$  occupies less than 70 cc. in bulk, is treated with 5 cc. of a 10 per cent. solution of  $\text{BaCl}_2 \cdot 2\text{OH}_2$ , and is either left over-night in the cold, or it is heated and filtered as soon as the precipitate has completely fallen (two hours or less). The solution is filtered, with or without suction, through a paper which has been previously washed with  $\text{HCl}$  (which greatly hastens filtration), and the precipitate is washed with cold water, dried, ignited, and weighed. From the weight is deducted that of the  $\text{BaSO}_4$  obtained in a

blank experiment with the same quantities of  $\text{HCl}$  and  $\text{HNO}_3$ . The precipitate is seldom, if ever, contaminated with iron, though the ash of the paper is generally pink. If there be any excess of iron present it is easily estimated by heating the precipitate with  $\text{HCl}$ , diluting, and making a colour titration with  $\text{KCN}$ .

Some experiments, which are recorded in the following table, were made for the purpose of comparing the results obtained by this method with those obtained by the evolution method. When the  $\text{H}_2\text{S}$  was absorbed in  $\text{HCl}$  and bromine the results were slightly lower, and the same tendency was observed when caustic soda was the absorbent. Alkaline lead solution was next tried, which is a perfect absorbent for  $\text{H}_2\text{S}$ , but the subsequent treatment of the precipitated  $\text{PbS}$  is troublesome. It is difficult to thoroughly wash the precipitate and filter paper without the use of acid, which accounts for the first two results being rather high; and when even very dilute acid is used in washing, traces of  $\text{PbS}$  are dissolved, judging by indications observed in the course of the third experiment with lead. On the other hand if the precipitate be dissolved up in  $\text{HCl}$  and  $\text{KClO}_3$  low results may be caused by the formation of insoluble  $\text{PbSO}_4$ . Ammoniacal cadmium chloride solution gave the best results, but in this case I noticed that in dissolving the  $\text{CdS}$  in  $\text{HCl}$  and bromine minute particles of sulphur were apt to escape oxidation. This may account for the first cadmium result being slightly low. In the next three experiments the few undissolved particles were treated with fuming  $\text{HNO}_3$  or  $\text{HCl}$  and  $\text{KClO}_3$ . The results of the last three experiments with cadmium solution, and the mean of the whole series shows that whether the steel or iron be oxidised direct with aqua regia, or whether the  $\text{S}$  be evolved as  $\text{H}_2\text{S}$ , practically the same results are obtained, which supports the conclusion that a concentrated solution of ferric chloride, whether nearly neutral or strongly acid, has no appreciable solvent action upon barium sulphate in the presence of barium chloride.

Absorbent.	The Steel used contained, when tested by the Aqua Regia Method	
	Sulphur *125 Per Cent.	Sulphur *111 Per Cent.
$\text{HCl}$ (1.10) saturated with bromine .....	{ *104 *114	{ *106 *105
Pure $\text{NaOH}$ containing $\text{NaBrO}_3$ .....	*108	{ *114 *106 *108
Alkaline lead solution: The $\text{PbS}$ was washed with soda and then water. Weighed as $\text{PbSO}_4$ .....	**	{ *123 *126
The $\text{PbS}$ was washed with soda and then water, then twice with highly dilute nitric acid (2 vols. acid of 1.2 sp. gr. in 100 vols.), then with water. Weighed as $\text{PbSO}_4$ .....	..	*103
The $\text{PbS}$ was oxidised by $\text{HCl}$ and $\text{KClO}_3$ and the solution was precipitated by $\text{BaCl}_2$ .....	..	*103
Ammoniacal cadmium chloride: The $\text{CdS}$ was dissolved in $\text{HCl}$ and $\text{Br}$ and the solution precipitated by $\text{BaCl}_2$ ..	..	{ *103 *106 *114 *115
Mean of all the experiments.....	*109	*111

#### DISCUSSION.

Mr. J. B. COLEMAN said he had little experience of the direct oxidation method, but had noticed that in precipitating sulphates in hot solutions containing a large amount of iron, the iron came down with, and coloured the precipitate, and it needed an acid to get rid of that iron. He had used the absorption methods, and had tried several absorption agents:—Hydrochloric acid and bromine; alkaline lead solution, and ammoniacal cadmium chloride, and found that

both the latter absorbed perfectly. Bromine was somewhat more difficult and unpleasant. If the direct method were so exact as Mr. Archbutt's paper seemed to show, he should be inclined to try it.

With regard to the effect of dilution upon the precipitation of the sulphate, he had noticed quite recently that in estimating sulphates in spring water, different students obtained varying results with the same water, so that he had considered the water to be very changeable. More recent experiments by students on the effect of greater or less concentration of such waters had led him to believe that in dilute solutions the precipitation of the sulphate was not so complete in a given time as in the more concentrated ones, and he was pleased that a probable explanation of it had been given. He would like to know if Mr. Archbutt had ever found the iron coming down with the barium sulphate?

Mr. WHITELEY agreed with Mr. Coleman as to the colouration of barium sulphate by iron, and had noticed (in connexion with mineral water analysis) the difficulty of getting it to precipitate in very dilute solutions. He had looked upon it as a question of mass, although it might be due to the slight solubility of barium sulphate in certain salt solutions.

Mr. ARCHBUTT, in reply, said he had never found any iron come down at all in cold solution. All he had noticed was that it seemed impossible to wash the filter paper perfectly clean. The ash turned pink, although the precipitate was white. But in a nearly neutral solution the sulphate must not be precipitated whilst hot, or iron would come down. With regard to a comparison of the two methods—absorption and direct oxidation—he had only made the two comparisons which he had mentioned, and the results were nearly the same by both methods. If he were more experienced in the absorption method he might, perhaps, get results exactly the same.

*Meeting held at Bingham's Restaurant, Nottingham,  
on Wednesday, December 11th, 1889.*

MR. LEONARD ARCHBUTT IN THE CHAIR.

# METHODS OF BACTERIOLOGICAL RESEARCH, WITH SOME ACCOUNT OF BRAN FERMENTATION.

BY J. T. WOOD.

BEFORE explaining the use of these simple pieces of apparatus which are required for the investigation of bacteria, I will give a slight outline of the origin and growth of the now widely extended science of Bacteriology.

The word bacterium is derived from the Greek derivative of *βακτηρον*, diminutive, a stick, because the first forms noticed were rod-shaped, but now that all forms, from granules (*micrococci*) to long thread-like forms (*leptothrix*), are known, they are better described by the word "micro-organisms" or the French word "microbe" which was introduced in 1878 by Sedillot. As early as the 17th century Leuwenhoek noticed these organisms, and in 1773 O. F. Müller named two genera *Monas* and *Vibrio*. In 1838 Ehrenberg published "Die Infusionsthierehen als vollkommene Organismen," and about the same time Dujardin was engaged in research on the minute organisms in infusions; they classed the forms which they described as infusoria, and as such, members of the animal kingdom. At that time naturalists had hardly begun to realise that movement was *not* solely the property of animals. It was Cohn (Untersuch. über d. Entwickelungsgesch. d. microscop. Algen u. Pilze) in 1853 who first proved on grounds of morphology and development that bacteria are plants. Nägeli (Ueber *Nosema Bombycis*) in 1857 showed that bacteria or schizomycetes, the German spaltpilze, should be classed with the fungi instead of algae. Since that time investigators have grown apace, and scores of names might be mentioned of able scientists who have

added and are adding to our knowledge of these organisms (Pasteur, Billroth, Klebs, Koch, Nägeli, Zopf, &c.), and bacteriology has become an important science, especially in the field of medical research.

[Mr. Wood here explained the principal methods employed in studying the development and action of bacteria. He illustrated this by describing different media for their cultivation, and exhibited the different pieces of apparatus required, e.g.:—A Leitz's microscope and all accessories; hot air steriliser; hot filter and flask arranged for filtering nutrient gelatine, agar-agar, &c.; cultivation flasks and tubes; damp chamber and plates; potato cultivations, &c.]

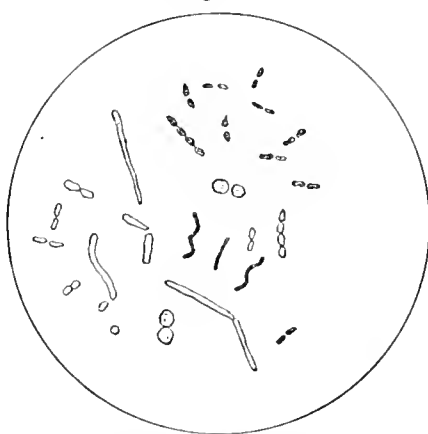
After this brief sketch of the method of bacteriological research, I will now show you a few drawings and give you a short account of some bacteria whose action and development I have specially studied, and which occur in the tanning and preparatory processes in which I am engaged. Tanning, to apply the words which Dr. W. S. Squire (this Journal, 1884, 406) used in reference to brewing, has, like all ancient industries, become perfect in the course of ages by pure empiricism. The practical man, warned by failures in one direction or another, learned by sheer practice that method which yielded the best results without troubling himself about the why and wherefore. But the "rule of thumb" processes of tanning, more so than brewing, have shown themselves under the light of scientific investigation to be of a most complex nature, belonging to a domain of organic chemistry very little explored.

In preparing skins for light and soft leathers they are first washed to remove the bulk of the lime, they are then passed through a "bate" or liquid composed of dogs' dung and water at a temperature of between 30°–40° C.; this rather disgusting process has been in use for many centuries, and yet no efficient substitute has been discovered for it, although many different attempts have been made.

The latest, of German introduction, consists in the application of a solution of cresotinic or cresotic acid (oxytoluic acid),  $C_6H_3.CH_3.OH.CO.OH$ .

Skins in the bate lose their plumpness and firmness and become soft and slippery, or "fall" as we term it, and at the same time the last traces of lime not removed by washing are dissolved out. Many of the liquids tried freed the skins completely from lime, but the resultant leather has proved too hard and rough. This failure to find a substitute for the manure bate is, I believe, in a great measure due to the fact that the action is partly caused by bacteria. On examining a "pure liquor," as the bate is called, with a power of about 600 diameters, the liquid is seen to be crowded with bacteria. The illustration (Fig. 1) shows bacteria I have observed at different times

Fig. 1.



Organisms in Bate Liquor.

in bate liquor. They are nearly all motile, possessing flagellæ, and many are putrefactive ferments, or saprophytes. I have isolated in a plate cultivation several species of bacillus, which liquefied the gelatin. This explains why skins which

are left too long in the bate lose weight and even fall to pieces.

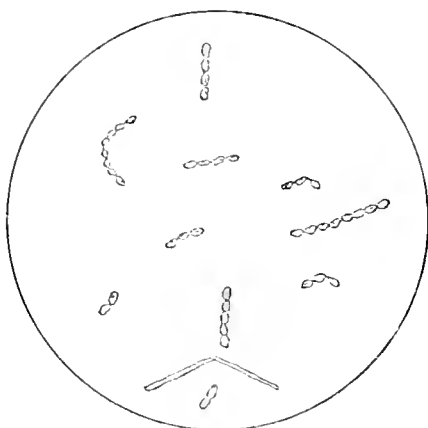
This destruction of skin is one of the great dangers in leather manufacture; frequently, small portions of the fine surface or "grain" of the skin are attacked by the bacteria and eaten away, so that the leather is covered with lines and markings, or "flaked." I have not thoroughly investigated the various microbes contained in it, but amongst others are *Bacterium Termo*, *Bacillus Subtilis*, *Micrococcus Fulvus*, and probably *Bacterium Free* of Cohn. The liquid is usually slightly alkaline, and ammonia and other gases are given off in small quantities.

After bating, the skins go through a truly fermentative process known as "drenching."

They are placed in vats containing a mixture of bran and water, about 0.4 per cent. of bran, at a temperature of 30°–35° C. This ferments vigorously for 18–24 hours, with evolution of considerable quantities of a gas and formation of weak organic acids, which have a slight swelling or "filling" action on the skin, and cleanse the pores and make it in a fit condition to receive the tannin. On examination with a high power of the microscope, the liquid is seen to be crowded with active bacteria.

Fig. 2 is a drawing of the organisms seen in the "drench" in the early stages of the fermentation, mag. 1,000 diameters.

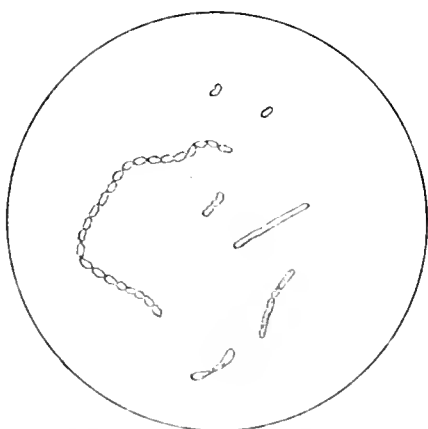
Fig. 2.



Organisms in Bran Liquor. Mag. 1,000 diam.

This was taken from a vat that had been started about three hours; the bacteria form chains, the short chains (like the pairs or dumb bells) are motile, but do not move very rapidly. As the fermentation proceeds these form longer chains (Fig. 3), some of which are very beautiful objects. The other

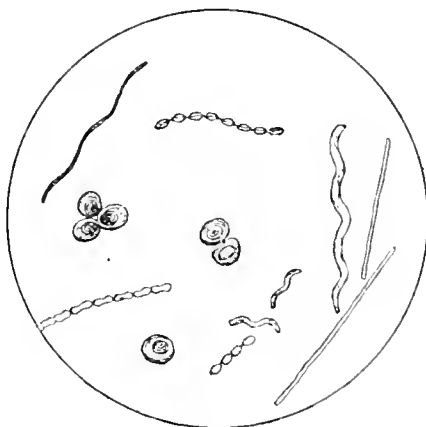
Fig. 3.



Chains of Bran Ferment. Mag. 1,000 diam.

organisms shown in the figure are probably introduced along with the skins from the bate liquor, and later on, as the nutriment of the bran ferment becomes less, they begin to develop (Fig. 4), first causing butyric fermentation and

Fig. 4.



Bran Fermentation, advanced stage. Mag. 1,000 diam.

finally putrefactive fermentation, so that the skins are rapidly destroyed. The butyric acid formed by these bacteria does not of itself attack or destroy the skin, as the quantity formed is not sufficient; the destruction of the skin is caused by other forms (probably the bacilli above mentioned), which attacks it directly. The fermentation does not appear to have been thoroughly investigated, and I had always thought that the gas given off was carbon dioxide; one day on introducing a lighted taper into a tube full of the gas, expecting to see it extinguished, to my surprise the gas sharply exploded. This was due to its admixture with air, for, on collecting another tube full, and carefully lighting it, mouth downwards, the gas burned with a bluish flame. It contains carbon dioxide and hydrogen or methane and other hydrocarbons, and has a peculiar disagreeable smell. About 1 litre of gas is evolved from 20 litres of the fermenting liquid, that is, 80 grms. of bran give a litre of the gas in the 18–24 hours required for the fermentation at the temperature of 30°; the life of the microbes themselves is much longer.

I first tried to isolate the special ferment, which causes this fermentation, by plate cultivations with the nutrient gelatin of Klein's formula, but, after many attempts, that the bate organisms, bacilli, &c. developed, instead of the one of which I was in search.

The first pure cultivation I obtained was by the fractional method.

A very minute quantity of liquid from a normally fermenting vat was introduced by means of a freshly made capillary pipette, into a sterile tube containing bran infusion; this was placed on the incubator, and the organism in greatest abundance, *i.e.*, the one causing the fermentation, develops first; as soon as the liquid in the tube was observed to become cloudy, indicating the development of the bacteria, a minute portion was used for inoculating a fresh tube; this second "attenuation" was then carried on to five or six more tubes.

I found the fifth tube to contain one species of bacteria, giving invariably similar growths in other tubes, and producing the same fermentation in sterile bran infusions. They are active mostly in the form of pairs or dumb bells, each cell  $0.75 \mu$  by  $1.25 \mu$  (Fig. 5), varying slightly in size, some forming chains; those on the surface become surrounded by a kind of jelly (Fig. 5) and form an iridescent pellicle on the surface of the liquid, the zoogloea form. When the nutriment of the liquid is exhausted this film, which is not very dense, sinks to the bottom, and the liquid above becomes somewhat clear. I have followed the development up to this point, and have seen no formation



of spores, that is true or endospores; the sediment consists of segments of the cells, which act as spores (arthrospores), and if introduced into a fresh nutrient medium, reproduce and carry on the fermentation. After the fermentation the liquids, both in the vats and tubes of pure cultivations, are always slightlyropy. Its action and morphological appearance

Fig. 5.



Bran Ferment from Pure Cultivations. Mag. 1,000 diam.

in other nutrient media are different. In a bran infusion containing glucose it forms long thread-like or leptothrix forms in one stage; the forms on the surface are usually most regular in size, but the ferment is not aerobic, active forms being found in almost equal abundance in every part of the liquid. It grows *very well* on sterile potatoes, and appears as a white-brown slime spreading on either side the track of the needle by which the inoculation was made.

This rapid and easy growth seems to show that the ferment can utilise the starch in its undissolved and unchanged condition, and explains why a vat made up with bran and cold water will ferment the same as one in which the bran was first scalded, only much more slowly. The same gas is, however, given off, and the fermentation appears in every way to be the same. On testing the liquids both before and after the fermentation with Fehling's solution no reduction takes place, showing that there is no change of the starch into maltose.

On testing with iodine all freshly made up vats and sterile tubes give the blue starch colouration strongly; after the fermentation they do not give it, showing that all the starch is decomposed.

A sterile tube (which gave the blue iodine colouration) was inoculated from one of the tubes of pure cultivation and tested every day by means of a drop withdrawn by a sterile pipette on a white plate. At the end of the seventh day the iodine reaction did not appear, showing that all the starch was decomposed. This occurs much sooner in the vats, owing to the large amount of the ferment acting.

The total constituents per 100 cc. of the infusion are:—

	Grm.
Starch .....	*176
Cellulose .....	*068
Fibrin .....	*03
Fat .....	*016
Ash .....	*626

It will be seen that the starch must be the principal body decomposed, the next being the cellulose.

Tappiner (*Zeitschrift für Biologie*, 24, 105) has shown that certain ferments decompose cellulose with the formation of fatty acids and evolution of carbon dioxide, hydrogen, and methane, the organisms requiring some nitrogenous matter for their nutriment. The bran ferment appears to decompose the starch and part of the cellulose of the bran, the fibrin and other albuminous bodies serving for its nutriment. The skins have no influence and under ordinary

circumstances take no part in the fermentation. A vat filled with cloths instead of skins ferments in exactly the same way as vats filled with skins. Some bacteria, when grown in liquids containing starch, are stained blue by the application of iodine, showing that the starch has diffused into the cells in a soluble form. The bran bacteria do not give the blue iodine reaction in any stage of their growth, which I have yet observed. The starch is decomposed, but no soluble amyloid body which is coloured blue by iodine exists in the cells. In appearance the ferment much resembles the viscous ferment of wine mentioned by Pasteur  $1\mu$ — $1.2\mu$ , in length, and in its action the mucic fermentation investigated by Kramer (this Journal, 1889, 811), and may cause the slight ropiness of the liquids in a similar way. This viscous matter is a modification of cellulose; carbon dioxide is given off but no hydrogen in the mucic fermentation (Kramer).

Not much can be surmised from the form alone, as it varies according to the nutrient conditions and from other causes; in the present case I have shown the forms of my pure cultivations to vary considerably when grown in different media.

It will be seen from the constitution of the fermenting liquid, that the fermentation I have just described, and the reactions which take place, are exceedingly complicated, and at present I am unable to state the exact manner in which the ferment acts, much less to give the chemical equations representing the decompositions effected.

The nature and constitution of starches and albuminoids is one of the most difficult problems of organic chemistry, and I can get no reliable information about the particular fermentation I have introduced to you. I am hoping to be able to follow the subject and to have the pleasure at a future meeting of giving a more complete account of the action and products of the bran ferment.

#### DISCUSSION.

Professor CLOWES said he wished to congratulate one of his old students on the excellent paper given. It was a source of pride and satisfaction to him that an old student of University College had sufficient enthusiasm not only to apply some of the knowledge gained there but to so greatly extend it that he was able to bring before the Society new matter, and to launch out upon a research likely to have most important results. He supposed that Mr. Wood's object was to find out the organisms producing the changes he desired in tanning, to cultivate these and then use them in the pure state so as to carry on the work with certainty and without the interference of other organisms. Anyone who had read Pasteur's researches on "beer" would have some idea as to what might be done by successful research of this kind.

Mr. J. B. COLEMAN expressed a doubt whether a 0.4 per cent. bran extract was sufficiently strong in which to determine the products present after fermentation. He thought that if dextrin, glucose and maltose were the intermediate products formed a stronger solution was necessary: of course for starch the iodine test was sufficiently delicate. He also questioned if it were correct that solid starch was incorporated, as such, by the organism. It was known that in similar solutions acids were always present after the fermentation, and that these and the small amount of albuminous matter present had the power of forming a modified and soluble starch; this modified starch thus forming a medium in which the ferment can grow. It was known there were acids formed in the present instance, and that might, therefore, be the explanation of the process.

Mr. WOOD said that probably the explanation just given would be correct. He certainly had proved the absence of starch in tubes, on potatoes and in the vats after fermentation, and it was therefore certain that the ferment did in some way destroy the starch, this being also evident from the large volumes of gas formed viz:—about 100 litres from a vat of about 1,500 litres capacity. As to what other bodies were formed it was impossible to give a definite opinion as the medium was so complicated.

Mr. WHITELEY said that Mr. Wood's remarks emphasised the necessity first of obtaining a pure cultivation, and second of observing its effects in simpler media.

Mr. WOOD said that, acting on the advice of Mr. A. Brown, he had already in part done this and obtained what he considered to be a pure cultivation, but he had yet to try its effects successfully on the different constituents of bran, a research which involved more time than he had yet had at disposal.

The CHAIRMAN asked as to the relative value for this class of work of a low priced  $\frac{1}{2}$ th immersion object-glass as compared with a dry glass of lower magnifying power but of the very best make, such as a  $\frac{1}{2}$ th by Powell and Lealand. He also suggested that the difficulty of filtering the media quickly might be got over by using suction.

Mr. SMALL asked if other nutrient materials had been tried instead of dung for the bates? Would other liquids have a similar effect?

Mr. WOOD: Such bates had been tried, *e.g.*, an American bate containing principally glue and glucose; but the greater proportion of new bating materials were simply devices to dissolve out lime. A solution of acid had, he believed, been tried, but was of no practical use for light skins.

## Glasgow and Scottish Section.

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J. Gibson.	A. Whitelaw.

Hon. Treasurer: W. J. Chrystal.

Hon. Local Secretary:

G. G. Henderson, Chemical Laboratory, University of Glasgow.

### SESSION 1890.

The next Meeting will take place in Edinburgh on February 4th.

Papers to be read by—

Dr. J. B. Readman. "An Account of the Manufacture of Phosphorus." Part I.

Mr. W. H. Gemmell. "On Moisture in Pulp."

Notices of Papers and Communications for the Meetings to be sent to the Local Secretary.

Meeting held in the Societies' Rooms, 207, Bath Street, Glasgow, on Tuesday, 7th January 1890.

MR. R. R. TATLOCK IN THE CHAIR.

## THE FIXATION OF NITROGEN.

BY CHARLES A. FAWSITT, F.R.S.E., F.C.S.

THE "fixation of nitrogen" is a problem which has for a long time exercised the minds and taxed the ingenuity of chemists. It is a subject of great importance from a commercial point of view, but up to the present time no satisfactory solution has been found—at least, one which will

allow of it being worked economically on a manufacturing scale. The most likely compounds to be formed if its fixation is satisfactorily effected are cyanogen or ammonia or an oxide of nitrogen.

Quite recently several valuable contributions have been made to this subject, including those by Mond, Readman, and Breneman. The latter paper, which appeared in *American Jour. Soc. Chem.*, contains a very complete history of the subject, and has been of great service to me in writing this paper. I do not intend to give a complete history of the subject, as it would occupy too much time, and could not be of interest, as much of the work done has been a repetition of some published previously. In fact, all along it has been very much a case of "follow the leader;" and to show how little progress has been made, it is sufficient to mention that the trials which were made 50 years ago more nearly approached a solution of the difficulty than anything which has since been done—at least, that has been made public.

Before passing on to the "fixation of nitrogen," I will speak for a moment of the properties of nitrogen. This element, as you are all aware, is of great importance. By its presence in the atmosphere as a diluent, we are enabled to prolong our stay in this world to a far longer period than if we had an atmosphere richer in oxygen, and no gas could be substituted for it without exerting an influence prejudicial to animal and vegetable life. As regards its chemical properties, it is distinguished by its inertness, not showing, under ordinary conditions, a marked affinity for any other element; but it is owing to this inertness, and its tendency if combined to recover its freedom, that gives such valuable properties to our explosives, in which the nitrogen and oxygen are only in a feeble state of combination, thereby causing a state of unstable equilibrium, and a slight disturbing cause is sufficient to bring into play the stronger affinities of carbon and hydrogen, setting free the nitrogen, which helps very materially to give the explosion such sudden effect.

Nitrogen, as it exists in the atmosphere, is essential to animal and vegetable life, although it is difficult to trace its action. It exists in all albuminoid bodies, and in many organic substances is the chief component. In the mineral kingdom it exists as  $\text{KNO}_3$  and  $\text{NaNO}_3$ , which form the starting point for nearly all our nitrogenous compounds.

*Fixation of Nitrogen in Air.*—In the atmosphere we find several nitrogenous compounds, nitric acid, carbonate, nitrite, and nitrate of ammonia, which play a very important part in the economy of nature even, although the actual quantities are proportionately small to its other constituents. The ammoniaical compounds may partly be accounted for by decomposition of organic matter; but the chief agent in their production is electricity, and several processes have been based on conditions under which it was thought they must be produced in the atmosphere. Schönhein accounts for the formation of  $\text{NH}_4\text{NO}_3$  and  $\text{NH}_4\text{NO}_2$  by supposing that under the influence of the electric discharge the molecule of nitrogen splits up at the same time as that of  $\text{H}_2\text{O}$ , and we get a direct combination,  $\text{N}_2 + 2 \text{H}_2\text{O} = \text{NH}_4\text{NO}_2$ . Again, if a mixture of nitrogen and oxygen are submitted to a series of electrical discharges, we get nitrous fumes, which, in contact with moisture, forms nitric acid; and it may depend very much on the amount of the moisture in the atmosphere whether we get  $\text{HNO}_3$  or  $\text{NH}_4\text{NO}_3$ .

Analogous somewhat to the fixation of nitrogen in air is the formation of  $\text{NH}_4\text{NO}_3$  when a mixture of nitrogen and hydrogen is burnt.

We have also nitrogen combining with acetylene under the influence of electric discharge  $\text{N}_2 + \text{C}_2\text{H}_2 = 2 \text{HCN}$ , which is interesting from the fact that if the electric discharge between two poles of gas carbon was passed through a dry mixture of  $\text{H}_2$  and  $\text{N}_2$  it would most likely be formed from its elements. From these examples you will notice that a certain amount of work in the form of heat has to be done before the nitrogen molecule is dissociated so as to allow of it combining with the other elements.

*Nitrides.*—When certain elements are heated in nitrogen gas a combination is effected, and we get a nitride. For instance, we have boron, silicon, tungsten, and titanium

nitrides, and these compounds when heated in steam decompose, giving off their nitrogen as ammonia. It has been advanced as a solution of the problem as to how ammonia is evolved from the lagoons in Tuscany, that boron nitride is formed and decomposed, but it is more likely to be the result of heat on organic matter.

I shall now endeavour to bring under your notice the more important work which has led up to the attempts to manufacture cyanides, &c. by fixing nitrogen.

As early as 1813 a white substance, supposed to be a chloride, was found as a deposit at some blast furnaces in Germany, but on subsequent testing was found to contain a considerable proportion of cyanide. This was scarcely credited, but received ample confirmation two years later, when similar deposits were found at some furnaces which had been erected by Neilson near Glasgow. This salt on analysis gave 43.5 per cent. KCN and 46.5 per cent.  $K_2CO_3$ . The alkaline properties of this substance had previously been discovered by the workmen, who were in the habit of taking it home to their wives as a substitute for soap powder.

Considerable interest now began to be centred in the matter by chemists, and two of the most noted, Messrs. Bunsen and Playfair, made an extensive research in order to find, if possible, a solution of the problem. Their investigations were carried out on a blast furnace at Alfreton, which seemed to have been specially designed to produce KCN, as it produced no less than 2 cwt. per day. They proved moreover that it was produced in the lower and hottest part of the furnace, whilst it was decomposed into  $N_2 + CO$  in the upper part, acting evidently as a reducing agent on the fresh ore. They analysed the gases drawn from different heights in the furnace and found that the absorption of nitrogen was very considerable where the KCN was formed. The relation of nitrogen to oxygen being 79.2:22.8, whereas in ordinary air it is 79.2:20.8. It was thought by certain chemists that there had not been a "fixation" of atmospheric nitrogen at all, the nitrogen in the KCN coming from the coal. In order to prove the truth of this, two experiments were made, one in which a quantity of sugar charcoal impregnated with chemically pure carbonate of potash was heated in a gun-barrel in a stream of pure nitrogen gas, and the other in which a similar mixture in a similar tube was heated in a stream of pure  $CO_2$ . The latter experiment produced no cyanide, the former a very considerable quantity, proving conclusively that nitrogen is fixed by alkalis carbon as cyanide.

Someone may be tempted to ask where the potash came from in the blast furnace in order to produce 2 cwt. of KCN per diem. It was found to come from the ore and coal, which contained 0.7 per cent. and 0.07 per cent. respectively.

Several eminent chemists, including Berzelius, Marchand, &c., now came forward, who said that, in order to produce cyanide from alkalis carbon, it was necessary to have moisture present, the reason assigned being that ammonia had to be produced in the first place. At this time it was well known, being mentioned by Bunsen and Playfair, that cyanides were easily produced by passing ammonia over alkalis carbon. This theory received considerable support, but subsequent experiments did not confirm it, and it seems strange how it can have received any support, as, in order to produce ammonia, we must suppose that cyanide is produced in the first place, which simultaneously produces ammonia in presence of steam; but given a certain quantity of cyanide produced, we cannot expect to receive a further quantity after it has been transformed into ammonia, and again into KCN.

Delbrueh now added another important link by proving that cyanogen was formed when nitrogen was passed over heated carbon. I think, however, that this requires confirmation, as we shall see further on.

Chemists now began seriously to consider the possibility of manufacturing cyanides, ferrocyanides, and ammonia. An inexhaustible supply of nitrogen was to be got from the atmosphere for next to nothing. The working also of the process did not appear to offer insurmountable difficulties, and, on the other side, a large demand existed for ferrocyanides,

the price for which was very much higher than at the present time.

Before bringing under your notice a few of the principal patented processes, it will be as well to mention the available sources of nitrogen.

1st. We have the atmosphere.

2nd. The waste gases from the vitriol chambers.

3rd. The waste gases from the absorption towers in the ammonia-soda process, the  $O_2$  in which, I am informed by Mr. Mond, can be reduced to less than 1 per cent.

4th. The ordinary products of combustion: for example, furnace gases.

5th. The waste gases from the manufacture of oxygen by barium oxide method, which I am told consist practically of pure nitrogen.

If air is used we must remove the oxygen, or else it would convert all the cyanide into cyanate, and, finally, into  $N_2 + CO$ . In the preparation of ammonia, the presence of cyanate is not hurtful, as it gives ammonium carbonate with steam. To effect this removal, carbon and iron are usually used, but with the latter it is difficult to free air entirely from  $O_2$  without using a very large apparatus, increasing very much the cost of working.

It can be completely combined with carbon, but the  $CO$ , acting as a diluent, impedes the production of cyanide, &c. Adler proposes barium or calcium sulphide, but this would be troublesome and expensive. A lower oxide of nitrogen has been proposed, but never, so far as I know, successfully used.

The processes proposed for the manufacture of cyanides, ferrocyanides, and ammonia may be divided into four classes:—

1st. By passing nitrogen or nitrogen plus aqueous vapour or nitrogen plus carbon monoxide over carbon impregnated with the salt of an alkali or alkaline earth.

2nd. Passing ammonia over carbon or alkalis carbon.

3rd. Those in which electricity is used as the agent for effecting a combination of nitrogen and hydrogen.

4th. Those not classed under the other three heads.

#### CLASS 1.

The first patent taken out in this country was by Newton, 1843, for a foreigner, and as the Possoz and Boisiere's experiments were being conducted in France at this time, it strikes me that it was for their process. At any rate, as the two processes are identical in details, it will suffice, seeing it is more complete, if I describe the work done by the latter.

At this time Possoz and Boisiere were hard at work in France with a small apparatus, and had managed to turn out 15 tons of prussiate of potash in a year, but owing to the high price of coal and the want of good fireclay they resolved to transfer their operations to Newcastle, where the matter was taken in hand by Messrs. Bramwell and Hughes, who imparted a great amount of skill and energy in carrying out the process, leaving no stone unturned which would conduce to its success. From the experience previously gained in France it was confidently expected the process would turn out successful, and great interest was centred in the trial.

The retorts were vertical, 10—12 ft. long, 2 ft. in diameter. The upper part was made of fireclay 9 in. thick; the lower part of iron, where the cyanised charcoal was cooled. Slits were cut in the retorts so as to allow of the furnace gases being drawn through the mass of alkalis carbon in the retort. It was found before this was done that the centre of the mass was too cold. The retorts could be heated to a temperature at which the best Stourbridge fireclay softened. The method of procedure was to fill the retorts with wood charcoal which had been impregnated with 20—30 per cent. of  $K_2CO_3$ . Air was now drawn through the retorts until the cyanising process was deemed complete, when the contents of the retort were lowered by means of a revolving screw into the cooling chamber. When the next batch was cyanised that already in the cooling chamber was by the screw lowered into a tank containing a solution of ferrous sulphate, where it was converted into prussiate of potash. The charcoal after lixiviation and drying was ready for

another operation. When the conditions, such as flow of nitrogen, heat, and percentage of  $K_2CO_3$ , &c. were favourable, 50 per cent. of the theoretical yield was obtained, and the yield of prussiate from a bench of eight retorts was two tons per week. The quality was very fine and obtained a ready sale. These trials were commenced in 1844, and continued uninterruptedly until 1847, during which time thousands of pounds were lost. But although it was a failure, Messrs. Biamwell and Hughes deserve great credit for having attacked the problem with a determination and perseverance rarely seen.

The chief causes of failure were—1st. The loss of potash, three parts being lost for one part of prussiate made. This loss could partly be accounted for, as some combined with the material of the retort and some was left in the charcoal after lixiviation, but over and above this there was a loss which was never accounted for. 2nd. The large amount of charcoal which had to be lixiviated for a small return of prussiate told heavily against its success. 3rd. The wear and tear of the plant was very great, chiefly owing to the high temperatures employed.

From the experience gained it was thought that some of the KCN was produced from the nitrogen in the charcoal, as when alkalis charcoal was heated in air-free space they still found cyanide, but as wood charcoal contains only a very small percentage of nitrogen it could not have amounted to much.

The yield of KCN varied with the temperature of the retorts, being greatest when hottest. The yield was also greater with wood charcoal than with coke, also with potash than soda. The yield of prussiate per unit of potash was greater than by the old process. Great stress was laid on the fact that water must be excluded from the apparatus, as they found that when present the yield of cyanide fell proportionally. It seems strange that about this time two patents were taken out, in which the presence of water was claimed as an improvement. Ten years later an extensive trial was made at Odenwald, in Germany. Coke balls of uniform size were saturated with  $K_2CO_3$  and heated very strongly in vertical cylinders, but although the heat was very strong very little cyanide produced, and the process was abandoned. It was thought that the heat had not been sufficiently strong, as on looking in the retorts the centre of the mass was always very much cooler than the part next the inside surface of the retort. I pass over several patents as there is no feature of interest. In 1862 Marguerite and Sourdeval patented what was at the time considered a valuable improvement, namely the substitution of barium carbonate for those of the alkalis, the great point claimed being its infusibility, thereby presenting a much larger surface to the action of the nitrogen gas, and not "clogging" up the pores of charcoal. This process received a fair trial, but was given up as a failure. In 1882, L. Mond patented a certain form of apparatus in which barium carbonate is used, but as his experience is published in the last July number of our Journal it will be better for those interested to refer to it. But it is very satisfactory to note that he speaks so hopefully of the future of the process.

Some years ago I conducted a series of trials at St. Rollox with a view of producing ammonia by acting on alkalis charcoal with nitrogen derived from the atmosphere, but the result was to add another to the long list of failures; but although apparently there have been so many, the experience gained in time past may help in some measure to solve the problem in the future.

My first experiments in 3-in. and 4-in. M.I. tubes gave such good results, often getting 50 per cent. of the theoretical yield of ammonium sulphate, that it was deemed advisable to erect a larger experimental plant consisting of 10 vertical retorts 6 in. diameter, 9 ft. long, 2 in. thick. The drawings will explain the construction and working of the same. The alkalis charcoal, containing 25–30 per cent. carbonate of soda, was charged into the top of retort A, which was provided with a tight-fitting door, the charge having fallen into the part of the retort exposed to the fire B, was heated there for 4 to 6 hours and then lowered by the screw C into the cooling box D, another charge taking the place of the one drawn down. After remaining in the cooling

box until another charge was to be lowered, it was taken out at the door E into luted iron boxes and hoisted up to be transferred to the retorts where it was steamed, the ammonia passing over to a vessel containing sulphuric acid, where it was absorbed and worked up in the usual way. The nitrogen was supplied from a blower through the pipe H at

Fig. 1.

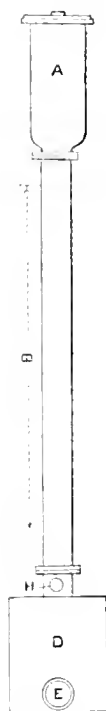
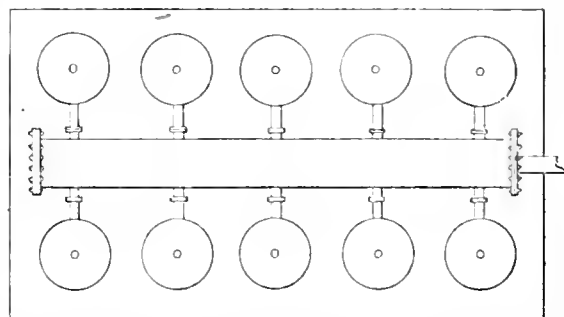


Fig. 2.



Fig. 3.



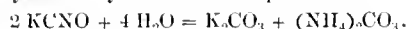
the rate of about 1–2 cubic feet per minute for each retort, and in order to extract the oxygen it was on its way passed through two vertical retorts 9 ft. long and 9 in. diameter, filled with iron turnings and kept red hot. It was found, however, that it was very difficult to deprive the air of all its oxygen, and it usually gave 3–5 per cent.  $O_2$ . The apparatus was worked continuously for some months, but did not yield  $(NH_4)_2SO_4$  which anything like paid working expenses, and which had been thought possible from the smaller trials. There was no new feature in the process other than the construction of the apparatus, and as one looks at it now it would have been much better to have profited by previous experience. The chief cause of failure lay in the fact that a good conversion could not be obtained in the C. I. retorts as they would not stand the temperature required. The loss of alkali also amounted to 10–20 per cent., but if special arrangements were made this could be all accounted for. The

cyanised charcoal contained about equal quantities of cyanide and cyanate owing to the presence of  $O_2$  in the nitrogen. In my small trials I did not find barium carbonate give so good a yield as soda or potash.

The best results were always obtained with a strong heat; in fact, when the exit pipes contained a deposit of cyanide which had volatilised, it was conclusive evidence of a good experiment. The reactions involved in the formation of ammonia in the above process are usually condensed into one,  $2 KCN + 4 H_2O = K_2CO_3 + 2 NH_3 + CO + H_2$ , whereas it would be more correctly expressed by showing it in two stages, formate of potassium being formed in the first stage,  $KCN + 2 H_2O = KCHO_2 + NH_3$ .

Then  $2 KCHO_2 = K_2CO_3 + CO + H_2$ .

From cyanate only one reaction is possible—



In 1887 Siepermann conducted a series of trials in order to find out which temperature was most suitable for the production of cyanides, using alkalis carbon, and his results were that a red was better than a white heat. Weldon also, in provisional patent, 1877, says it was owing to the strong heats employed that better results had not been obtained. I think the mass of evidence is contradictory, Possoz and Boisiere's experiments being very conclusive.

The conditions existing in the blast furnace seem to furnish a good basis for the preparation of cyanides, and it has been proposed to erect one with this end in view. It has not been very satisfactorily settled as to what is the

reaction which takes place when nitrogen acts upon alkalis carbon. It is usually expressed so—

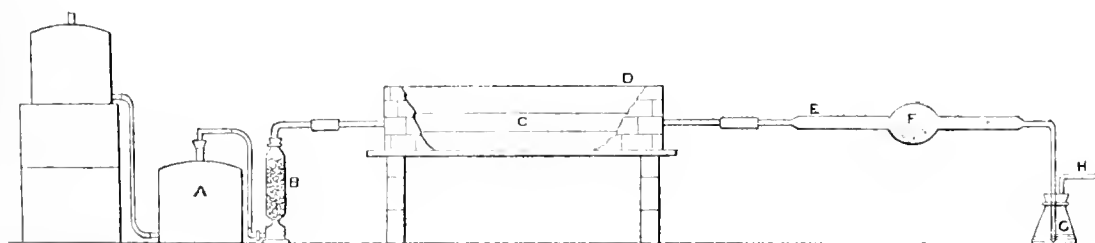


but this I am afraid tells us very little of the real truth. There are three theories advanced; one is that nitrogen in contact with highly heated carbon is converted into cyanogen, and on the reduction of the salt of the alkali or alkaline earth it combines with the metal to form cyanides. What supports this theory is that the temperature required to form KCN is lower than for NaCN, so the temperature for the reduction of  $K_2O$  is lower than that for  $Na_2O$ .

Another theory, advanced first, I think, by Berzelius, is that potassium acetylene ( $C_2K_2$ ) is first formed, and this in presence of  $N_2$  forms KCN. But it seems strange that if such a compound as  $C_2K_2$  exists that it has never been prepared, as it must be produced with facility, and not only the potassium compound, but also that of sodium, barium, &c.

Until recently the first theory appeared to me to be the most likely, as the formation of cyanide always proceeded quickly when the reduction temperature was reached, and taking into account the statement of Delbrück that cyanogen is easily prepared by passing nitrogen over heated carbon, it seemed to be almost proved. There was one point, however, which seemed to be against it, and that was the fact that no method for producing metallic barium by heating the carbonate with charcoal is known, and still the formation of  $Ba(CN)_2$  proceeds easily. In order to satisfy myself as to whether cyanogen was produced in the way just stated, and imitate, if possible, the reaction as near as possible, I asked my co-worker, Mr. Anderson, to carry out an experiment on the following lines:—Having procured a stock of

Fig. 4.



pure nitrogen by shaking up air with pyrogallate of soda, it was passed out of A through (see Fig. 4) a drying tower B, then over wood charcoal heated to a white heat in the iron tube C, 3 ft. long, 1½ in. diameter. The gas, which should contain CN, was now passed through a bulb tube E containing a piece of metallic potassium in bulb F, and when it was deemed that the whole apparatus was full of nitrogen the potassium was heated strongly. A moderate current of gas (about 30 litres in all) was kept up for two hours. The gas on leaving bulb F passed through a flask G containing water. At first, when the potassium melted, it remained perfectly clean on the surface, but when the temperature rose to the boiling point it gave off a white vapour consisting principally of potassium, but it gradually, where the heat was strongest, assumed a green colour, and finally almost black. I think it was owing to a little impurity in the potassium, as on heating a similar piece in pure  $N_2$  the same appearances were observed. On testing the deposits in different parts of the tube metallic potassium was found to be present; also carbon, but not a trace of cyanide. This experiment was repeated with sugar charcoal with an exactly similar result. In order to ensure the charcoal in each case being free from enclosed air, undecomposed organic matter, &c., the iron tube containing it was heated very strongly for two hours in a stream of nitrogen before starting the experiment. At present, therefore, an explanation of the reaction is wanted.

A third theory has been proposed as possible, namely, that a nitride of potassium is produced which combines with carbon to form KCN, but if such is the case it seems strange that it has never yet been prepared. It is possible

that the compound  $K_2C_2O_2$  is produced as in the manufacture of  $K_2$ , and this in presence of an excess of carbon and nitrogen is reduced,  $N_2$  at same time taking place of  $O_2$ . This would amount to very much the same as the theory on which  $C_2K_2$  is said to be formed.

## CLASS 2.

Under this we find that Scheele in the last century obtained KCN by heating sal-ammoniac,  $K_2CO_3$ , and charcoal, and was followed by Clouet and Kuhlmann, who found that cyanogen was produced by passing ammonia over heated charcoal,  $4 NH_3 + 3 C = 2 NH_4CN + CH_4$ .

It was well known that  $NH_3$  passed over alkalis charcoal produced cyanide, Graeger having obtained 93—95 per cent. of the theoretical yield of cyanide by passing ammonium carbonate over alkalis charcoal contained in iron tubes 1½ in. diameter. In 1857 Kamrodt tested the latter idea and proposed to utilise the waste gases produced in the manufacture of prussiate by the old method. Brinquell about this time brought out a process in which animal matter and alkali were dispensed with, advantage being taken of Clouet's and Kuhlmann's idea in which  $NH_4$  was merely passed over heated charcoal. If  $NH_4CN$  could be easily produced in this way its transformation into KCN or prussiate could easily be effected.

Fleck says that he obtains 95 per cent. of the theoretical yield of cyanide from ammonia by treating a hot mixture of charcoal, sulphur, and potash with  $(NH_4)_2SO_4$ , the waste ammonia being again utilised, the sulphocyanate of potash reduced to cyanide by heating with iron filings. This

process on testing afterwards was found unsuccessful in practice.

Romilly found that on allowing illuminating gas to bubble through ammonia and afterwards igniting and allowing the flame to impinge on the surface of a drum revolving in a solution of  $\text{KHO}$ ,  $\text{KCN}$  was produced. Still later we have the experiments of Readman, detailed already in the last October number of this Society's Journal.

Early in 1843 a patent was taken out by Laming for the production of cyanides by passing ammonia over red-hot charcoal. In 1860 Lucas patented a process for impregnating charcoal with  $\text{K}_2\text{CO}_3$ , introducing the mixture into two vertical retorts which were strongly heated, ammonia was now passed in, when the potash was converted into cyanide. If prussiate was wanted iron filings were added to the mixture of  $\text{K}_2\text{CO}_3$  and charcoal. This process was, I believe, worked on a considerable scale, but proved a failure.

Webster impregnates wood with  $\text{K}_2\text{CO}_3$ , then chars the same in retorts and passes in  $\text{NH}_3$  for the preparation of cyanides.

### CLASS 3.

Under this head numerous processes have been patented, following very much on the same lines, but so far as I know none have been worked on a manufacturing scale. I select two or three from the number. Chisholm passes the products of combustion through heated retorts containing carbon, steam being at the same time injected. He says that the nascent hydrogen combines with the nitrogen to form ammonia, but if the combination is not complete the gases as they leave the retort are submitted to an electric discharge which is said to be of great service.

Müller prepares hydrogen by leading steam over heated carbon, and stores it in a gasometer. Nitrogen is likewise prepared by passing air over carbon, absorbing the  $\text{CO}_2$  produced, and storing the nitrogen. The gases, in proportion 3 vols.  $\text{H}$  1 vol.  $\text{N}$ , are now passed through a tube, being at the same time subjected to an electric discharge when, he says, ammonia is formed.

Young submits  $\text{H}_2$  and  $\text{N}_2$  to action of electric discharge in presence of aqueous vapour; he claims this as a decided advantage.

### CLASS 4.

Under this head we have a large number of different ideas brought out. Solvay proposes to impregnate fuel with calcium chloride and mix it with limestone or other base. The mixture is then charged into a cupola, steam and air being admitted through the incandescent fuel. Chloride of ammonium is now formed and condensed. It is claimed that by the use of a decomposable chloride the limits of temperature for the successful carrying out of the reaction are not so narrow as when alkalis carbon is used in the ordinary way.

Bassett produces boron nitride by heating a borate with charcoal and alkaline carbonate, the reduction of the latter salt to metal bringing about the formation of amorphous boron, which in an atmosphere of nitrogen forms boron nitride. This compound is then decomposed by heating in presence of aqueous vapour and the ammonia absorbed in the usual way.

Dufrene passes air or nitrogen over charcoal which has been soaked in platine chloride and heated to produce finely divided platinum. Hydrogen is passed over the prepared charcoal under pressure, when ammonia is said to be formed.

Twineh states that he gets nitrogen into nascent state by depriving air of its oxygen with nitric oxide. This nitrogen is then brought into contact with nascent hydrogen made by passing steam over a metal or by action of zinc on an alkaline hydrate, when  $\text{NH}_3$  is formed. There are very many others, but as they are for the most part comprehended under those already mentioned it is of little use detailing them.

*Future of the Process.*—In conclusion it will be advisable to consider whether or not there is any hope of the process in the future, and, if asked, I should answer in the affirmative. As previously mentioned, Mond takes a hopeful view of the process in which barium oxide is used as the

base. Taking present prices as a basis I should say there is a very much better chance for success if cyanide or ferrocyanide are the products aimed at, as shown by following figures:—

1 ton of $\text{N}_2$	will produce	4.7 tons	$(\text{NH}_4)_2\text{SO}_4$	at 12% per ton=	567.
1 " $\text{N}_2$	"	5.0 "	$\text{K}_4\text{Fe}(\text{CN})_6$	" 63% "	=315.
1 " $\text{N}_2$	"	4.6 "	$\text{KCN}$	" 162% "	=745.

You will see that the  $\text{N}_2$  converted into  $\text{KCN}$  has 15 times, and if into ferrocyanide, nearly six times the value that it has if turned into sulphate of ammonia, and as new sources of application for  $\text{KCN}$  have lately arisen, and thereby increased the demand in a very marked degree, it seems as if attention should be more specially directed to this article. It seems strange converting  $\text{K}_2\text{O}$  or  $\text{BaO}$  into cyanide for the production of  $\text{NH}_3$  when cyanide commanding such a high price has to be decomposed into ammonia, an article of comparatively little value. As to which base is the best for the preparation of cyanides, opinion is divided generally between  $\text{K}_2\text{O}$  and  $\text{BaO}$ ; of course it depends very much on whether ammonia,  $\text{KCN}$ , or prussiate is to be the ultimate product. If  $\text{KCN}$ , taking into account the various results, one would fix on  $\text{K}_2\text{O}$  as if  $\text{BaO}$  were used.  $\text{BaCN}$  would require to be reconverted into  $\text{KCN}$ , which, however, would be easily accomplished by adding  $\text{K}_2\text{SO}_4$ . If ammonia was desired, taking into account the statement by Mond, one would declare for  $\text{BaO}$  as, owing to its relative infusibility and non-volatility, it is specially suited to regeneration. I think, however, we should not overlook the claims of ammonia as a base, as it possesses important advantages over the others. If it is a fact that  $\text{NH}_4\text{CN}$  can be easily produced by passing  $\text{NH}_3$  over heated charcoal, as has been stated by several chemists, it seems to be of great importance, because we have here a base which has no action on the retort, and as neither it nor the  $\text{NH}_4\text{CN}$ , seeing they are both volatile, are retained by the charcoal no lixiviation would be required. As regards the conversion of the  $\text{NH}_4\text{CN}$  into  $\text{KCN}$  this could be accomplished by passing the former as it issued from the retort through a hot concentrated solution of  $\text{KHO}$ , whereby the  $\text{NH}_3$  would be set free, and could be returned to the retort to be again converted into cyanide. One great point in having a volatile base is, that as the products of the reaction are formed they are removed out of the sphere of action, and so the reaction is allowed to go on without interruption. Pressure, if it could be applied, would no doubt help this process to a satisfactory conclusion.

It seems to be a question of apparatus more than a modification of any of the processes named which will help to solve the problem, and it seems as if fireclay retorts of not too large dimensions, heated very strongly, would be required, and the nitrogen will have to be almost free from other gases. Considerable attention is being devoted to the question at present, and any experience that I have gained is at the disposal of anyone interested in the matter. (See also pages 61—64.)

The discussion was postponed until March 4th.

## THE SALT DEPOSITS OF KHEWRA, IN THE PUNJAB.

BY E. RODGER.

It is well known that the Indian Government derives a considerable revenue from the duty upon salt, amounting to about 6,000,000*l.* annually, and with a view to the correct realisation of this income, exercises a jealous supervision over all the sources for the supply of this substance. These sources of supply are various. Large quantities of salt are imported into India from this country, a considerable amount is obtained from sea-water by means of evaporation by solar heat, and similar means are used to prepare it from the waters of salt lakes, notably the Sambhur Lake, near Jeypore, in the Rajputana district, and, lastly, there are, in the north-west corner of the Punjab, the extensive deposits of rock salt which are the subject of this paper. The



deposits are situated in the Salt Range, a chain of hills which may be considered as one of the spurs of the Himalaya mountains, although hardly connected directly with them, and the mines are not very far from the battlefield of Chillianwallah. There is now a railway which runs up almost to the entrance of the mine, by means of which Khewra can be reached in about 12 hours from Lahore.

The hills referred to, which seem to be singularly barren, rise boldly and abruptly from a level alluvial plain, through which flows, at no great distance from the base of the mountains, the river Jhelum, one of the tributaries of the Indus. Khewra itself is a small village, built well up the hillside, depending entirely on the salt mines for its existence, and almost entirely inhabited by the miners and their dependents.

One mine only is being wrought at present; it is a very extensive one, called the Mayo mine, which name was bestowed on it in memory of a visit paid by Lord Mayo during his tenure of office as Governor-General.

So far as I could judge, the mine seems to be very economically worked, except as regards the small salt, to which, however, I will refer later.

The manner of working is as follows:—

On the salt being reached, which is done by driving a tunnel for a short distance horizontally into the hillside, a chamber is cut in the salt, 45 ft. deep, from front to back, of a convenient height and length (varying in the early stages with the height and width of the salt deposit). Then a wall of the solid salt, 25 ft. thick, is left standing, with only a 12-ft. gallery running through it, and another 45-ft. chamber is commenced beyond. The wall of salt is left, of course, to support the roof. The workings are carried on at various levels, for the salt runs in layers for 900 ft. above the lowest level at which it is worked, that is to say, above the level of the lowest entrance to the mine. It is arranged that the chambers in the various levels shall coincide with one another, and thus it happens that when one chamber is worked out, another is commenced directly above it, and, being worked downwards, in time the two form only one chamber, but of double the usual height, and in some cases more than two chambers are thus run into one.

Some of the chambers formed by this process are 180 ft. high and 45 ft. broad; one which I saw was so high that blue lights burned below totally failed to illuminate the upper part at all, and, to show the height, a small fire-balloon was made use of. When I say that there are upwards of 20 chambers open, several of them as large as this, besides all the galleries and small cuttings, some idea may be formed of the vast amount of salt which has been removed, and this without doing more than touching the outskirts of the deposit.

About 1,200 labourers are employed in all, and they average about eight annas a day of wages on piece work. The workers are all Mahomedans, and they have a number of curious customs, one being that they work only nine months in the year, the remaining three being spent among the higher hills in holiday making.

The salt is blasted out by means of powder and the lumps are then cleaned if need be, and are afterwards broken to a convenient size.

The tools used are few and simple. A long iron bar or "jumper" for boring the blast-hole (which the miners do by thrusting the bar with both hands, not by striking it with a hammer as we would do), a small hammer for loosening the jumper when it jams, and a scraper for clearing away the powdered salt from the hole, the small dust thus obtained being used for tamping.

A tramway runs through the principal galleries of the mine; it is worked by coolies, who are found to be more economical than mechanical haulage, and large numbers of women are employed as porters to carry the salt from the working spots to the loading stations on the tramway lines. As is usual in subterranean workings the temperature of the mine is very constant, warm in winter and cool in summer. At the time of my visit, in the month of February, the thermometer was about 86° F. inside the mine, and I am told that this temperature is hardly ever much exceeded, so that during the hot weather the officials retire into the mine for the sake of coolness while doing office work, which at other times is done above ground.

The small salt to which I referred in the beginning of the paper consists of the small splinters and fragments produced by the blasting and by the trimming the lumps; it is unsaleable they say, and is used to fill up old workings and prevent collapse. This seems at first sight to be a great waste, but if these disused chambers must be filled up, perhaps this small salt is the cheapest material at hand.

The deposit of salt runs much deeper than they work, as it is not considered advisable to work below the level of the plain; first, on account of water, and secondly, to avoid the extra labour of carrying the salt up to the outer level. The deposit is exceedingly compact and solid, exhibiting very few cavities or fissures, and the mine, therefore, lacks the picturesqueness of some of those salt deposits in which these abound. So far as I know, only one cavity of any size has been found, which, like most similar ones in other salt mines, is partially filled with brine, and has the roof covered with crystals, but it is only of small extent.

I did not see any analyses of the salt, but the superintendent of the mine informed me that the best samples contained about 99 per cent. of chloride of sodium and 1 per cent. of all impurity, including water and insoluble matter, while the worst contained about 96·4 per cent. of chloride of sodium and 3·6 per cent. of impurity, mostly insoluble matter. I may here mention that fine crystals are rare, and that when extracted the mineral is of a pale pinky red hue, when viewed in large masses, when finely ground it is as white as snow. The duty upon salt at the time of my visit was Rs. 2·8 per maund (1 maund equals 82½ lb. nearly), while the cost of the salt itself at the mine is a mere trifle. I need, therefore, hardly point out that smuggling salt would be a very profitable business, and therefore, to prevent any contraband traffic, the mine and its approaches are heedfully guarded by watchmen, and a number of means are used to prevent pilfering, &c.; but these precautions need only be alluded to in passing.

When removed from the mine, the salt is stored in a store near the railway station, to which a siding runs. The salt is packed in bags, and transported in closed trucks, the doors of which are locked, to prevent the contents being tampered with.

In conclusion, I may say that these mines have been worked for many years, how long is uncertain, but they have been a monopoly of the Indian Government since the annexation of the Punjab in 1849, and for long before that date were worked by the Sikhs.

Lastly, I wish to take this opportunity of recording my indebtedness to the courteous superintendent of the mines for the kindness with which he received me, an unexpected visitor, at an awkward time, and for the care which he took that I should be shown everything of interest.

#### DISCUSSION.

The CHAIRMAN said he observed from Mr. Rodger's description of the mines that the method of working them was much the same as employed at Stassfurt. He observed also that, as at Stassfurt, the salt was of a pinkish colour, and it would be interesting to know from what that colour arose. At Stassfurt it was caused by crystals of peroxide of iron, which formed beautiful objects under the microscope. It would have been a benefit if Mr. Rodgers had brought samples home with him, so that some of our more enterprising members might have had the opportunity of examining them, particularly as to the presence of bromine and potash salt.

Mr. RODGER, in reply, said he had no opportunity of ascertaining whether bromine and potash were present or not, as they could readily understand when they considered in what an out-of-the-way part of the world the mines were situated. There chemical analysis was not brought to any degree of perfection, as the salt was looked upon only as an article of food. Regarding the presence of peroxide of iron, he had no doubt but such did exist, but whether or not it was in crystals he could not say. In reply to a query as to whether the mines were situated far from the sea, he stated that Karachi was the nearest port, which would be distant, he thought, from 800 to 1,000 miles.



## THE QUANTITATIVE ESTIMATION OF COLOURING MATTERS BY MEANS OF THEIR ABSORPTION SPECTRA.

BY T. L. PATTERSON, F.I.C., F.C.S.

PERHAPS there is no property so characteristic of an elementary body as that observed when the light which it emits in a state of incandescence, is examined under proper conditions by means of the prism. The smallness of the portions of matter required to be operated on, the delicacy of the reactions, and the definiteness of the results, all tend to make prismatic analysis a most exact branch of physical science and a very attractive study.

The same cannot be said of absorption spectroscopy. Here we have no lines whose position can be measured with exactitude, except in one or two instances. Neither can we operate on such minute quantities of matter. Nor is the range of bodies great which can be examined in this way. Only coloured bodies, as a rule, are available; and of these only those which absorb light selectively. There are, however, a goodly number still left which we may turn to practical account; and it is to some of these and the method of their determination that I ask your attention to-night.

Several attempts have been made to estimate colouring matters by means of their absorption spectra. So far as I have been able to ascertain, Viorordt\* in 1871 was the first to arrange apparatus for this purpose. His method consisted in the use of two slits—evidently in line—in front of an ordinary spectroscope, one for a standard solution of the colouring matter under observation, the other for one of unknown strength. By adjusting and measuring the width of each slit by means of a micrometer screw where absorption in the two solutions was equal, he was able to calculate the strength of the unknown one.

Hugo Schiff, in the same year,† proposed to estimate the colouring matter in aniline salts and blood, &c. by diluting a strong solution of known strength until a certain thickness had the same intensity of absorption when examined by an ordinary spectroscope as a similar thickness of the solution of unknown strength. By observing the amount of dilution the colouring matter was readily ascertained.

In 1874 Hemmig‡ used the absorption spectra obtained when light of equal intensity was passed through two columns of coloured liquid. One of these was a weak solution of known strength, gradually augmented until the absorption bands were equally intense. From the concentration of the known column and the height of both, he calculated the amount of colouring matter in the unknown one.

H. Krüss§ in 1882 proposed the use of a slit with adjustable sides, by which he estimated the coefficient of absorption for solutions giving band spectra, but I am not familiar with the details of this process. And

G. Krüss|| in 1887 described a universal spectroscope for qualitative and quantitative analysis, of a complicated construction, with two slits in front like Viorordt's, and a slit in the eye-piece for cutting off all but those parts of the spectra under examination. The slits were all controlled by graduated micrometer screws, and the intensity of the absorption bands measured thereby.

As far back as 1873 I devised a method for estimating the amount of colouring matter in solutions which gave band spectra, by adapting a glass tube to the low power objective of a microscope, which worked vertically in a larger tube fitted to the sub-stage. On examining the light, transmitted through a coloured solution placed in the lower tube by means of a spectrum eye-piece, having a comparison prism, over which was adjusted a sealed tube containing the standard solution, I was able to determine the height of a column of liquid of known strength, which gave the same band absorption as the tube over the comparison prism.

The height of the column of liquid was adjusted by means of the sub-stage pinion, the head of which was graduated for the purpose. On now placing another solution of unknown percentage, but known strength, in the sub-stage tube, and adjusting the height of the column to give the same absorption as before, the two spectra being viewed alongside each other in the eye-piece, it was easy to calculate the value of the unknown one. The quantity of colouring matter being the same in each column, the relative proportions were inversely as their heights, as in an ordinary colorimetric estimation.

I used this apparatus occasionally and got fairly good results, but the method is tedious and requires very careful adjustment of the light, and all the parts of the instrument. The body of the microscope must not be moved during an observation, or if so, it must be brought back to the same position it occupied before disturbance, otherwise the intensity of the light—varying as it does inversely as the square of the distance—would make some difference in the results. By moving the sub-stage in the path of the ray this error is avoided. Observations are made by setting the sub-stage milled head to the zero mark, and lowering the body till the tube on the object glass touches the bottom of the sub-stage tube. On turning down the sub-stage the liquid flows in by displacement and can be regulated to the proper height by noticing the coincidence of the bands in the eye-piece. Besides the care required in making an observation, another difficulty presents itself; for the separate comparison of two columns of liquid against a standard necessitates two readings and consequently doubles the errors.

To avoid these I sought for a better method, and hit upon the idea of adapting a slit and spectrum eye-piece to a suitable colorimeter. The only one I know of which lends itself to this kind of work is that made by Laurent, of Paris. In this instrument two tubes with flat bottoms for holding the solutions to be examined are supported side by side on suitable clips or brackets. Two narrow cylindrical tubes with flat ends are arranged to move vertically in the larger tubes, by means of rack and pinion on the sides of the instrument, with a range of 50 mm. graduated to half a mm. Over each tube a glass prism is placed, which reflects the light transmitted from a plane mirror beneath into an eye-piece, in which the two rays are focussed as semi-disks in one plane. Colorimetric measurements are easily made with this instrument, and the readings are very close.

I found considerable difficulty getting a spectrum eye-piece fitted to the colorimeter. On taking it to a well-known spectroscope maker in London he told me it was impossible, and would have nothing to do with it. Mr. Hilger fitted an eye-piece but it did not suit. Ultimately I got Mr. White, of Glasgow, to fit an adjustable slit just over the reflecting prisms; and adapted the prismatic eye-piece of a micro-spectroscope myself, with the result that I now have an instrument which works perfectly. I mention these difficulties, not because they are of any consequence now, but to point out that one should not be discouraged because an optician cannot comprehend the principle involved in adapting a particular piece of apparatus to a special end. He must work it out for himself.

The slit opens out wide, and, as now designed, the instrument may be used with the eye-piece suited for each, either for colorimetric or spectroscopic work—qualitative or quantitative.

In using the instrument, light reflected from the mirror beneath passes axially up through the solutions in the two tubes and moveable columns, where it is received on the plane faces of two prisms cut so as to totally reflect the light twice at right angles. Both beams now pass the slit in a divided slice of light—so to speak—which is analysed by a direct vision spectrum eye-piece, in which two spectra of equal intensity are seen. Gas or lamp light is better for this work than daylight, because it has no absorption lines to interfere with the bands; and the less refrangible end of the spectrum is brighter than with any but direct solar light. It is better to use a screen, as shown in the sketch, to shade extraneous light off the eyes. A little micrometer is adapted to the eye-piece by which the bands or lines can be measured

\* Chem. Soc. Jour., Vol. XXIV., pp. 602 and 759.

† Chem. Soc. Jour., Vol. XXIV., p. 760.

‡ Chem. Soc. Jour., Vol. XXVII., p. 395.

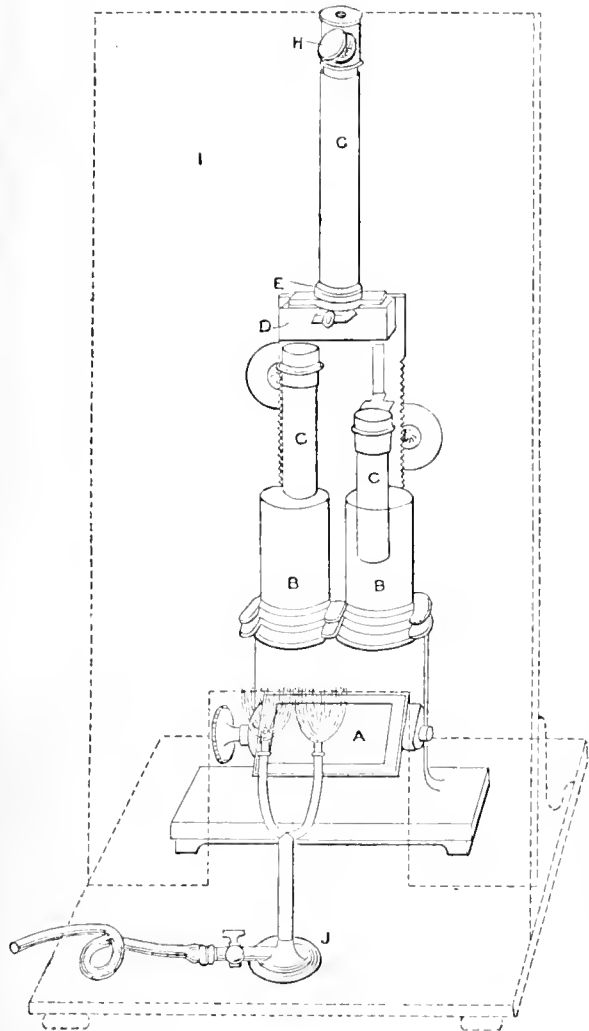
§ Chem. Soc. Jour., Vol. XLII., p. 1229.

|| Ber. Chem. Ges., Vol. XIX., p. 2739.

or mapped. Of course the Fraunhofer and bright line spectra can be examined as well with this instrument as with any other, but the dispersion being necessarily small it is not possible to measure the lines with any degree of accuracy. This spectroscope is properly adjusted for work and ready for use when the slit has a proper width, and the two spectra are seen in the eye-piece to have an equal intensity; and when the index of each cylindrical column points to zero on the scales, with the base of the columns just touching the bottoms of the glass tubes.

Fig. 1 is a general view of the spectrum-absorptimeter. Figs. 2 and 3 are front and side views showing the path of light through the instrument. The corresponding parts of each figure are lettered alike.

Fig. 1.



- A—Mirror.
- B—Solution observation tubes.
- C—Displacement glass column lined with black paper or black velvet.
- D—Reflecting prisms.
- E—Adjustable slit.
- G—Spectrum eye-piece.
- H—Micrometer scale.
- I—Wood screen with dead black surface.
- J—Double gas jet. The centres of burners correspond with the optical centres of the instrument. The burners are set so as to present the edges of the flame to the instrument, not the flat side as shown in the figure.
- K—Direct vision prisms.

Fig. 2.

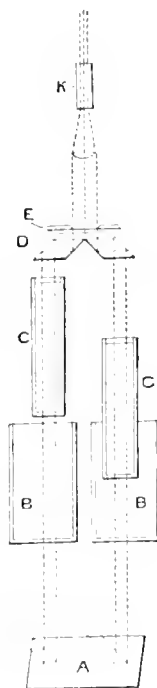
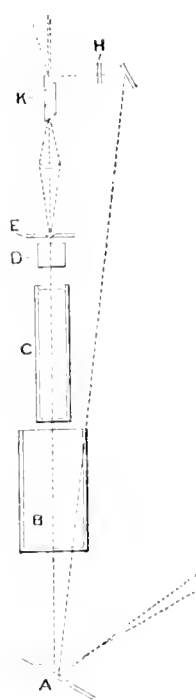


Fig. 3.



If now a solution of any colouring matter which absorbs light selectively, say a dilute alkaline alcoholic solution of alizarine of known strength, be run gently into one tube from a pipette, the two characteristic bands which this body displays will be seen to come gradually into view, on turning up the milled head of the glass cylinder and allowing the liquid to flow in between the base and the bottom of the glass tube. By raising or lowering the cylinder it is easy to get the exact thickness of liquid at which the bands are seen to the best advantage. If another solution of alizarine of unknown strength—it may be more or less concentrated—be run into the other tube, and the milled head turned so as to raise the glass column, a point is easily reached at which the two spectra viewed in the eye-piece are seen to be equal. On reading off the heights of the two columns, the strength of the unknown one is found to be inversely proportional to the height of the columns and the strength of the known solution. Let the height of the standard column be  $a$ , and its strength  $b$ , the height of the unknown column  $c$ , and its strength  $x$ . Then  $x$  is found by the formula  $\frac{a b}{c} = x$ .

I have said it is easy to get the exact thickness of liquid at which the bands are seen to the best advantage. This requires some explanation. It is not advisable when making comparisons to work with the bands black or even very dark. They should possess a somewhat smoky colour, if I may so speak, which is a dark shadow rather than a black bar; and not absorb all the light from that part of the spectrum in which they appear. The most characteristic band should always be used when there are more than one; and it is advisable in most cases to use a wide slit above the prisms to shut out all portions of the spectra except that under observation. Only in this way is it possible for the eye to judge of the shades of intensity obtained by movements of the milled head, and find the point of adjustment where no difference can be observed between the bands in the two spectra.

With these precautions a number of readings are fairly close and may be compared with those obtained with a colorimeter. But the accuracy depends to a large extent on the sharpness of vision in the observer, and a little acquaintance with the instrument. With small percentages

Fig. 4.

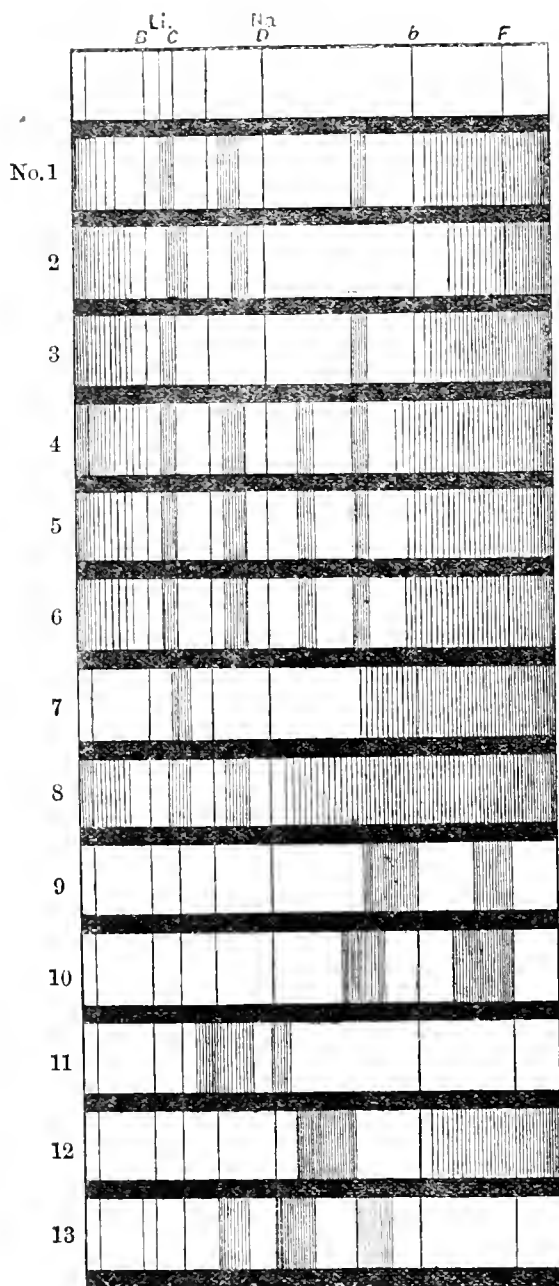


DIAGRAM OF ABSORPTION SPECTRA.

1. Olive oil, yellow.
2. Olive oil, green.
3. Linseed oil.
4. Rape oil, brown.
5. Rape oil, green.
6. Hemp seed, brown.
7. Hemp seed, refined.
8. Menhaden oil (mixed with brown rape).
9. Eosin, V. E.
10. Erythrosine, R. E.
11. Aniline green.
12. Magenta.
13. Alizarin.

great accuracy may be obtained, whilst with large percentages the readings will be less truthful, but the process is not intended for such unless in exceptional circumstances.

I have made a number of experiments to test the accuracy of reading, and illustrate the method of work.

Olive oil gives a well defined band in the red end of the spectrum No. 1, which is suitable for testing the closeness of independent readings. A series of readings were made with the same specimen of oil in both tubes of the instrument. When the left-hand column was 8 mm. high, a suitable absorption band was obtained for making the observations. The right-hand column was then altered and adjusted until absorption in the two spectra appeared to be equal. The following are the readings of the right column the left remaining stationary at 8 mm.

	Mm.
1st.....	8
2nd.....	7.75
3rd.....	8.50
4th.....	8.25
5th.....	7.50
6th.....	8.25
7th.....	7.75
8th.....	8.00
9th.....	7.50
10th.....	8.50

The mean of these is 8 mm. with an error of  $\pm 0.5$  mm.  $\pm$

Solutions of magenta in alcohol give a broad band of absorption in the green part of the spectrum No. 12, not very suitable for this kind of work, because broad bands are not so easily compared as narrow ones; but I have chosen magenta to show the amount of error to be expected in some cases.

A stock solution of this colouring matter was prepared containing .012 grm. in 400 cc. of alcohol = .00003 grm. per cc. A column of about 1.25 mm. was all that was required of this solution to give a suitable band for examination, but this column is too short for close comparative work; therefore 5 cc. were diluted to 100 cc. with alcohol and used as a standard in the following experiments.

Four solutions, *a*, *b*, *c*, and *d*, were made containing respectively 20, 10, 4, and 3 cc. of stock solution in 100 cc. of alcohol. Each were examined separately against a column of the standard solution, and five readings were taken of each solution when the absorption in the two spectra appeared equal, after re-adjusting for each reading.

*a*. The readings for this solution were 8, 7.5, 7.5, 7.25, and 8, the mean of which is 7.65 mm. when the standard column stood at 30 mm. Calculated from the strength of the solution the reading should have been 7.5 mm.

*b*. The readings were 14.75, 14.5, 15, 14.75, and 15.1, mean 14.8 mm., against a standard column of 30 mm., and the calculated height is 15 mm.

*c*. Read 38, 37.25, 37, 37.5, and 37.1, mean 37.37 mm., when the standard column stood at 30 mm., the calculated height being 37.5 mm. And

*d*. Read 42, 42, 41.5, 41, and 40, mean 41.5 mm., when the standard measured 25 mm. The true height should have been 41.66 mm.

Aniline green gives a well-defined band in the red No. 11, somewhat like chlorophyll, but more refrangible. A series of experiments were made with this colouring matter in the same manner as with magenta. The weight dissolved was the same, as also was the solvent and the dilutions; therefore I need not repeat them here.

*a*. When the standard column measured 33 mm. the readings with this solution were 7.6, 7.25, 7.5, 7.5, and 7.25, mean 7.42 mm. Corrected for strength, the height should be 7.5 mm.

*b*. With the standard column at 25 mm., read 12.1, 12.5, 12.5, 12.6, and 12.1, mean 12.36 mm., instead of the calculated height of 12.5 mm.

*c*. Against the standard at 25 mm. read 32, 31.5, 30, 29.75, and 32, mean 31.05 mm. instead of 31.25 mm. And

*d*. With standard at 20 mm. read 33.25, 33.5, 33, 33, and 33, mean 33.15 instead of 33.33 mm.

Tabulating these results and calculating them to 100, we find the error in reading to be as follows:—

TABLE NO. 1.—MAGENTA SOLUTION.

—	Calculated Height of Column.	Mean Height of Five Readings.	Colouring Matter found.	Error due to Reading.
	Mm.	Mm.	Per Cent.	
a	7.50	7.65	98.04	— 1.96
b	15.00	14.80	101.36	+ 1.36
c	37.50	37.37	100.35	+ .35
d	41.66	41.50	100.39	+ .39

TABLE NO. 2.—ANILINE GREEN.

a	7.50	7.42	101.08	+ 1.08
b	12.50	12.36	101.13	+ 1.13
c	31.25	31.05	100.64	+ .64
d	33.33	33.15	100.54	+ .54

The eye-piece slit was used with the green solutions, but not with magenta, as, in the latter case, better results were got without it. The errors recorded are less with dilute than with strong solutions, but none are excessive when we consider that this is the most trying method of testing a process which is only intended for estimating small percentages of colouring matter. It would be easy to multiply such experiments, but those I have given will suffice to show the error of reading and the minute quantity of

colouring matter required to make a quantitative estimation. The *d* solutions contain only  $\frac{.00009}{100.000}$  of a gram. per 100 cc., and much less would be required if the solutions were concentrated.

In the experiments just detailed, I have dealt with simple colouring matters; but when the absorption bands occur in different parts of the spectrum, there is no reason why two, or even more, should not be determined in the same solution. By way of illustration I will mention the results of some experiments with mixtures of solutions of the two colouring matters—magenta and aniline green—we have been considering. Known quantities of these were mixed in varying proportions, thus—

I.—Contained 3 cc. stock solution of magenta and 20 cc. stock solution of aniline green in 100 cc. alcohol.

II.—5 cc. magenta to 20 cc. green.

III.—10 cc. " 20 cc. "

IV.—20 cc. " 20 cc. "

V.—10 cc. " 10 cc. "

VI.—20 cc. " 10 cc. "

VII.—20 cc. " 5 cc. "

VIII.—20 cc. " 3 cc. "

all made up to 100 cc. with alcohol.

I need not record the various spectroscopic readings. It will be sufficient to say that the mean of 5 were taken in each estimation, against the same simple standards as were used in the previous experiments. The errors were small with aniline green, but larger with magenta, especially in the strongest solutions. Calculating the cc. of colouring matter in 100 cc. of alcohol in each experiment, to 100 of mixed colouring matter, and recording the result of the readings in percentages, the errors are brought into relief. This has been done in the following table.

Table No. 3, showing the percentage of magenta and aniline green, found in mixtures of these colouring matters:—

TABLE NO. 3.

Number of Experiment.	Magenta.			Aniline Green.		
	Calculated.	Found.	Error.	Calculated.	Found.	Error.
	Per Cent.	Per Cent.		Per Cent.	Per Cent.	
I. ....	13.04	13.51	+ 0.47	86.96	87.24	+ 0.28
II. ....	20.00	19.42	— 0.58	80.00	78.74	— 1.26
III. ....	33.33	32.42	— 0.91	66.66	64.10	— 2.56
IV. ....	50.00	48.87	— 1.13	50.00	49.75	— 0.25
V. ....	50.00	40.66	— 0.34	50.00	49.70	— 0.30
VI. ....	66.66	63.54	— 3.12	33.33	33.11	— 0.22
VII. ....	80.00	77.00	— 3.00	20.00	19.83	— 0.17
VIII. ....	86.96	83.79	— 3.26	13.04	12.94	— 0.10

Experiment V. was made with half the colouring matters present in No. IV. solution. The magenta estimation is very near the truth, but I consider it doubtful in the absence of other experiments. A careful examination of Table 3 reveals a tendency to over-estimate the height of the working column, giving a — error in every determination except the first. The high percentages are not closely estimated especially in the case of magenta. But from 50 per cent. down the — error has a ratio decreasing with the percentage of colouring matter, confirming what I have already said, that better results are obtained with small percentages. The decreasing ratio and close reading are very marked in the estimation of aniline green. A slight general absorption of the whole spectrum takes place with two colouring matters in the same solution, which under certain circumstances abnormally darkens the band under examination; but it did not affect these estimations. Green, for instance, darkens the total spectrum a little, but where it was present in largest quantity, as in experiments I. and II., the error in the magenta estimation was least, and *vice versa*.

Turning now in another direction, let me cite a few examples illustrating the application of this method of analysis to ordinary quantitative work.

The estimation of alizarin in alizarin paste is an operation which involves a considerable amount of time and manipulative skill in precipitation, filtration, and washing. Considerable difficulty, too, is experienced in getting rid of associated colouring matters. With the spectroscope the quantitative estimation is as easily performed as is that of aniline green or magenta, and the foreign colouring matters interfere very little with the observations. Three samples of alizarin paste were examined in this way; .025 grm. of each was dissolved in alcohol and rendered alkaline with a sufficiency of alcoholic potash to give the characteristic absorption No. 13, and made up with alcohol to 100 cc. For comparison, a specimen of pure alizarin was prepared by sublimation from alizarin paste, after solution in caustic soda, precipitation with  $\text{BaCl}_2$ , and washing, followed by decomposition with  $\text{HCl}$  and washing, &c. A standard solution of the pure sublimate was then made containing .004 per cent. of alizarin. On comparing these solutions in

the spectrum absorptimeter—to give the instrument a name—and calculating the results from the mean of five readings which closely agreed, the first was found to contain 5.33 per cent. alizarin, the second 7.69 per cent. alizarin, and the third 23.36 per cent. alizarin. Of course all here depends on the purity of the standard specimen, and every effort should be made to secure it. I do not say mine is absolutely free from impurity, having only been made for the purpose of this illustration; but so far as it is pure the results are very close to the truth. The first sample is said to be 7 per cent. and the second 10 per cent. Both are very old specimens. The third was in the condition of putty, and has, I may mention, a somewhat curious history. It came from Germany in a lot of beetroot sugar, and passed the samplers unnoticed until the sugar was being melted in the refinery. The alkalinity of the sugar developing the purple colour of the alizarin and dyeing the men's hands was the occasion of drawing attention to it. On examination it was found that the sugar had been filled into bags which had been previously used for packing alizarin paste. The insides of the bags were smeared with alizarin, and some of them contained masses of 20 or 30 lb. of that colouring matter. Altogether between 3 and 4 cwt. were recovered from that lot of sugar. I should add that I am inclined to believe the presence of alizarin in this connexion accidental, although the cause of it was never satisfactorily ascertained.

A piece of Turkey-red cloth weighing .963 grm. and measuring 24 square inches was treated with concentrated sulphuric acid, and the colouring matter precipitated with water, filtered, and washed. The alizarin on the filter was dissolved in alcohol and alcoholic potash and made up to 200 cc. On comparing this solution with the standard mentioned in the previous experiments, and figuring out the result, it was found that the cloth contained 0.83 per cent. alizarin and 0.4306 grm. per yard.

The colouring matters in sweetmeats, which are now so attractive and various, can be determined in this way—that is to say, those of them that have absorption spectra. The spectra of known colouring matters, of the same or similar tint, are compared with that of the sweet under examination until one is found to correspond. The amount is then easily ascertained. One example will suffice here.

Twenty grms. of a pink tablet were dissolved in alcohol. The solution gave two bands in the blue which exactly coincided with those exhibited by an alcoholic solution of V E eosin No. 9. When compared with a solution containing 0.001 per cent. of this colouring matter the tablet was found to contain 0.00294 per cent. This is a very small, and no doubt harmless quantity, yet it was easily recognised and estimated. The eosin family seem to be largely used for this purpose.

Many of the coloured gelatin films used in confectionery give absorption spectra by which the colouring matters can be identified. I have found aniline greens and blues, magenta and eosin pinks, some of which fluoresce in the film almost as much as they do in solution. One grm. of a flesh-coloured specimen was dissolved in 400 cc. equal parts water and alcohol. The spectrum agreed with that of V E eosin, and revealed the presence of .162 per cent. of this colouring matter when compared with a standard solution containing .001 per cent. dissolved in water and alcohol. A purple film treated in the same way yielded .66 per cent. of magenta.

A paraffin candle was found to be coloured with erythrosine R E; No. 10 spectrum. Five grms. were dissolved in gasoline, and shaken up with alcohol in a separator. The colouring matter was thus transferred to the alcohol. After removal the coloured solution was made up to 100 cc. with the washings from the gasoline, and compared with a standard containing .001 per cent. of erythrosine. Twenty mm. of the standard equalled 9.38 mm. of the candle solution, indicating .043 per cent. of erythrosine.

Sometimes a coloured impurity may be turned to account for the quantitative estimation of the substance in which it naturally exists, and although not always present in exactly the same proportion, it can give some definite information which may lead to useful results. The analysis of oils is a case in point. I have often found the spectrum absorptimeter useful in approximately determining the amount of

adulteration where certain vegetable oils were mixed with those of animal origin. Chlorophyll is of course the colouring matter which enables us to recognise the mixture. It is always present in certain unrefined vegetable oils as an impurity, and its quantity is fairly constant in fresh specimens of the oil. One glance at an oil with a pocket spectroscope is sufficient to reveal the absorption bands when they exist; and the quantity of the oil giving them can afterwards be determined in the absorptimeter. The spectra of oils giving chlorophyll bands have often been referred to and mapped, but I am not aware that any attempt has hitherto been made to utilise the bands for quantitative work. Unfortunately chlorophyll is not always stable. The bands are often eliminated by refining and long exposure to light, especially those in the more refrangible end of the spectrum. Hence fresh samples should always be used for comparison, and when kept for any time, should be preserved in the dark. The band in the red is well defined\* and very characteristic. The other three are all more or less faint and only seen in good fresh specimens. The intensity of chlorophyll absorption in hemp-seed, brown rape-seed, green rape-seed, linseed and olive oil is in the order just named; and the bands have the same width and position for an equal intensity. Their spectra are shown in the diagrams, Nos. 1 and 3—6. Green olive oil differs from the others in this, that it has only two absorption bands, one of them very faint, and these are more refrangible as seen at No. 2. In linseed and olive oil the secondary bands are very faint, and seldom met with. It is therefore comparatively easy to distinguish green and yellow olive and linseed oil from others of the band-giving class. When the oil in a mixture is not one of these or when there is any doubt, it may be recognised by chemical tests and its quantity found by the absorptimeter. It is a good plan to map the absorption spectra of oils which give them on sectional paper for reference, so that when a mixed sample turns up, it can be seen by means of the eye-piece scale which of the oils already mapped the mixed specimen must be compared with. The comparative estimation should not be made unless the bands in the two spectra occupy the same position, and can be brought to the same intensity.

The most common adulteration of this kind which I have noticed is that of mixing linseed with fish oils. But olive and rape are also mixed with these and other oils. Whale, seal, and porpoise oil are all mixed with linseed in varying proportions, and no one whom I have asked seems to know the reason why; indeed, the oil merchants pretend to be totally ignorant of the mixture. Unless whales and other fish have become vegetarians and store up chlorophyll in their fat cells, there must be some manufacturing reason for chlorophyll bands appearing so often in oils of this class. Perhaps the price of vegetable oils, which is usually above that of fish oils, puts them out of the category of adulterants into that of improvers; for their addition undoubtedly brings up the colour of dark fish oils, gives them more body, and reduces their smell. But this only removes them from one category to another, and is no justification for the mixture. Whether the soap boiler, the jute manufacturer, and the leather dresser, who use fish oils in large quantity, are prepared to homologate this practice, I cannot pretend to say, but it is worthy of their consideration because of the extent to which it is carried.

Take the following examples:—

1. A sample of whale oil gave a strong band of absorption in the red corresponding with the band in linseed oil. On comparing these two oils in the instrument the height of the whale column was 11.09 mm. (mean of five closely-agreeing readings) against 5 mm. of linseed, indicating  $\frac{5 \times 100}{11.09} = 45.08$  per cent. of linseed oil.

2. Another sample of whale oil absorbed light like green olive, and 15.63 mm. were equal in intensity to 3 mm. of that oil. It therefore contained  $\frac{3 \times 100}{15.63} = 19.19$  per cent. of green olive oil.

3. A sample of seal oil was found to give the linseed spectrum, and 24.13 mm. were found to equal 8 mm.

\* I have some samples which have not lost the *a* band although exposed to light for several years. In dark oils the band is very persistent and alters very little during many years.

linseed, which shows this oil to have contained  $\frac{8 \times 100}{24 \cdot 13} = 33 \cdot 11$  per cent. of linseed oil.

4. *Menhaden Oil*, the product of a small fish very plentiful off the coast of North America, is, I fear, not to be had pure. I have examined three samples and found all mixed with vegetable oil. Two of these were said to be pure, and contained respectively about 14 per cent. and 25 per cent. of linseed oil, whilst in the third, 18·44 per cent. of brown rape oil was present. The presence of rape was confirmed by Valenta's test,\* and the saponification equivalent. The spectrum of this oil, No. 8, is much more intense than that of the darkest pure linseed oil I have seen, and some of the secondary bands are visible. Yet its outward appearance is not different from that of a pure brown fish oil.

5. A sample of lard oil, purchased as pure and genuine, will be the last example. It was found spectroscopically to exhibit the band characteristic of green olive; and when compared with a pure fresh sample at the time of purchase, now upwards of two years ago, indicated the presence of 11·7 per cent. of green olive oil.

As I happen to have this sample of lard oil beside me still, as well as the green olive against which it was tested, I thought it would be instructive to compare them again. The lard oil stood on a shelf in the laboratory, exposed to light all the time, but the olive was carefully protected from light, for testing purposes, by gumming black paper around the bottle. The lard oil was also tested against a fresh sample of green olive of recent date. On making the experiment, the lard oil indicated 8·8 per cent. against the old sample of green olive, and 7·02 per cent. against the new one. The result is very interesting, as it indicates a loss of chlorophyll by the bleaching action of light equal to about 4·68 per cent. of the olive oil originally found in the lard, whilst the olive oil protected from light may be said to have lost little or nothing of its chlorophyll during two years. 1·78 per cent., the difference between the last two estimations, may be due as much to difference of sample as to loss of chlorophyll. The experiment illustrates what I have said above on the persistency of the *a* band and the action of light on chlorophyll.

In drawing this paper to a close, let me say that I have only referred to a few applications of this instrument and method of analysis; they are chiefly those to which I have had occasion to apply them myself, and others will suggest themselves to every chemist. The colouring matter in cheese and butter can be determined; so also might fuchsine and elderberry juice when present in wines. Some of the impurities in anthracene absorb light selectively, which property might be utilised for their estimation. There are many processes in the arts where fairly accurate results have to be got in the least possible time, where this spectrum method might be found useful; as, for instance, in many dyeing operations. Whilst there are others in ordinary laboratory practice, where the quantity of colouring matter is so minute and difficult to separate and weigh, that this method of determination will be preferred to a gravimetric one.

In conclusion, I beg to acknowledge the valuable assistance of my friend Mr. John McGlashan, who has made all the experiments in connexion with this paper. I have also to thank Mr. Hugh Lusk for preparing the diagrams, and several friends who kindly procured for me pure samples of oils.

#### DISCUSSION.

The CHAIRMAN said that Mr. Patterson, with his characteristic ingenuity and perseverance, had overcome what was the first step to a solution of the difficulty which lay in the way of the determination of colouring matters by means of their absorption spectra. A great deal had been said as well as written on this subject, but he had never before found anything entirely conclusive so far as quantitative determinations were concerned. He was, however, now satisfied that, with the ingenious arrangement which Mr. Patterson had explained, a new and powerful instrument had been

brought into the hands of chemists. He was quite unprepared for the accuracy of the results obtained with small quantities, and even with the larger proportions of colouring matter which Mr. Patterson had shown, and he was also gratified at the great utility which seemed to be in prospect for that branch of work. It would scarcely be credited by those who were not involved in commercial questions relating to colouring matters how much trouble and disagreement there existed with regard to the estimation of mixed colouring matters. For example, a railway company contracts for a large quantity of "cochineal lake," and of course stipulates that it shall contain no other colouring matter. That particular colour had become a very favourite one for passenger carriages, and as it had to stand the sun and weather it was necessary that it should be of an unalterable character. It was a very common practice, however, to introduce other colouring matters, such as "eosines," and the result was that the colour of the railway carriages, after being exposed to the sun for a short time, faded. He had had a case of this kind before him quite recently.

There were so many applications for which the process would be serviceable that he believed it would materially aid in opening up a new branch of analysis altogether. The estimation of the proportions of some of the coal-tar colours which were used for colouring sweetmeats was a very important one. He had found some of these colours in confections lately, but the idea of determining accurately the proportion was not entertained, for the want of reliable methods.

One particularly interesting branch of this investigation was that relating to oils. He did not know whether oils were more adulterated now than formerly, but it was a fact every second sample that came into his hands was adulterated. He was glad to think that the comparative examination of the chlorophyll absorption bands gave every hope of serving not only to distinguish some particular kinds of vegetable oils, but of determining their proportions. Mr. Patterson suggested that there was a difficulty in accounting for the presence of these higher-priced vegetable oils in commercial fish oils, which were generally very cheap. They were no doubt introduced more to improve the appearance than to cheapen the article. The argument was often brought forward in practice and in courts of law, that it was quite impossible such vegetable oils could be present, for they were more expensive than the oil with which they were mixed. The only other reason he could assign for such adulteration was that a merchant sometimes got a job lot of these vegetable oils very cheaply, perhaps at a bankrupt sale or under special circumstances, was at a loss to obtain a suitable outlet for it, and so found it convenient to mix them with another, although ordinarily a cheaper oil. But the chemist had nothing to do with that. All he had to do was to make sure that his methods of analysis were unimpeachable, and his practice correct; then he would almost invariably find that these anomalies were explicable through the existence of special conditions or circumstances.

Mr. BIGGART asked if the presence of cotton-seed oil in lard could be detected by Mr. Patterson's process.

Mr. ELLIS asked how two colouring matters, where the bands happened to overlap each other, could be determined.

Mr. PATTERSON, in reply to Mr. Biggart, said that his process could not be applied to cotton-seed oil because it gave no absorption spectrum. It could be applied only to those oils he had mentioned, with a few others of which he had not got samples, and he would just like to mention that too much confidence was not to be placed on his or any other process. With reference to Mr. Ellis's question, in such a case the colouring matters could not be determined together. They would require to be separated, if possible by treatment with different solvents, or in other ways.

\* See Allan's Com. Org. An., Vol. II., pp. 25 and 40.



Meeting held on Monday, 3rd December 1889, in the Rooms of the Philosophical Institution, Queen Street, Edinburgh.

PROFESSOR CRUM BROWN, D.S.C., F.R.S., IN THE CHAIR.

## NOTES ON THE ALKALOIDS.

BY D. R. DOTT, F.R.S.E.

THERE exists considerable diversity of definition of the term alkaloid, which is indeed regarded by Professor Armstrong as indefinable. The author objects to a definition which makes the term apply only to compounds of a markedly poisonous character, or to compounds of a vegetable origin, considering that an alkaloid should be regarded as such on account of its constitution and chemical properties, and without reference to its source or physiological action. It is maintained that an alkaloid is any alkali-like or basic organic compound, whether of the  $NH_3$  or  $NH_2$  type, and that the term is also applicable to the basic compounds in which phosphorus or arsenic takes the place of nitrogen. The views enunciated by Professor Armstrong\* regarding the properties of nitrogen are criticised, the author contending that there is little evidence to show that nitrogen is a substance of great activity in the elementary or atomic as distinguished from the molecular state. A study of the alkaloids does not obviously confirm the theory, as many of these bodies, such as cinchonine and eegonine, are physiologically very feeble substances while on the other hand certain glucosides, like digitalin and strophanthin, are powerfully toxic. Whence it is hardly probable that the toxicity of the poisonous alkaloids is in any direct sense due to their nitrogen.

The remainder of the paper is occupied by some general remarks on the alkaloids and their salts. Although all alkaloids are probably potentially crystalline, some few have obstinately resisted all attempts to crystallise them. Among these is beberine. The author was the first to prepare a crystalline salt of that base, and was enabled to confirm the accuracy of the formula  $C_{15}H_{21}NO_3$ , ascribed by Flückiger from an analysis of the amorphous base. The hydrochloride crystallises in trimetric prisms, which is the form nearly always assumed by alkaloidal muriates. Whence it is probable that many double salts of alkaloids could be prepared, although the double muriate of morphine and codeine is almost the only one which is prepared in practice. The custom (common in this country) of describing salts of the formula  $BHCl$  as hydrochlorates is condemned as erroneous, the expression more probably applying to the so-called chlorates,  $B.HClO_3$ , hydrochloride being the more correct designation of salts of the former formula. Reference is then made to a question of some interest, which has received very little attention. It is well known that while some of the alkaloids are strong bases, whose solutions turn red litmus blue, other members of the class do not affect litmus, and only form well defined salts with the stronger acids. In some cases the combination of acid and base is so feeble that decomposition is caused by water.

It has not been determined which of all the known alkaloids is the strongest. The author tried a series of experiments to determine which of the opium bases is the strongest, by treating equivalent weights of two of them with an equivalent of hydrochloric acid in presence of water; and also, in some instances, by digesting a molecular weight of one salt in solution with a molecular weight of another alkaloid. Conclusive results are not always obtained. Codeine is the strongest and narcotine or narceine the weakest. Though doubtless the point is principally determined by the relation which the nitrogen bears to other groups in the molecule, it appears that the solubility of the base is also a factor in the case. For instance, codeine is far more soluble both in water and in alcohol than is morphine, so that the former is in a more favourable position for combining with the acid. In the case of an alkaloid which forms two or more hydrochlorides, there cannot

as a rule be any difficulty in determining which of the salts is the normal. In the case of the cinchona alkaloids, however, which contain two nitrogen atoms of different value in the molecule, and form two platonic salts, the question is not so simple, hence in some books the ordinary quinine sulphate  $Q_2.H_2SO_4$  is described as the basic, in others as the normal salt. It is a matter of old observation that several of the organic bases when precipitated by caustic alkali dissolve in excess, though it was not till a few years ago that Chastaing proved that this is due to the formation of definite compounds of the alkali and alkaloid. He regarded morphine as resembling phenol in so far as it contains a hydroxyl atom which readily allows its hydrogen to be replaced. This having been shown to be the case in regard to morphine, it became interesting to know whether other alkaloids which are readily soluble in alkalis contain a similar hydroxyl group. With this view the author and Stockman† experimented in narceine, but did not succeed in replacing a hydrogen by a methyl or ethyl group, by the method which easily effects the desired change on morphine.

In conclusion, reference is made to the alkaloids of the ammonium as distinguished from the ammonia type, it being considered that the former class have not received all the attention they deserve. This probably arises from the fact that they have a tendency towards a similarity of physiological action. The author approves of the names used by Crum Brown and Fraser,‡ as "methylnorphium iodide" and "ethylstrychnium chloride." The name "morphine methiodide" suggests a compound quite analogous to morphine hydriodide, which is misleading, as the former body does not yield morphine by any method of treatment, but always other products, and possesses also a physiological action quite distinct from that of the morphine salts. When morphine or codeine is heated in a sealed tube with hydrochloric acid, apomorphine is produced, but when the so-called "morphine ethochloride" is similarly treated no apomorphine appears, which is additional proof of the essential dissimilarity of the two classes of compounds. These facts appear to constitute a strong confirmation of the accuracy of the opinion held by Professor Armstrong and others, viz., that the ordinary alkaloidal salts are merely molecular compounds. It seems not unlikely that these ammonium-like alkaloids may become chiefly useful as media for the preparation of new alkaloids, for as methyl-codeium hydroxide yields by heating with an alkali, the potent compound methocodine,§ we may anticipate that an analogous action will occur in other instances.

## Communication.

### DEVELOPMENT OF VOLTAIC ELECTRICITY BY ATMOSPHERIC OXIDATION OF COMBUSTIBLE GASES.

BY LUDWIG MOND, ROME.

THIS Journal, Vol. VIII., No. 12, p. 990, contains an abstract of a note on the above subject by C. R. A. Wright and C. Thompson taken from the Proceedings of the Royal Society, 46, 372—376. In this abstract the following occurs:—

"The battery described by Mond and Langer (this Journal, 1889, 550) is substantially identical in principle with the batteries previously described by Wright and Thompson (Proc. Roy. Soc. 44, 182)." This statement is, in my opinion, incorrect.

On referring to the earlier communications on this subject by Wright and Thompson, including the paper quoted in the abstract (Proc. Roy. Soc. 44, 182) it will be found that none

† Unpublished experiments.

‡ Trans. Roy. Soc. Edin. xxv., 603.

§ Unpublished experiments of the author.

¶ Grimaux, Comptes Rendus, xcii.

\* Journ. Chem. Soc. Ind. 1888.  
† Pharm. Jour. 1890.



of their previous papers mentions with a single word the construction of a gas battery involving the distinguishing principle of the battery described by Mond and Langer, viz., the use of porous plates impregnated with an electrolyte and coated on both sides with electrodes. The first intimation that Messrs. Wright and Thompson ever made and experimented with such a battery is given in their note communicated to the Royal Society on the 20th June 1889, and it thus becomes evident that they have not previously described a battery "substantially identical in principle" with Mond and Langer's. The best proof, however, of how far Messrs. Wright and Thompson have been from approaching to Mond and Langer's battery so late as April 1888, the date of their previous communication to the Royal Society, is afforded by the concluding paragraph of that paper, which in summing up the results obtained with the various batteries therein described, states:—

"In no case could any current capable of depositing a few milligrammes of silver per day be obtained with an E.M.F. as great as 1 volt. . . . ."

Batteries similar to those described by Wright and Thompson in their note of June 1889 have been made by Dr. Langer and myself as far back as March 1885, but it has taken us until February 1888, the date of a patent specification giving a full and detailed description of the battery in its present form, to perfect our invention—a fact which will surprise no one at all conversant with the difficulties of the problem.

## Journal and Patent\* Literature.

### I.—GENERAL PLANT, APPARATUS, AND MACHINERY.

#### PATENTS.

*An Improved Measure and Funnel for Liquids.* T. Brown, Newcastle-on-Tyne. Eng. Pat. 18,259, December 4, 1888. 6d.

THIS is a combined measure and funnel with a spring valve attachment, and is intended for use in measuring liquids. The funnel can be made of any size, and has its sides graduated to any measure required, thus obviating the necessity of first pouring the liquid into a measure. The lower part (or neck) contains a ball-joint and valve, kept in position by a spring attached to a rod extending upwards to the top of the handle, where it is worked by the pressure of the thumb.—E. S.

*Improvements in the Manufacture of Filtering Material, applicable to the Filtration and Purification of Water, Sewage Effluents, Saccharine Juices, Aleoholic Liquors, and other Liquids and Fluids.* H. R. Gregory and G. MacDonald, London. Eng. Pat. 18,177, December 12, 1888. 4d.

THE process consists essentially in reducing ferric oxide by heating it in contact with gaseous fuel. Small pieces of iron ore, preferably hematite, are packed into a retort heated externally, preferably by producer gas. When the

charge is at a cherry-red heat gaseous fuel is admitted into the retort and brought into thorough contact with the ore. At the end of four or five hours if the exit gas be inflammable the process is finished and the charge raked out and allowed to cool. Ordinary coal gas or other gaseous fuel may be used instead of producer gas. The retorts may be oscillated, rocked, raked, &c., by machinery. The magnetic oxide so produced is available for filtering water, sewage, sugar syrups, alcoholic liquors, &c.—J. M. H. M.

*Improvements in Apparatus for Distilling, Evaporating, or Concentrating Liquids.* W. Lawrence, R. C. Garton, and C. H. Garton, London. Eng. Pat. 83, January 2, 1889. 11d.

THIS invention applies to multiple-effect distilling apparatus, in which the vapour raised from the liquid in the first vessel is made use of for heating the partly-heated liquid transferred to the second, that arising from evaporation in the second vessel for heating the liquid transferred to the third, and so on, until all the heat introduced in the original steam is exhausted. Thus the liquid "may pass in succession from vessel to vessel as many times as the columns of liquid in the pipes may, added together, amount to the pressure of the initial steam and liquid in the first vessel." The claims of the patentees principally embrace the construction and arrangement of steam jackets with internal corrugated heating surfaces, pipes, and cylinders of spiral and other shapes, as well as means for causing the liquid under treatment to flow over the heating surface in thin films. There are three sheets of drawings.

—B.

*Improvements in and Apparatus for obtaining Fresh Water from Salt Water.* J. S. Stevenson and H. Stevenson, South Shields. Eng. Pat. 186, January 4, 1889. 8d.

THE object of this invention is to provide means whereby fresh water can be obtained from salt water with fuel, which, together with the apparatus, occupies but a small amount of space, and is of small weight as compared with the amount of fresh water produced. The boiler for evaporating the salt water is heated by liquid fuel (as petroleum), and is preferably made with an internal flue, and return flues, and has a hand-hole with a packing of india-rubber or asbestos, so that cleaning can be readily done. The steam passes into a condenser which consists of an inner pipe with thin walls, and surrounded by a jacket in which cold water circulates. The liquid fuel is stored in a tank, "and kept under a pressure sufficient to maintain a continuous feed against the pressure of the vaporised oil by an air pump, which pumps air into the tank above the level of the liquid." "The liquid under this pressure is then conveyed to burners on the Bunsen principle." The apparatus is especially suitable for use on ships' lifeboats.

—E. S.

*Improvements in Apparatus for Distilling Sea or other Water.* J. Newton, London, and D. A. Quiggin, Liverpool. Eng. Pat. 538, January 11, 1889. 8d.

THIS invention relates to that class of apparatus in which pure water is produced by the distillation of sea or other water, the source of heat being the heat in steam, which may be impure, and is not therefore allowed to mix with the pure water produced in the apparatus. The primary steam, however produced, is admitted into a vertical evaporator, and passing through tubular heating surfaces, preferably helical coils, is condensed and forms impure water which is allowed to flow away. The secondary steam generated in the evaporator passes into other and similar evaporators, and produces pure tertiary steam, which, on its way to the condenser, passes through a feed heater, and heats the feed water which is supplied to the evaporators. For details the specification and drawings must be consulted.—E. S.

\* Any of these specifications may be obtained by post, by remitting the cost price, plus postage, to Mr. H. Reader Luck, Comptroller of the Patent Office, Southampton Buildings, Chancery Lane, London, W.C. The amount of postage may be calculated as follows:—

If the price does not exceed 8d. . . . .	1d.
Above 8d., and not exceeding 1s. 6d. . . . .	1d.
" 1s. 6d., " " 2s. 4d. . . . .	1½d.
" 2s. 4d., " " 3s. 4d. . . . .	2d.

*An Improved Composition or Preparation to prevent Incrustation in Steam Boilers.* W. D. Pratt, Nottingham. Eng. Pat. 607, January 12, 1889. 4d.

Irish moss or other gelatinous seaweed is boiled in water to form a glutinous decoction. To this are added "malt culms" and a small quantity of dextrin. These are thoroughly mixed and poured into moulds in the form of blocks, preferably 10 in.  $\times$  10 in.  $\times$  3 in., and dried in a kiln. The proportions of the ingredients vary with the hardness of the water. One pound of the composition to each horse-power of the boiler, or to each 100 gallons of water, is stated to keep the boiler free from incrustation for about six months.

—E. S.

*Improvements in Apparatus for Mixing, Incorporating, or Effecting the Circulation of Liquids and Semi-Liquids in Vessels.* S. B. Boulton, T. B. Haywood, H. E. Boulton, and E. R. Gabbett, London. Eng. Pat. 840, January 16, 1889. 8d.

The patentees produce the circulation of liquid in tanks or cauldrons by rotating therein an upright central shaft to which is secured an inverted conical casing open at both ends and fitted with internal radiating blades, which is entirely immersed in the liquid. Centrifugal action causes the liquid to rise within the casing, thereby drawing fresh liquor from below and discharging at the top. Or the conical casing may be reversed, withdrawing the liquid from the surface and discharging below, the effect being to produce circulation of the liquid in the tank without the use of mechanical stirrers. The specification is accompanied by one sheet of drawings, showing variations in the arrangement and in the shape of the propeller.—B.

*Improvements in or applicable to Edge-Runner Mills for Crushing or Grinding Cement, Ores, and the like.* W. R. Taylor, Rochester. Eng. Pat. 1772, January 31, 1889. 6d.

The patentee directs a blast of air between the grinding surfaces of any comminuting machine, such as an edge-runner, thus removing the finer portions of material, already sufficiently ground, from between those surfaces, so as to aid their action on the coarser particles. A typical apparatus consists of an edge-runner, on the spindle of which, at its lower end, is a bell or inverted cup, whose rim fits into a circular groove in a boss on the bed-plate concentric with the spindle, thus forming a chamber into which air can be forced by a pipe from the lower side of the bed-plate. The air is led away by pipes from the sides of the bell or inverted cup to the rear of the edge-runners, and acts as described above. If desired, scrapers may also be added to loosen the material for the better action of the blast.—B. B.

*An Improved Evaporating and Condensing Apparatus for Producing Fresh Water from Sea-Water or other Fluid for Drinking Purposes and for Boiler Feed.* H. Ferguson, London. Eng. Pat. 11,742, July 23, 1889. 8d.

The object of this invention is "to afford means by which the use of high-pressure steam for evaporating the sea-water in the evaporator can be employed, by which a high duty may be obtained from an apparatus of small weight, and occupying little space." Special arrangements are made for the easy and rapid circulation of the sea-water in the evaporator, and also for the production of dry steam from the same, in order that the water resulting from the condensation of the steam may be practically pure and fresh. For details, the drawings attached to the specification must be consulted.—E. S.

*Improvements in Signalling Apparatus for Indicating the Presence of Explosive Gases in the Atmosphere.* E. Stern and M. Kaufmann, Cologne, Prussia. Eng. Pat. 13,668, August 30, 1889. 6d.

See under XI., page 81.

*An Improved Method of Charging Liquids with Gas.* H. H. Lake, London. From C. A. Catlin and C. L. Sweetland, Rhode Island, and R. Cox, New Jersey, U.S.A. Eng. Pat. 16,037, October 11, 1889. 8d.

The system and apparatus here described are intended to be applied to mixed gases only, the object being to separate a pure gas from the mixed gases, and simultaneously to combine the pure gas with the liquid under treatment in a simple and expeditious manner. This is done by allowing the gas to traverse the upper portions of a succession of closed vessels in a continuous stream, whilst the liquid occupies the lower part of the same vessels and is discharged intermittently from each vessel to the next in a direction contrary to the flow of the gas. The liquid in each vessel is agitated to meet and mingle with the gas in the form of spray, and may be retained there until any desired degree of saturation or chemical combination has been reached.

The drawing accompanying the specification shows one arrangement of apparatus, which has been designed with special reference to the production of an acid sulphate solution of magnesia, by exposing water holding powdered oxide of magnesium in suspension to the fumes of burning sulphur, which consists principally of sulphurous acid and nitrogen. It is, however, applicable also to the combination of other solutions and gases. The apparatus consists of several cylindrical closed vessels, which are placed one above the other with their axes horizontal. Through each passes a horizontal shaft driven by independent gearing, and provided with agitating blades, which are immersed in the liquid to about the height of the shaft. The upper portions of the vessels are connected externally by pipes at alternate ends, for conducting the gas which enters at one end of the lower vessel to ascend and successively traverse all the vessels from end to end. Other connexions exist, fitted, however, with shut-off valves, for discharging the liquid from one vessel to the other as may be required. The liquid first enters the upper vessel and the gas the lower, causing a contrary movement, with a view to allowing the enriched liquid, as it descends, to meet with gas of ever richer composition. The claim is for both the method and suitable apparatus.—B.

## II.—FUEL, GAS, AND LIGHT.

*The "Godillot" Furnace for Inferior Fuels.* Brüll. Bull. Soc. d'Encouragement, 1889, 289—296.

This is a report made to the *Société d'encouragement* by Mr. Brüll on behalf of the *Comité des arts mécaniques*, and refers to a fire grate and furnace arrangement perfected by G. Alexis-Godillot, of Paris, for burning inferior and indifferent fuels.

The object of the inventor is the construction of a furnace in which the refuse from many classes of manufacture can be satisfactorily utilised in the heating of boilers and for other mechanical purposes. Such refuse, which is generally produced in considerable quantities, consists of small particles of poor quality for heating purposes, and contains a great deal of moisture. It follows that a furnace for the proper consumption of this fuel must be capable of dealing with large quantities at a time, which makes it necessary to resort to automatic feeding. The ingenious way in which the inventor has carried out the requirements is shown in the accompanying drawings, in connexion with the heating of an ordinary French boiler.

The fire grate is composed of a number of horizontal steps one above the other, in the form of a half-circle in plan. It projects from an upright wall in front which carries the fuel inlet and the automatic feeding arrangement. The fuel being pushed continuously over the crown plate of the grate, descends all round over the steps, in a conical layer of material which gradually attains to incandescence, and arrives on the lower floor of the grate thoroughly consumed and burnt out. Referring to the drawings, A is the fire-

box; B the charging trough, in which works C, the feeding screw, which is provided with a tapering axle for the prevention of clogging; D is the half-circular step grate; E an opening for lighting the fire; F the ashpit; G a sight tube for

ascertaining the state of the fire and the accumulation of ashes on the ashpit floor; H the boiler with heaters underneath; I, I, channels for heating the air before it enters the fireplace; J, J, doors for access to the fireplace; K ashpit

Fig. 1.

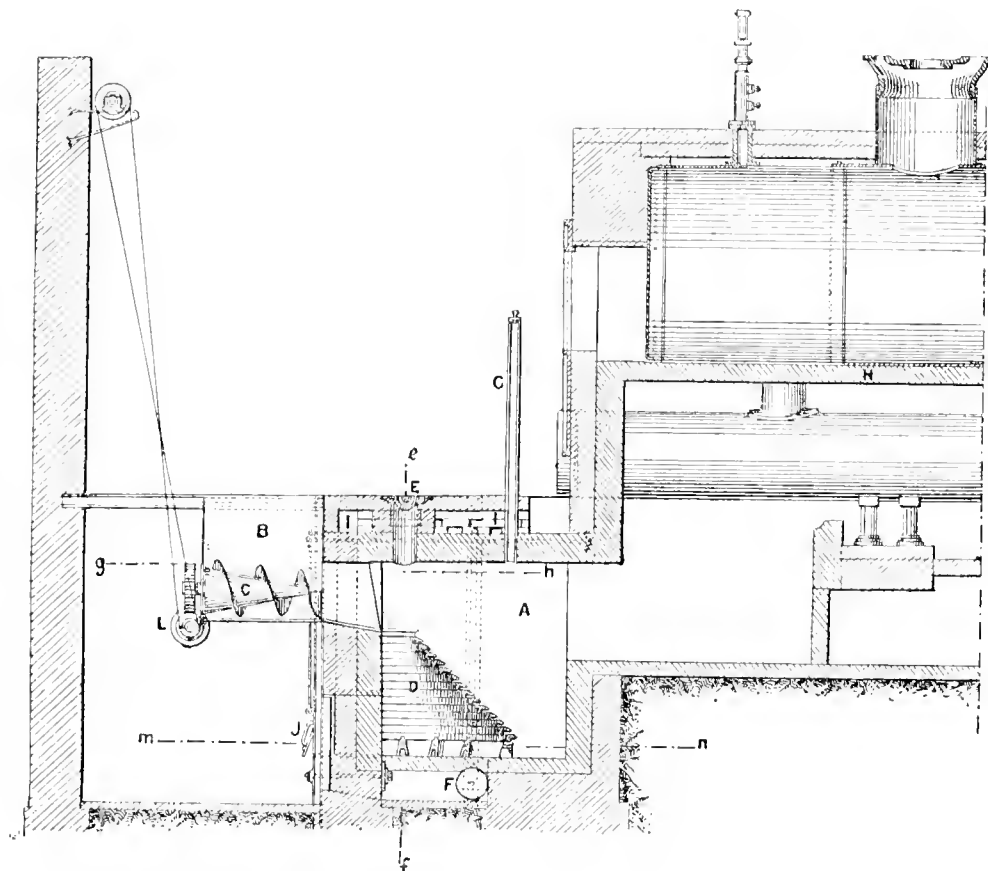


Fig. 2.

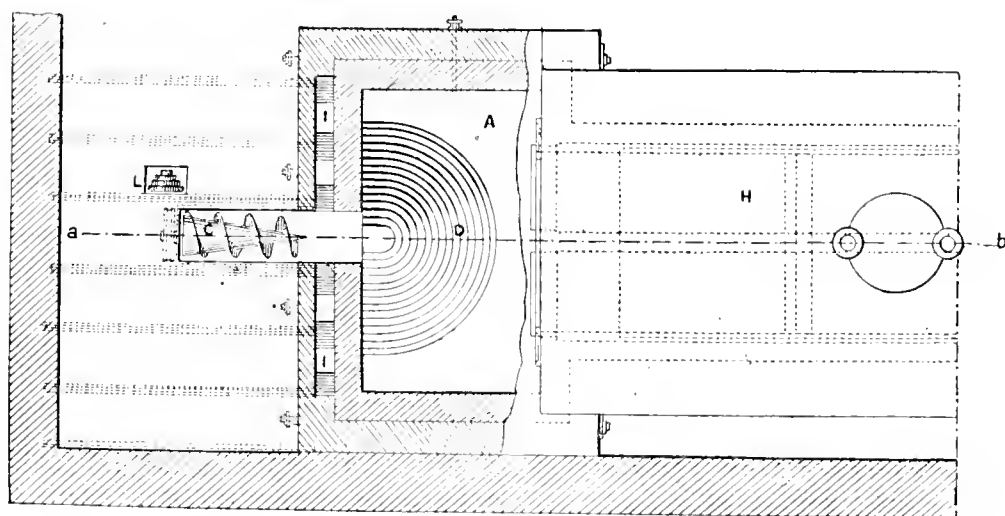
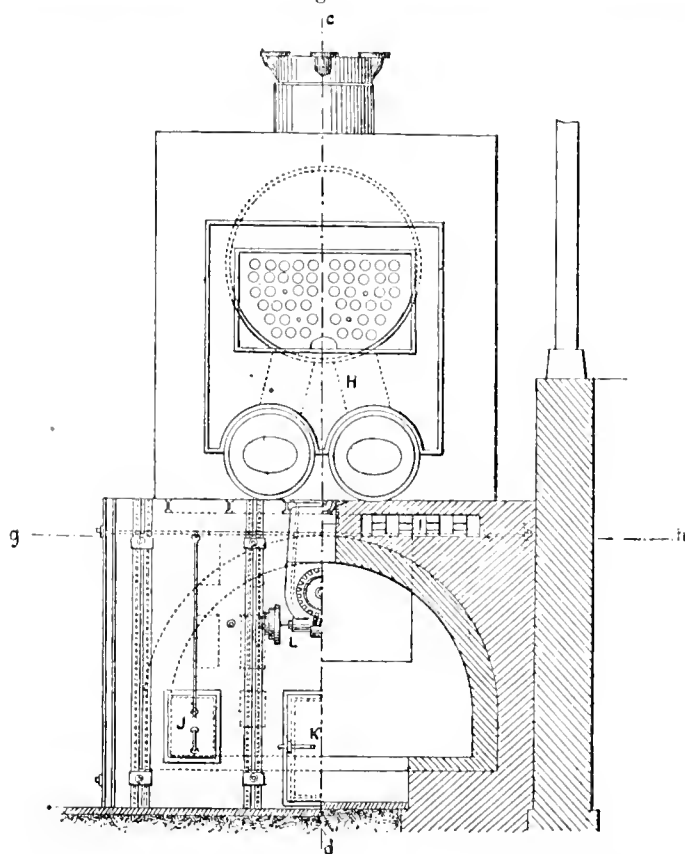


Fig. 3.



door; L gearing for driving the feeding screw. This system of firing would appear to have now been in use for some time and been applied by the inventor to a variety of materials such as sugar cane megass, spent tan, wood shavings and bark cuttings, dye wood chips, refuse from flax fibres, &c., some of them containing more than 60 per cent. of water. Some 20 reports of varying applications of the furnace were submitted to the committee together with several records of special experiments. One of the latter is reproduced in the report and refers to experiments lasting for two days, carried out at Mulhouse-on-the-Rhine on the 3rd and 4th of June 1885 with two boilers having 2,150 square feet of aggregate heating surface, and fired with chips of wood. The table accompanying the report shows that the chips contained 62.3 per cent. of water and were burnt on the first day at the rate of 0.51 lb. of moist chips per square foot of heating surface per hour, during which time 0.73 lb. of water were evaporated per square foot of heating surface. On the second day the boilers were forced, and the consumption of fuel and water amounted to 1.22 and 1.8 lb. respectively, for the same units of time and surface. The amount of evaporation in each case was calculated from water at freezing temperature, and the steam taken at the average working pressure of 22 lb. per square inch above the atmosphere. The extent of the grate surface is not given in the report, and it is therefore impossible to calculate the amount of fuel that can be profitably consumed under the circumstances per square foot of grate surface, a point of equal importance with that of the consumption of fuel per square foot of heating surface.—B.

*Roumanian Petroleum.* S. Taubert-Bârladu. Zeits. f. angew. Chem. 1889, 605—606.

The richest petroleum districts of Roumania are situated at the south-east of the Carpathian Mountains, where the

oil is obtained at five different spots. In many places, especially at Ploiesti, the ground is charged with gas to such a degree that it is only necessary to bore a hole and a jet of inflammable gas issues at once. The working of the oil is conducted in shafts and galleries, the roofs and walls being closed in with boards. The total yield from Walachei amounts to 9,000 tons, and the product contains 20—23 per cent. of solid paraffin. It is exported to Vienna to a considerable extent and yields about 15 per cent. of petroleum naphtha on refining. The wells are 50—70 metres in depth, some as much as 120 metres. Deeper borings and large refineries have been established recently by foreign enterprise, and the output of oil has increased rapidly of late. The oil districts stretch along the southern side of the Carpathians to an extent at present unknown, in the provinces of Prahova, Dimbowitzza, and Buzen, and the fields appear to be connected with those of Galicia (this Journal, 1889, 768). Two varieties of crude oil are distinguished (1) *Păcura*; and (2) *Titeiu*. The latter contains the material for refining purposes and yields 78 per cent. of burning naphtha. It cannot be imported into Austria-Hungary on account of the high duty, but is freely exported to Germany (where there is no duty on the raw product), and after refining the lighting oil can be sold at 18.4 marks per hectolitre, whilst American oil commands 27 marks. (This refers to 1884.) The petroleum from Momesti and Cusin flashes at 17°—19° C. (10° below Russian petroleum), and begins to distil at 80°, yielding a large proportion of distillate up to 150°. Roumanian oils from eight districts are described by Istrati (Jahrbuch des Organischen Laboratoriums zu Bucharest, 1888—89) and contain from 42—65 per cent. of photogen, 5—20 per cent. of petroleum naphtha, and 11—25 per cent. of solid paraffin. The residues yield nearly 50 per cent. of photogen when carefully rectified. A sample from Păcureti (Prahova) yielded 50 per cent. of lighting oil of sp. gr. 0.800 and distilling between 125°

and 280°. Austrian and American crude petroleum both yield about 50–55 per cent. of good burning oil, whilst the Russian product does not give more than 20 per cent.  
—C. A. K.

*Relative Cost of Lighting by Gas, Petroleum, and Electricity.\** Gas World, 12, 65.

M. ROLLAND, engineer, Mons, recently read a paper before the Engineering Association of the Liège School, in which he discusses the relative cost of lighting by petroleum, coal gas, and arc and incandescent lamps. Full tables of his results are published in "Le Gaz," 1889–90, No. 4.

With regard to petroleum, his basis is a price of 6.54d. per gall., a consumption of 0.0248 gall. per hour, a light of 29.3 candles at 45° (40 candles horizontally), an allowance of 0.096d. per hour per lamp for attendance, and 0.14d. for upkeep, wicks, glasses, rags, &c.; then *per candle-hour* the petroleum costs (for light at 45°) 5.453 and the extras 4.704, or together, 10.157 thousandths of a penny. If the lamp consume 25 per cent. more petroleum for the same light, the hourly cost per candle power is 11.52 thousandths of a penny.

With regard to coal gas, he gives a series of data, from slit burners to Wenham No. 4:—

Types of Burners.	Angles.	Candle Power.	Hourly Consumption.	Cub. Ft. per Candle.
Slit burner.....	0	16.9	8.90	0.523
	45	17.2	9.04	0.526
Argand.....	0	21.9	8.44	0.385
	45	19.1	8.51	0.438
Clamond (new).....	45	21.1	6.71	0.318
Welsbach.....	0	14.4	3.36	0.233
	45	10.5	3.66	0.349
Cardinal.....	45	21.9	7.73	0.353
Siemens' regenerative, No. 3.....	0	65.3	16.25	0.249
	45	46.9	16.10	0.344
Wenham, No. 2.....	0	28.4	8.80	0.310
	45	41.5	9.07	0.204
	90	45.8	9.04	0.197
Wenham, No. 4.....	0	99.0	24.19	0.244
	25	152.0	24.16	0.159
	45	170.0	23.84	0.141
	65	200.0	24.19	0.121
	90	202.0	23.63	0.118

Selecting from these the data of illumination at 45° downwards as being the most generally useful, we have, taking the price of gas at 3s. 4½d. per thousand cubic feet—

Burners.	Cost of Gas per Candle Hour; Thousandths of a Penny.	Upkeep and Interest; Thousandths of a Penny.	Total Cost per Hour; Thousandths of a Penny.
Slit burner.....	21.456	1.283	22.742
Argand.....	17.856	1.067	18.923
Clamond (new).....	12.960	1.243	14.208
Welsbach.....	14.227	1.423	15.650
Cardinal.....	14.400	0.912	15.312
Siemens, No. 3..	14.035	2.381	16.416
Wenham, No. 2..	8.294	1.411	9.705
„ No. 4..	5.731	0.970	6.701

\* Compare this Journal, 1888, 542.

Observe the quality of gas dealt with. With the first of these compare 28-candle gas in a 5-foot burner at a price of 3s. 6d., or 16-candle gas at 2s.; in this case the cost of gas is 7½ thousandths of a penny per candle per hour.

With regard to incandescent electric lamps, M. Rolland gives the candle-hour prices as follows: for an installation where there is spare power 15.36, for a special installation of a least 200 lamps with machinery 29.76, for a domestic installation 45.12 to 59.52 thousandths of a penny.

In the same address M. Rolland speaks of the efficiency of a gas burner varying according to the access of air and the temperature attained by the flame. According to him the luminous efficiency is almost exactly proportional to the temperature; whence, in part, the effect of heating the air supply. The heating of the incoming air should be done by the products of combustion, not by the flame directly. Heating the gas tends to produce deposits. The most advantageous supply of air is that at which the tip of the flame is just about to become brown; less than this, smokiness comes on; more than this, the flame becomes whiter but the lighting power falls off. To see this in an extreme form, double the length of the chimney of an argand lamp: the flame is whiter but the room is less brightly lit. This is of importance even when the air supply is heated. In larger burners the causes of loss of heat are less effective per unit of volume of the flame, and the incandescent shell is thicker in a more voluminous flame, whence the larger flame is hotter.

*Notes from the Caucasus.* Chem. Zeit. 13, 1159.

This is a reprint of an article on the inexhaustibility of the Balakhani naphtha basins, from the "Novoye Obozrenie." It goes over the same ground covered already by Professor Mendeléeff's article (this Journal, 1889, 753).—D. A. S.

*The Valuation of Coal-tar Pitch to be employed as a Binding Material for Patent Fuel (Briquettes).* F. Muck. Zeits. f. d. Berg.-Hütten und Salinenw. 37.

THE author gives the degrees of hardness, the temperature of softening, and the quantity and nature of the residue left on coking, for 24 samples of pitch. According to Preissig hard pitch is the more suitable for binding purposes, whilst Lunge is of opinion, and with reason, that soft pitch possessing a softening point of 50°–60° C. is the best. The author here remarks that in Westphalia soft pitches are generally employed. The tenacity of a sample is generally judged by the "chewing test," but in some instances this test is misleading and must be supplemented by a determination of the softening temperature. The commonly accepted opinion that soft pitches are blacker and more glossy than hard ones is pretty correct, but still mistakes may occur. The amounts of coke yielded by different samples of pitch vary very much and bear no relation to the degrees of hardness and temperatures of softening. Briquettes of good quality must not fall to pieces in the fire. According to the author this breaking up is due to the expansion of the pitch during heating. The author obtained from hard pitches in every case a voluminous residue on coking, whilst the soft pitches examined yielded, with one exception, a solid coke. It was found that those briquettes which easily crumbled in the fire were in all cases manufactured from pitch yielding a large residue on coking, whilst those which retained their shape when heated were prepared from soft pitch yielding a solid coke. For this reason the author earnestly recommends manufacturers to apply the coking test to those pitches which are to be used in the preparation of briquettes.—H. T. P.

*Estimation of Ferrocyanogen Compounds in the By-products of Gas Manufacture.* R. Gaseh. Journ. f. Gasbeleucht, 1889, 966–971.

See under XXIII., page 111.

*Aerated Fuel Company's System for Burning Oil.* Eng. and Min. J. 1889, 432.

In most systems for burning oil either a steam jet "atomises" the oil or the oil is heated in retorts and converted into gas before being burned. Such systems, from their nature, compel the use of more or less apparatus in the fire-box or furnace—in some cases the retort being placed therein, while in others the fire-box is filled with fire-brick, or other refractory material, to be heated and then used as a means to ignite the fuel. In this method compressed air is used for atomising the oil, and the pressure is so well governed that, no matter what the boiler pressure may be, a uniform pressure of five or ten pounds is kept, or whatever number of pounds per square inch is best suited for the perfect combustion of the oil, regardless of what may be on the boiler. This method of governing must result in a perfectly uniform heat of almost any intensity desired.

Another point of great advantage is that, where possible, the burners are placed so that the orifices are exactly opposite each other, thus intensifying the heat and burning the oil so completely that there is no dripping and consequently no necessity for a drip-pan or fire-brick to catch the oil that escapes from the burners without being atomised.

By this arrangement of burners, "brick arches," "breakers," or other devices in the fire-box of the boilers are not needed, so that in the event of the oil supply giving out from any cause, a wood or coal fire can be immediately built without any delay whatever, or, if occasion should ever require, both coal and oil can be burned at the same time.

It is claimed that one pound of oil will do the work of from three to six pounds of the best hard coal in forges, and do it better. There is less liability of overheating or burning stock, and in fact it is every way superior to and cheaper than coal, and only equalled by natural gas in economy and cleanliness. The system can be adapted to any forge, but as it is necessary to use from 10 to 25 pounds air pressure per square inch, a compressor of some kind is necessary. All oil can be below the burners, thus preventing the flooding of furnaces through carelessness, and meeting the requirements of the insurance companies.

Fig. 1 shows the application of this system at the Upson Nut Company's plant, at Cleveland, O., for the forging of nuts and bolts. The result here seems to prove most satisfactory, the cost of the oil being much less than coal, while the dies wear longer as there is less scale on the work, and in addition the uniformity of heat enables the workmen to do more and better work.

Fig. 1.

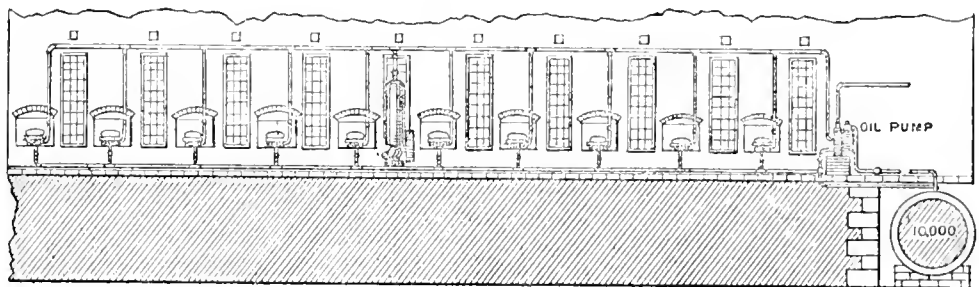
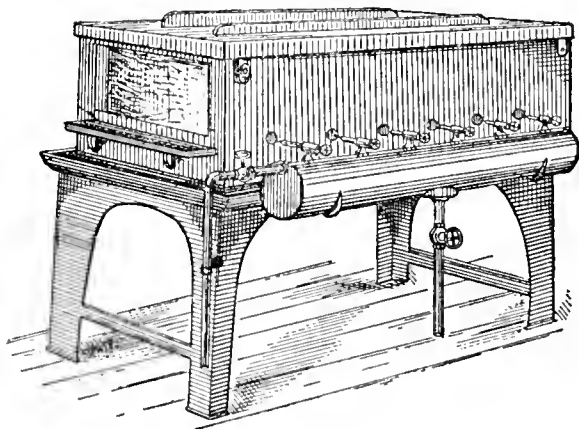


Fig. 2 shows a forge adapted to a large variety of general work. One cylinder containing six burners is placed on one side only, leaving one end open to receive the work. Both ends may be left open, if desired, for heating long pieces of iron or steel in the centre. By putting in an adjustable or moveable brick partition the operator may shut out as many burners as are not wanted, reduce the area of the fire-box, and only use one or two burners as may be best suited to the work being done. For general work this is a standard forge, and is used by the Boston and Albany railroad for hardening and tempering locomotive springs, heating large pieces of steel for forging, and for any work that requires a high degree of heat at short notice.

Fig. 2.



The most important feature about the system seems to consist in the placing of the oil supply at a level lower than the fire, thus entirely obviating the risk of sudden combustion of a large amount of oil, with its consequent danger, either through carelessness or unavoidable accident.

—O. H.

#### PATENTS.

*Improvements in or appertaining to the Separating and Collecting of the Solid Materials from Fume, Smoke, or the like.* W. P. Thompson, Liverpool. From F. P. Dewey, Washington, U.S.A. Eng. Pat. 14,644, September 17, 1889. 6d.

SCRUBBING with water often fails to remove the solid particles from fume or smoke, owing to the fact that they pass through the apparatus without being wetted thereby. It is proposed, therefore, to use instead various organic chemicals, either in aqueous solution or without water at all. The organic substances suggested are "crude petroleum and the products of its refining (except coke), organic acids and salts of the same, alcohols and alcoholic derivatives, waxes, fats, resins, turpene, bisulphide of carbon, and fixed drying and essential oils."—A. R. D.

*Apparatus for Manufacturing Heating Gas.* A. Kitson, Philadelphia, U.S.A. Eng. Pat. 14,922, September 21, 1889. 8d.

A GAS generator is divided by a vertical partition into two fuel chambers connected at the bottom by a base chamber. A mixture of highly-heated air and steam is blown in at the top of one chamber downwards through the incandescent fuel therein, and, passing through the base chamber, ascends through the second fuel chamber, whence it issues at the top as a heating gas of high quality, free from carbonic acid.

After working in this way for a certain time, the direction of the current is reversed, the blast being admitted at the top of the second fuel chamber, and so on alternately. In the base chamber, and about the bases of the fuel chambers, are disposed coils wherein the necessary heating and super-heating are performed. If desired the blast may be driven downwards simultaneously through both generators, the gases mingling in the base chamber, and being drawn thence for consumption. This arrangement allows the introduction of a hydrocarbon along with the air-and-steam blast, into one fuel chamber at a time if so preferred.—A. R. D.

*Desulphurising the Lime used in Gas Purifiers and recovering the Sulphur therefrom.* W. T. Walker, London. Eng. Pat. 15,655, October 30, 1888. 4d.

CARBONIC acid gas (preferably that "which is driven off in revivifying the lime from carbonic acid gas") is introduced into the lime which has become charged with sulphur or sulphur compounds, the carbonic acid displacing the sulphur compounds which pass into any suitable appliance for receiving or recovering the sulphur, for example, into a gas-holder, or direct into a condenser and scrubber, and thence into a "Claus Patent Kila," in which the sulphur is obtained and deposited.—F. S.

*Improved Process of and Apparatus for Manufacturing Heating or Fuel Gas.* B. Loomis, Hartford, U.S.A. Eng. Pat. 15,983, November 5, 1888. 8d.

THIS improved process relates to the alternate production of producer-gas and water-gas. In making the former, air with a little steam is drawn downwards through the fuel by an exhaustor. By this means the tarry and other vapours distilling from the newly-charged layers of coal are brought into contact with the heated fuel and converted into fixed gas. This arrangement also obviates many inconveniences that have arisen when the attempt has been made to attain the same end with a blast forced downwards by pressure applied above. The producer-gas, being drawn off at the bottom, is caused to pass first through a flue system (which acts subsequently as a steam superheater for the production of water-gas), and then through a water cooler before reaching the exhaustor. When the fuel in the generator is raised to incandescence the exhaustor is stopped, and steam superheated by traversing the flue system above mentioned, is admitted at the bottom of the generator. The resulting water-gas is drawn off at a point between the incandescent fuel and the recently added coal distilling above it, so that the vapours arising from the latter mix with the water-gas, and are fixed without undergoing a destructive decomposition.

—A. R. D.

*Improvements in the Manufacture of Coke and in the Ovens and Apparatus employed therefor.* J. Nimmo, Edinburgh, and J. Scotland, Airdrie. Eng. Pat. 18,918, December 27, 1888. 8d.

THE floor of the coke oven is moveable and mounted upon a horizontal axis, so that it can be made to tilt and discharge the coke upon a sloping screen placed below. The dust and refuse, passing through this screen, fall into a suitable receptacle, while the coke proper rolls forward into a bucket where it is quenched before being discharged into waggons for removal.—A. R. D.

*Improvements in or relating to Furnaces for Utilising Hydrocarbon as Fuel, and in Burners and operating Devices applicable therefor.* G. C. Dymond, London. From the Aërated Fuel Company, Springfield, U. S. A. Eng. Pat. 18,954, December 28, 1888, 11d.

A CLOSED tank is kept filled to a certain level with liquid hydrocarbon by a float valve of simple construction. Attached to this tank is a burner, formed injectorwise, so that compressed air forced into the tank leaves the burner nozzle mixed with the hydrocarbon in an atomised form. This

blast entering the furnace is caused to play upon some solid portion thereof, which soon becomes highly heated and completes the atomisation of the fuel, so that perfect combustion is secured and a very intense heat obtained (see previous page).—A. R. D.

*Improvements in Coke Ovens.* H. Simon, Manchester. Eng. Pat. 1271, January 23, 1889. 8d.

THESE improvements are intended to facilitate the removal of coke from those ovens which, being made in the form of vertical shafts somewhat wider at the bottom than at the top, allow their contents to drop out when a door at the bottom is opened. Extending lengthwise below the group of ovens is formed a firebrick structure sloping downwards each way from the central line, the slopes terminating in gratings. When discharged from the ovens above, the coke tumbles down the slope and over the grating (whereby the small is removed) into waggons placed below to receive it. The firebrick structure is utilised as a regenerator wherein the waste furnace gases are caused to heat the air required for combustion.

—A. R. D.

#### IV.—COLOURING MATTERS AND DYES.

*On Dehydrothioltoluidine.* P. Jacobsen. Ber. 22, 330—335.

THIS is one of several researches which have recently been carried out for the purpose of determining the constitution of primuline. The author heated this colouring matter with phosphorus and hydriodic acid to 200° for 8—12 hours. On opening the tubes H<sub>2</sub>S was evolved. The contents were mixed with water, filtered, and the residue extracted with hydrochloric acid. From this solution the base was precipitated with ammonia and crystallised several times from alcohol. The formula of the body is C<sub>11</sub>H<sub>12</sub>N<sub>2</sub>S, and it is identical with a thio-compound prepared by heating paratoluidine with sulphur in the proportions of 10:6 to 190°, and finally to 220°. Almost all toluidine had disappeared after six hours. To separate the dehydrothioltoluidine from other compounds, the melt is extracted with hot hydrochloric acid (sp. gr. 1.06), and the solution precipitated with water; the precipitate is purified by repeating this treatment, and finally separated by diluting and neutralising with ammonia. After crystallising several times from alcohol the melting point rises to 190°—191°, which is identical with that of the dehydrothioltoluidine obtained by the previous method. All its solutions exhibit a very fine blue fluorescence. In warm concentrated HCl the white base dissolves readily with an orange colour. On adding water a light yellow precipitate of the base is produced, changing to white on adding ammonia. As a further proof of the identity of the thio body obtained from sulphur and toluidine, and of that prepared from primuline by reduction, the author converted each into the phenol by the action of nitrite, and the phenol again into an acetyl derivative. Complete similarity of the products confirmed the above assumption of identity.

—A. R.

*The Action of Sulphur on certain Primary Aromatic Amines.* R. Anschütz and G. Schultz. Ber. 22, 580—586.

THE authors confirm the statements of Jacobsen (see previous abstract) concerning the production of dehydrothioltoluidine from sulphur and paratoluidine, and also describe the action of sulphur on amido-*m*-xylene. On heating these bodies in the proportion of two mols. of xylidine to two mols. of sulphur to 200°, a base of the composition C<sub>10</sub>H<sub>10</sub>N<sub>2</sub>S, readily soluble in alcohol, is the principal product. Its sulpho-derivatives are colourless.

If, however, more sulphur be taken than corresponds to the above proportions, and the temperature be raised to



200°—250° for some time, the chief product is insoluble in alcohol, and on sulphonating it the sodium salt is found to possess the property of dyeing un mordanted cotton yellow.

For purifying the first mentioned thio base of the formula  $C_{16}H_{16}N_2S$ , it is dissolved in a mixture of equal parts of sulphuric acid and water. On gradually adding water a yellow crystalline sulphate is precipitated. This is washed to remove xylydine sulphate, and then converted into the base with caustic soda. The crude product is now distilled, the distillate dissolved in a hot mixture of equal parts of sulphuric acid and water, and a sufficient quantity of water is added to cause a distinct turbidity. On cooling nearly all the sulphate crystallises out. After recrystallising it from alcohol it is converted into the base, which in its turn is crystallised from boiling alcohol, whence it is obtained in yellowish-white prismatic crystals. The base melts at 107°, and boils under a pressure of 13 mm. at 282°—284°. It contains only one amido group which can be diazotised. The azo-dyes prepared from it have the characteristic property of dyeing un mordanted cotton in a Glauber-salt bath. The acetyl derivative of the thio base is described, melting at 107°. On adding bromine dissolved in chloroform to the base a crystalline body of the formula  $C_{16}H_{16}N_2SBr_2 + CHCl_3$  separates.

Though the sodium salt of the sulpho-derivative of the base does not dye cotton, it combines with it and imparts a vivid blue fluorescence. Like primuline it can be diazotised on the fibre and further combined with naphthol and its sulphonic acids, forming dyes. Sulphur acts on amido-*p*-xylene at a temperature below 200° with formation of a thio base, readily soluble in alcohol, melting at 144°. Azo-dyes prepared from this body do not dye cotton.

When 3—4 parts of  $\psi$ -cumidine are heated with 1 part of sulphur to 185°—195°, until the evolution of  $H_2S$  ceases, the product is found to consist of two bases which are separated by treating with alcohol; the lemon-yellow portion which remains undissolved is with difficulty soluble in boiling alcohol. Its formula is  $C_{15}H_{20}N_2S$ , and its melting point 183°. From the alcoholic extract another base is obtained, melting at 123°, which has not yet been analysed.—A. R.

#### *Decomposition of Aromatic Diazo-Compounds in Alkaline Solution.* P. Friedländer. Ber. 22, 587.

OF the two methods hitherto at our disposal for eliminating aromatic amido groups, Griess' method of boiling the diazo-compound with alcohol labours under the disadvantage that phenolic others are produced along with the desired hydrocarbon, sometimes, in fact, to the exclusion of the latter, while E. Fischer's method of oxidising the hydrazine with ferric chloride, though giving generally better yields, is somewhat complicated. The author describes a new method, which is particularly applicable if the resulting products are insoluble in alkali. It is based on the fact that most diazo bodies dissolve in an excess of alkali in the cold without decomposition. On adding an alkaline reducing agent, preferably an alkaline solution of stannous chloride, and cooling with ice, nitrogen is at once evolved, and hydrogen takes its place. Thus aniline yields benzene and sulphamic acid benzene sulphonic acid.—A. R.

#### *On Acid Green.* P. Friedländer. Ber. 22, 588.

THIS product, which is now extensively used for dyeing wool in an acid bath, is prepared from ethylbenzylamine. The latter is readily obtained by heating benzylaniline and ethyl iodide or ethylaniline and benzylchloride. The new base boils at 285° with slight decomposition. The salts are very soluble. It behaves just like dimethylaniline towards benzylaldehyde; in presence of  $ZnCl_2$ , conc.  $H_2SO_4$ , or anhydrous oxalic acid, condensation is effected by heating a few hours to 100°—110°. After boiling out with water, the residue may be crystallised from acetone. The condensation product melts at 115°—116°. Its composition is that of a diethyldibenzylamidotriphenylmethane, as proved by its behaviour towards oxidising agents. Chloranil converts it into a dye of the malachite-green type. On heating the

lence base with fuming sulphuric acid at 100°, di- and tri-sulphonic acids are produced, which separate from their aqueous solutions as resinous masses on addition of sodium sulphate. On treating with the requisite quantity of lead peroxide in the usual manner, the very soluble green dyes are obtained. In evaporating the sodium salt a certain loss cannot be avoided, probably owing to the partial oxidation of the benzyl groups to benzaldehyde or its sulphonic acid.

*p*-Nitrobenzaldehyde, when condensed with ethylbenzylaniline yields a lence base, which, by reduction, sulphonating, and oxidation, is converted into a very blue "acid violet" (the sulphonic acids of *p*-amidodiethyldibenzylidiamidotriphenyl carbinol).—A. R.

#### *On the Reduction of Amidoazonaphthalene.* P. Friedländer. Ber. 22, 589.

ACCORDING to W. H. Perkin and Church, pyridine is a by-product of the reduction of amidoazonaphthalene. The author is unable to confirm this statement. Having reduced 100 grms.  $\alpha$ -amidoazonaphthalene with an acid stannous chloride solution, and concentrated the solution, the  $SnCl_2$  double compound of naphthylenediamine and of naphthylamine separated out. Any pyridine present should be found in the mother-liquor. After removing tin with zinc, the solution was treated with potassium chromate, which converts the naphthalene bases into insoluble dyes, but which would leave any pyridine present intact. After distilling with steam the distillate gave no reaction with bromine water, this being the most delicate reaction for pyridine, detecting so little as 1 part in 30,000.

Amidoazonaphthalene is best prepared by treating 1 mol.  $\alpha$ -diazonaphthalene with 1 mol.  $HCl$  naphthylamine in a cold aqueous solution, neutralising with soda and crystallising from xylene.—A. R.

#### *On Naphthoquinonedichloro-imide.* P. Friedländer and O. Böckmann. Ber. 22, 590.

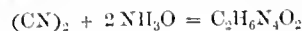
NAPHTHYLENEDIAMINE ( $\alpha^1 \alpha^4$ ) dissolved in an excess of hydrochloric acid, was treated with a solution of chloride of lime so long as a precipitate was formed. This was collected, washed, and crystallised from alcohol and xylene. The light yellow needles obtained melt at 136°—137°, and possess an intense smell of quinone. The body has both the composition and properties of a quinoneimide. Concentrated  $HCl$  acts on it in the cold even, but instead of a tetrachlorodiamine being formed, chlorine is evolved, and dichloro- $\alpha$ -naphthoquinone results. In paradiamido derivatives of naphthalene, the connexion of the nitrogen atoms seems to be much looser than in the benzene nucleus. On heating naphthoquinonediimide with sodium bisulphite, it dissolves with formation of a sulphonic acid of naphthylenediamine or amidonaphthol. The amide also reacts with primary and tertiary amines of the naphthalene and benzene series with formation of coloured condensation products.

—A. R.

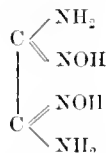
#### *The Reactions of Phenylhydrazine and Hydroxylamine.* E. Fischer. Ber. 22, 1930—1936.

PHENYLHYDRAZINE and hydroxylamine act similarly towards aldehydes, ketones, nitrous acid, and diazo compounds.

The author finds further that both combine with hydrocyanic acid under special conditions, and he has succeeded also in preparing a compound of cyanogen and hydroxylamine analogues to that of cyanogen and phenylhydrazine, which is already known. If cyanogen is passed into an aqueous solution of hydroxylamine, cooled to 0° until a white precipitate begins to form, and the solution evaporated, a crystalline mass remains. The compound has the composition  $C_2H_6N_4O_2$ , and is formed according to the equation—

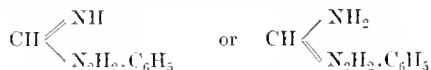


The compound belongs to the class of amidoxins, and may be called oxalamidoxim,



It is decomposed on boiling with dilute hydrochloric acid. It combines with acetic anhydride to form a new compound.

The white precipitate formed with an excess of cyanogen is not a homogeneous substance, and is probably mainly composed of a compound of oxalamidoxim and cyanogen. Phenylhydrazine combines with hydrocyanic acid to form a compound of the formula  $\text{C}_7\text{H}_9\text{N}_3$ . This is probably an amidine of the formula—



to which the author gives the name methenylphenylazidine.

Whereas hydroxylamine combines readily with cyanic acid to form a urea derivative, phenyl cyanate does not act so readily, and the compound formed consists of 2 molecules of the cyanate to one of hydroxylamine, and has the formula  $\text{C}_{14}\text{H}_{13}\text{N}_3\text{O}_3$ .

The simple urea derivative  $\text{C}_6\text{H}_5\text{NH} \cdot \text{CO} \cdot \text{NH}_2\text{O}$  may be obtained by adding phenyl cyanate slowly to a large excess of hydroxylamine.

Phenyl mustard oil and hydroxylamine combine with elimination of all sulphur according to Schiff; but if the action is carried on in the cold, colourless crystals of phenyloxysulphourea,  $\text{C}_6\text{H}_5 \cdot \text{NH} \cdot \text{CS} \cdot \text{NH}_2\text{O}$  are formed.

Carbonylsulphide in ethereal solution combines with phenylhydrazine to form the crystalline compound—



Heated to  $100^\circ$  in a closed tube it forms diphenylcarbazine,  $(\text{C}_6\text{H}_5\text{N}_2\text{H}_3)_2\text{CO}$ , with chloroformic ether, phenylhydrazine forms a compound  $\text{C}_6\text{H}_5\text{NH} \cdot \text{NH} \cdot \text{COOC}_2\text{H}_5$ . Carbonyl chloride also reacts with phenylhydrazine.—J. B. C.

#### The Sulphonic Acids of Benzidine and Benzidine Sulphone.

P. Griess and C. Duisberg. Ber. 22, 2459—2474.

BENZIDINE monosulphonic acid was obtained by one of the authors in the year 1881 by heating benzidine in an oil-bath at  $170^\circ$  with double its weight of fuming sulphuric acid. A small quantity of monosulphonic acid was so obtained which was erroneously taken to be a disulphonic acid isomeric with the well-known disulphonic acid which forms the chief product. The best method of making the monosulphonic acid consists in heating the acid sulphate of benzidine either in an oven for a short time at  $220^\circ$  or spread on enamelled iron trays in an air-bath for 24 hours at  $170^\circ$ , the mass containing in the latter case a slight excess of sulphuric acid. The impure product is extracted with alkali and the monosulphonic acid precipitated therefrom by the addition of acetic acid. It is purified by repetition of this treatment and is finally obtained chiefly in the form of four-sided plates by adding acetic acid to a boiling ammoniacal solution. It crystallises from hydrochloric acid in small tables, forms a sparingly soluble barium salt crystallising with five molecules of water, but is chiefly distinguished from the disulphonic acid by its yielding an easily soluble tetrazo derivative. This tetrazo derivative combines with phenols (naphthols) and amines (naphthylamines) or carboxylic or sulphonic acids of the same, forming dyestuffs of shades in general intermediate between those obtained from tetrazodiphenyl and tetrazodiphenyldisulphonic acid, and capable of dyeing unmordanted cotton in an alkaline bath.

Benzidine monosulphonic acid hydrochloride is made by dissolving the free sulphonic acid in hot hydrochloric acid diluted with about three parts of water. It separates out in the anhydrous state on cooling. The hydrochloric acid can be removed by treatment with boiling water.

Benzidine metadisulphonic acid is easily produced by heating benzidine with excess of ordinary concentrated sulphuric acid or by heating acid benzidine sulphate, with or without addition of sulphuric acid, to a higher temperature and for a longer time than suffice for the formation of the monosulphonic acid, as in the following example:—One part of benzidine sulphate is mixed with about two parts of sulphuric acid, the mixture heated at  $210^\circ$  for 36—48 hours and the disulphonic acid then separated from the small quantity of monosulphonic acid simultaneously formed. The yield amounts to about 90 per cent. As the properties of this acid have been previously described, it only remains to mention that the dyes it yields have less affinity for the cotton fibre than those obtained from the monosulphonic acid, but are generally of bluer shade than those made from the latter.

Benzidine orthosulphonic acid, formed by the reduction in alkaline solution and subsequent molecular transformation of *m*-nitrobenzene sulphonic acid, yields dyes for the most part incapable of dyeing unmordanted cotton.

The difference in behaviour of benzidine and azo compounds of the same towards sulphuric acid is striking. For whilst, for example, benzidine is attacked by fuming sulphuric acid only at a temperature of  $100$ — $120^\circ$ , the insoluble azo body produced by the combination of tetrazodiphenyl and 2 molecules of  $\beta$ -naphthylamine is sulphated by fuming acid in the cold, the sulphonic acid groups entering the benzidine nucleus and forming products identical with those produced from  $\beta$ -naphthylamine and benzidine mono- or disulphonic acid.

Benzidine trisulphonic acid is formed along with the tetrasulphonic acid by heating benzidine sulphate with a large excess of concentrated sulphuric acid to  $180^\circ$ — $190^\circ$  or by adding fuming sulphuric acid to a solution of benzidine in sulphuric acid heated to  $160^\circ$ — $170^\circ$ , and then heating till a sample, on testing, shows the presence of very little disulphonic acid. The barium salt of the trisulphonic acid crystallises in the form of white indistinct prisms containing 12 molecules of water, and is freely soluble in warm water, that of the tetrasulphonic acid being difficultly soluble. Advantage is taken of this fact in the separation of the two acids. The free trisulphonic acid crystallises with 2 molecules of water in delicate white plates.

Benzidine tetrasulphonic acid is formed together with the trisulphonic acid as mentioned above. It crystallises with 6 molecules of water in small white glistening needles; the barium salt forms white needles containing 8 molecules of water of crystallisation.

Benzidine sulphone is obtained on heating benzidine sulphate with excess of fuming sulphuric acid at a temperature below  $120^\circ$ . In order to purify the crude product it is extracted with boiling alcohol, dissolved in hydrochloric acid, and precipitated with sodium hydrate, by which treatment it is obtained as an anhydrous, bright yellow, finely crystallised mass, insoluble in water, alcohol, ether, and benzene. It melts at  $350^\circ$ . It possesses very weak basic properties, but its salts are decomposed when treated with a large amount of water. Strong solutions of potassium hydrate and hydrochloric acid, even at high temperatures, are without action on it. Fused with sodium hydrate at  $180^\circ$  it yields mono-hydroxy-benzidine. When the amido groups are removed by means of the diazo reaction, diphenylene sulphone identical with that prepared by Stenhouse and Graebe is obtained. The sulphate of benzidine sulphone crystallises with  $\frac{1}{2}$  molecules of water.

The azo dyes formed from benzidine sulphone are characterised by their decided blue shade. As is well known, tetrazodiphenyl combines with naphthionic acid, forming "Congo red." Now, tetrazodiphenyl sulphone gives with this acid a full violet. Again, tetrazodiphenyl yields with naphthol sulphonic acid ( $\text{OH}:\text{SO}_3\text{H} = 1:4$ ) a Bordeaux red dyestuff, "azo-orceiline," whilst tetrazodiphenyl sulphone under the same conditions forms a dull blue.

Benzidine sulphone monosulphonic acid is made by heating benzidine sulphone with fuming sulphuric acid to  $100^\circ$ . According to the duration and temperature of the operation, different sulphonic acids, namely, mono-, di-, tri-, and tetrasulphonic acids are obtained, of which the two former are sparingly, the latter freely, soluble in water. The

monosulphonic acid crystallises with 2 molecules of water, the barium salt with  $3\frac{1}{2}$  molecules.

Benzidine sulphone disulphonic acid forms pale yellow needles or prisms containing  $1\frac{1}{2}$  molecules of water of crystallisation. It is difficult to prepare, as the sulphonation tends to proceed further to the formation of the tri- and tetrasulphonic acids. The barium salt contains  $4\frac{1}{2}$  molecules of water of crystallisation. The sodium salt is easily soluble in hot, sparingly soluble in cold water, and crystallises in large yellow needles. The tetrazo derivative of this acid separates out from solution as a pale yellow voluminous precipitate which on standing becomes powdery and apparently anhydrous. Combined with naphthols or naphthol sulphonic acids, bluish red to violet dyes are formed; with the naphthylamines and naphthylamine sulphonic acids red to bluish violet dyes are obtained. But the most important dyestuffs are those formed on combination with the alkylated and phenylated naphthylamines, which not only possess the valuable property of dyeing un mordanted cotton, but, with the assistance of sodium sulphate, can be used to dye wool shades of blue fast to milling. These dyestuffs are known as "sulphone-azurines."

Ortho-toluidine is acted upon similarly to benzidine by concentrated, monohydrated, and fuming sulphuric acid, the two former producing mono- and disulphonic acids, whilst fuming acid forms toluidine sulphone and mono- and disulphonic acids of the same. The methods of preparing these substances are similar to those employed for making the corresponding benzidine compounds. Toluidine acid sulphate heated in an oven yields a mixture of mono- and disulphonic acids, separable by acetic acid. The monosulphonic acid is difficultly soluble in water; its barium salt crystallises with 4 molecules of water. Like that of benzidine monosulphonic acid, the tetrazo derivative is easily soluble. On the other hand, the disulphonic acid of toluidine, unlike that of benzidine, is easily soluble. The sodium salt crystallises with either 3 or 4 molecules of water, the barium salt in needles with 3 molecules. Toluidine sulphone closely resembles the benzidine compound, but does not yield sulphonic acids so easily as the latter.—E. B.

*The Chlorination and Bromination of Aniline, Ortho-toluidine, and Paratoluidine in presence of Excess of Mineral Acid.* R. Hafner. Ber. 22, 2524—2541.

ANILINE dissolved in 97 per cent. sulphuric acid is only slightly attacked by chlorine even after long exposure to the action of the gas, a small amount of *p*-chloraniline being so produced. Ortho- and *p*-toluidine, under similar conditions, are more freely attacked, the former yielding a monochlorinated derivative of unknown constitution, the latter a little *m*-chloro-*p*-toluidine ( $\text{CH}_3 : \text{Cl} : \text{NH}_2 = 1 : 3 : 4$ ), but chiefly *o*-chloro-*p*-toluidine ( $\text{CH}_3 : \text{Cl} : \text{NH}_2 = 1 : 2 : 4$ ).

With 65 per cent. sulphuric acid the chlorination is incomplete, but it is complete when 40 per cent. acid is used, symmetrical trichloraniline being chiefly formed with small quantities of lower chlorinated anilines and phenols, and, in the case of the weaker acid, also tarry matters.

Similar to the effect of concentrated sulphuric acid is that of 40 per cent. hydrochloric acid, *p*-chloraniline and apparently isomeric monochloro- and dichloroanilines being formed, but a portion of the aniline remaining intact. All the aniline is acted upon when dissolved in 30 per cent. hydrochloric acid, the products being *p*-chloraniline, dichloroaniline and trichloraniline, the last-named being also formed together with phenols and tarry matter from a solution in 20 per cent. acid.

In 40 per cent. hydrochloric acid *p*-toluidine is transformed into *m*-chloro-*p*-toluidine, *o*-chloro-*p*-toluidine, *m*-dichloro-*p*-toluidine ( $\text{C}_6\text{H}_3 : \text{NH}_2 : \text{Cl} : \text{Cl} = 1 : 4 : 3 : 5$ ), and chlorinated cresols.

Treatment of aniline dissolved in 97 per cent. sulphuric acid with bromine for 10 days, with frequent stirring, was practically without effect. At the end of three months a small amount of tribromaniline was formed. Bromine was likewise without action upon *o*-toluidine in 97 per cent. acid.

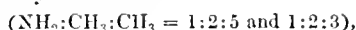
On addition of iodine, bromine acts, to some extent, on aniline dissolved in 97 per cent. sulphuric acid, forming chiefly symmetrical tribromaniline.

In 40 or 65 per cent. sulphuric acid aniline is acted upon by bromine with the formation of tribromaniline, brominated phenols being also produced in the case of the weaker acid. Concentrated hydrochloric acid does not protect aniline or *o*-toluidine from the action of bromine, which forms respectively tribromaniline and *m*-dibromo-*o*-toluidine ( $\text{CH}_3 : \text{NH}_2 : \text{Br} : \text{Br} = 1 : 2 : 3 : 5$ ), with scarcely any by-products. Symmetrical tribromaniline is at once formed as a white precipitate on adding excess of bromine to a solution of aniline hydrobromide in potassium bromide. It is noteworthy that the compound, *o*-chloro-*p*-toluidine ( $\text{CH}_3 : \text{Cl} : \text{NH}_2 = 1 : 2 : 4$ ), is, as far as known, formed only as described above.—E. B.

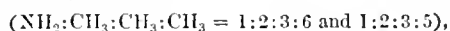
*The Colouring Matters of the Triphenylmethane Group.*  
E. Noetting. Ber. 22, 2573—2582.

I. *The Formation of Rosaniline.*—The action of oxidising agents, or more particularly of arsenic acid, upon those homologues of aniline in which both the ortho and meta positions relatively to the amido group are occupied, has been further investigated (see this Journal, 1888, 744). The following five compounds:—

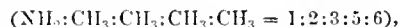
Two orthoxylinides—



two cumidines—



and duridine—

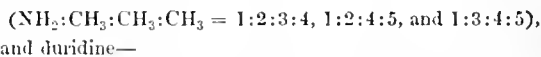


were submitted to oxidation with arsenic acid, alone and in conjunction with *p*-toluidine, asymmetric-*m*-xylylidine, and mesidine (cumidine). Magenta was not in any case obtained, but, instead, yellow or violet-grey dyestuffs, presumably of the acridine class. It therefore appears that the presence of a methyl group in the meta position to the amido group in an amine which has its para position free, renders it incapable of forming magenta when oxidised with a paramethylated amine. The author, further, decides the question as to the behaviour on oxidation of the meta-methylated derivatives of the paramethylated amines of the first group in Rosenstiehl and Gerber's classification. Experiments were made with the following bases:—

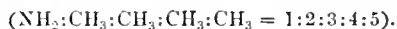
Paraxylylidine—



three cumidines—



and duridine—



All of these, when oxidised in conjunction with aniline, orthoxylylidine, or adjacent metaxylylidine, yield magenta in large quantity. It thus seems that a paramethylated amine is always suitable for making magenta, whether its meta position is substituted by a methyl group or not. Each of the ten paramethylated homologues of aniline, when oxidised together with two molecules of aniline, ortho-toluidine, or adjacent metaxylylidine, forms a magenta. Thus a series of 30 magentas is obtained. By taking equal molecules of two amines, that is, for instance, single molecules of aniline and ortho-toluidine, instead of two molecules of one of them, the number of magentas is increased to 60. But some of these are alike, so that the total number becomes reduced to 52. The following magentas are identical, namely, those obtained from:—

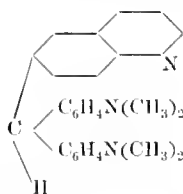
1. Metaxylylidine and 2 mols. of aniline = *p*-toluidine, *o*-toluidine, and aniline.
2. Mesidine and 2 mols. of aniline = *p*-toluidine, adjacent *m*-xylylidine, and aniline.
3. Metaxylylidine, *o*-toluidine, and aniline = *p*-toluidine and 2 mols. of *o*-toluidine.
- 4 and 5. Mesidine, *o*-toluidine, and aniline = *m*-xylylidine, adjacent metaxylylidine, and aniline = *p*-toluidine, adjacent *m*-xylylidine, and *o*-toluidine.
6. Mesidine, adjacent *m*-xylylidine, and aniline = *p*-toluidine and 2 mols. of adjacent *m*-xylylidine.

7. Mesidine and 2 mols. of *o*-toluidine = *m*-xylydine, adjacent *m*-xylydine, and *o*-toluidine.

8. Mesidine, adjacent *m*-xylydine, and *o*-toluidine = *m*-xylydine and 2 mols. of adjacent *m*-xylydine.

II. *Combinations of Tetramethyldiamido-benzhydrol and -benzophenone.*—Since it appeared that magentas containing one metamethylated benzene nucleus can exist, it seemed probable that tetramethyldiamido-benzhydrol would condense with metamethylated amines whose para positions were free. Such was found to be the case, the leuco-bases of violet-blue colouring-matters being so produced. In the same way it was found that condensations could be effected with such metamethylated bases as monomethyl-*m*-toluidine, dimethyl-*m*-toluidine, diethyl-*m*-toluidine, diethyl-*p*-xylydine, and adjacent diethyl-*o*-xylydine. The tertiary bases just mentioned also condense with the ketone-base in presence of phosphorus oxychloride with the direct formation of blue violet colouring-matters. Finally, meta-chloro-, bromo-, and iodo-anilines condense with the hydrol-base forming the leuco-bases of violet dyes of slightly less bluish tone than those of the *m*-toluidine derivatives.

III. *Quinolyldiphenylmethane Colouring Matters.*—It has been shown by E. and O. Fischer that the substitution by an acetyl group of one of the hydrogen atoms in the amido group of tetramethyltriamidotriphenylmethane renders the shade of the resultant body green, whereas substitution by methyl increases the blueness of the violet dye. It seemed of interest to ascertain the effect of submitting tetramethyltriamidotriphenylmethane to Skraup's reaction to obtain, if possible, a body of the following constitution:—



This body was easily obtained. On oxidation it yielded a blue-green dyestuff, exceedingly fugitive to light, and consequently without technical value. It thus appears that the quinolination of the phenyl-amido group has the same effect as the acetylation of the same. It remains to be seen whether this quinoline green is identical with that obtained by condensing quinoline with tetramethyldiamidobenzophenone. The isomeric tetramethyltriamidotriphenylmethane produced by the reduction of metanitrobenzaldehyde-green yields a quinoline derivative which also on oxidation forms a green dyestuff.—E. B.

*Report No. I. On New Colouring Matters.* E. Knecht. Jour. Soc. Dyers and Colourists, 1889, 106—107.

THESE colours may be conveniently classified as (1) dyes used principally for wool and silk, and (2) dyes used principally for cotton, and again into substantive and adjective dyes.

#### 1. DYES USED PRINCIPALLY FOR WOOL AND SILK.

(a.) *Substantive Dyes.*—*Jet Black.*—This dye appears in commerce as a dark brown powder in two shades, G. and R., soluble in water with a dark violet colour. The aqueous solution of *Jet black G.* is turned green on the addition of dilute sulphuric acid, while ammonia turns the original violet solution redder. Stannous chloride produces a dark-coloured precipitate. *Jet black R.* yields with sulphuric acid a blue-black precipitate; ammonia produces no change, and stannous chloride a black precipitate. Both shades appear to be mixtures of dyes which resemble in their general reactions the direct cotton dyes.

The application of *Jet black* in wool dyeing is somewhat remarkable, inasmuch as it is best applied (when used as a self-colour) in a neutral bath containing about 20 per cent. of common salt. The dye-water is not exhausted. *Jet black* yields thus fine shades of black on wool, which are not

materially altered by exposure to light for one month, nor by treating for 15 minutes in a 1 per cent. soap solution at 60° C.

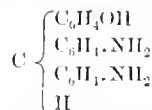
*Jet black* also dyes wool in an acid bath, and may, therefore, be used along with acid dyes for compound shades. When dyed on the fibre, shade G. is turned green when boiled with dilute sulphuric acid (1 : 10). Ammonia partially restores the colour. Stannous chloride turns the fibre reddish brown, and strong sulphuric acid extracts the dye with a blue colour. Nitric acid produces a dull yellow spot with a brown rim.

Shade R. gives somewhat different reactions. Dilute sulphuric acid turns the colour of the fibre bluer, which is partially restored by ammonia. Stannous chloride almost decolourises, while nitric acid produces a bright yellow spot with a brown rim.

*Indazine M.* (this Journal, 1889, 280) dissolves in water with a violet colour, which is not changed by either dilute sulphuric acid or ammonia. The solution is turned bluer on the addition of stannous chloride, and yields with bichromate of potash a black precipitate. Indazine is best applied on wool in a bath slightly acidulated with sulphuric acid, yielding blues somewhat like those obtained with the indulines in shade, which may not find much application as self-colours, but will be found useful in mixed shades. Indazine dyed on wool is turned redder by light; hot soap solution discharges the greater part of the colour.

When dyed on wool, indazine gives the following reactions:—Dilute sulphuric acid turns it bluer, but the colour is restored by ammonia. The colour is almost completely discharged by stannous chloride. Strong sulphuric acid turns the fibre green, and nitric acid produces a brown spot with a dark green rim.

*Patent Blue.*—This colouring matter comes into commerce in five different shades, in the form of blue powders, which would appear (according to Germ. Pat. 46,384, 1888) to be derivatives of meta-hydroxydiamidotriphenylmethane.



The leuco-bases of these colouring matters are obtained by the condensation of meta-nitrobenzaldehyde with tertiary amines, reducing the nitro compounds thus formed to amidotetra-alkyldiamidotriphenylmethanes, and transforming the latter by means of nitrous acid into the corresponding hydroxy compounds. They may also be formed directly by the condensation of meta-hydroxybenzaldehyde with tertiary aromatic amines. The hydroxy leuco-bases obtained by either of these methods are transformed into the corresponding colouring matters by oxidation in dilute acid solution with peroxide of lead or manganese.

Sulphonated dyes may be prepared by sulphonating the hydroxy leuco-bases and oxidising the lime salts of the sulphonic acids with peroxides and the theoretical quantity of acid.

Patent blue dissolves in water with a peacock-blue colour, which is turned to a yellowish-olive on the addition of dilute sulphuric acid; while ammonia has no action.

On wool, patent blue produces beautiful green shades of blue, which are remarkable, not only for their beauty, but principally for their fastness (compared with other triphenylmethane derivatives) to light and soap. The patterns which the author exposed to light were only slightly dulled after a month's exposure, and towards soap showed themselves very much superior to indigo extract.

Dyed on wool, Patent blue gives the following reactions:—Dilute sulphuric acid turns the fibre green; the original colour is partially restored by ammonia. Nitric acid produces a bright yellow spot with a green rim. Strong sulphuric acid turns the wool green and, on standing, yellow.

*Azo-carmin* appears to be a sparingly soluble azo dye which comes into commerce as a red orange paste, soluble with an intense magenta colour in ammonia. The colour is reprecipitated on acidulating with dilute sulphuric acid. In concentrated sulphuric acid it dissolves with an intense

emerald green colour. It is dyed on wool in an acid bath, and yields shades similar to acid magenta, but much faster to light, and especially to soap. It is useful in the production of compound shades.

(b.) *Adjective Dyes*.—Several new alizarin colours have recently been brought out which appear to be formed from ordinary Alizarin blue, by treatment with concentrated or fuming sulphuric acid. Nothing has, however, been published so far on the composition of these new products.

*Alizarin Green*, S.W. comes into commerce as a thick orange-brown liquid, soluble in water with a brown colour. Dilute sulphuric acid produces a dark green precipitate on boiling, and leaves the liquid light red, which becomes blue on addition of ammonia. Caustic soda produces a dark blue solution in the cold, but on boiling a dark green precipitate is formed. Stannous chloride gives a violet precipitate.

On wool, mordanted with bichromate of potash and tartar, Alizarin green yields a somewhat dull and not very intense shade of blue-green which is useful in certain compound shades.

Dyed on wool, Alizarin green gives the following reactions:—Dilute sulphuric acid removes at the boil some of the colour; ammonia turns it greener. Stannous chloride turns the colour to a light dull violet. Stannite of soda (stannous chloride dissolved in excess of caustic soda) produces a dull violet, but on washing and exposing to the air the original colour is restored. Strong sulphuric acid turns the colour blue, and nitric acid produces a yellow spot with dull violet border.

*Alizarin Indigo-blue*.—This colour comes into commerce as a thick red-brown liquid, in two shades, which are distinguished by the marks S.W. and S.M.W. The mark S.M.W. gives the following reactions:—It dissolves in water with a brown-red colour. On the addition of dilute sulphuric acid and boiling, a dark blue precipitate is formed. Caustic soda turns the solution blue, stannous chloride violet.

Alizarin indigo-blue S.M.W. produces on wool, mordanted with bichromate of potash and tartar, a shade of blue which is probably the nearest approach to indigo which has hitherto been produced with alizarin colours. The shade undergoes no appreciable alteration under the influence of light or soap. The mark S.W. produces a greener shade.

Dyed on wool, Alizarin indigo blue S.M.W. gives the following reactions:—Boiled with dilute sulphuric acid, the liquid becomes light red, which is turned blue again on rendering alkaline with ammonia, the fibre turning slightly greener; some colour is dissolved off. Ammonia has no action. Stannous chloride turns the colour violet. Stannite of soda only partially removes the colour; the solution is light violet. Nitric acid gives a brown spot with a small yellow centre. Strong sulphuric acid turns the fibre green. The reactions shown by the S.W. shade were similar, except that stannous chloride was without action, and strong sulphuric acid gave a light blue.

*Alizarin Yellow*.—Several shades of this dye are in the market, but only two will be described here. Alizarin yellow would appear from its behaviour to be an insoluble azo dye. The shade marked R.W. forms an orange-coloured paste, sparingly soluble in water, but easily soluble in caustic soda with a deep orange-red colour, from which solution the colour is precipitated on the addition of an acid. Stannite of soda turns the alkaline solution brown. This shade produces on wool mordanted with bichromate of potash and tartar an orange somewhat similar to Alizarin orange in shade, but brighter. It is not very fast to light, but stands soaping fairly well.

The Alizarin yellow G.G.W. is a yellow paste, sparingly soluble in water, but easily soluble in caustic soda with a yellow colour. Stannite of soda has apparently no action on the alkaline solution. On a chrome mordant this shade yields on wool a yellow somewhat similar to fastie, but not so fast to light.

When dyed on wool, the Alizarin yellows are turned light yellow by dilute sulphuric acid, the colour being partially restored by ammonia. Stannous chloride turns the fibre

light yellow. Strong sulphuric acid turns the fibre more orange. The most characteristic reaction is that with nitric acid, which produces a yellow spot with a broad scarlet rim.

## 2. DYES USED PRINCIPALLY FOR COTTON.

(a.) *Substantive Dyes*.—*Carbazol Yellow*.—This colouring matter is produced by the action of 2 molecules of salicylic acid on tetrazo carbazol. The commercial product dissolves in water with a dark orange colour. Dilute sulphuric acid produces an olive green precipitate. Ammonia renders the colour of the solution a little brighter. Bichromate of potash turns the solution brown.

Carbazol yellow dyes cotton in an alkaline bath yellow. On wool mordanted with bichromate of potash it yields shades similar to those obtained with fastie, but they are not so fast to light as the latter. Dyed on the fibre, Carbazol yellow gives the following reactions:—Dilute sulphuric acid gives an olive green, which is partially restored to the original colour by ammonia. Nitric acid produces an orange spot with a dark blue rim. Strong sulphuric acid turns the fibre dark blue.

*Cotton Yellow G*. dissolves in water with a dirty orange colour. Dilute sulphuric acid produces a red brown precipitate. Caustic soda renders the aqueous solution more orange.

On cotton this colour produces a canary yellow in an alkaline bath.

In this dyed state, cotton yellow is turned greener by boiling with dilute sulphuric acid, the colour being partially restored by ammonia. Nitric acid produces a brown spot with a violet rim.

*Mikado Orange 4 R*. dissolves in water with an orange colour. Dilute sulphuric acid turns the colour to an olive yellow; ammonia has no action. In strong sulphuric acid it dissolves with a pure blue colour.

Mikado orange dyes cotton in a neutral or slightly acidulated bath a fiery orange. It shows at the same time very little affinity for the animal fibres. Thus, when cotton and wool are immersed in the same bath acidulated with sulphuric acid, the shade produced on the cotton is much more intense than that produced on the wool.

Mikado orange is formed by the action of glycerol on paranitrotoluene sulphonic acid.

*Mikado Orange G*. and *Mikado Brown*, judging from their behaviour towards strong sulphuric acid, appear to be mixtures.

*Erika* dissolves in water with a pink colour, which is not changed by dilute sulphuric acid or ammonia. Stannous chloride produces a red precipitate. On unmordanted cotton, Erika produces a magnificent pink in an alkaline bath.

Dyed on the fibre, Erika is turned red by dilute sulphuric acid; the original colour is restored by ammonia. Stannous chloride partially removes the colour. Nitric acid produces an orange spot. Strong sulphuric acid turns the fibre blue.

*Thioflavin S*. (see below under "Thioflavin T.") comes into commerce as a yellow powder readily soluble in water with a golden yellow colour. Dilute sulphuric acid produces an orange precipitate; ammonia is without action. Strong sulphuric acid dissolves it with a yellow colour with beautiful blue fluorescence.

Thioflavin S. is applied on cotton, like most of the other direct cotton dyes, in an alkaline bath (soap and soda or phosphate of soda), and produces a bright canary yellow. The bath is only partially exhausted, and should be kept.

(b.) *Adjective Dyes*.—*Thioflavin T*. is one of a class of new yellow dyestuffs, consisting of the ethylated or methylated derivatives of dehydrotoluidine (Dahl's thio-toluidine or primuline base). The portions insoluble in water which are formed are converted into Thioflavin S. by sulphonation.

Thioflavin T. comes into commerce as a yellow powder, which dissolves in water with a yellow, somewhat turbid colour. Sulphuric acid clears the turbidity and renders the solution light yellow. Ammonia produces a light yellow precipitate; likewise tannic acid in presence of sodium acetate.

Wool is dyed in a neutral bath, silk in a bath with Marseilles soap. The shades produced on silk are very bright, and show a decided greenish tint. Cotton is previously mordanted with tannic acid and tartar emetic. Very bright yellows are obtained on cotton, which are said to stand light well.

Report No. II. Jour. Soc. Dyers and Colourists, 1889, 170-171.

### 1. DYES USED PRINCIPALLY FOR WOOL AND SILK.

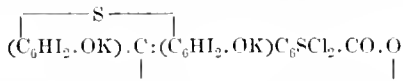
(a.) *Substantive Dyes.*—*Roxamine.*—This dye comes into commerce as a dark brick-red powder, easily soluble in water, with a scarlet colour. The addition of dilute sulphuric acid to the aqueous solution causes no change, but the colour is turned much bluer on the addition of ammonia. In strong sulphuric acid the dye dissolves with a violet colour.

Roxamine is dyed on wool with sulphuric acid and sulphate of soda, and produces a rich shade of scarlet. From its behaviour it appears to be an azo-dye, but it differs from most of the ordinary reds inasmuch as it dyes the wool thoroughly and evenly, while, at the same time, the bath is exhausted. The colour produced on wool is fast to light, but not to soap.

Dyed on wool, roxamine gives the following reactions:—Dilute sulphuric acid has no action. Boiling with dilute sodium carbonate strips the colour. Strong sulphuric acid produces a violet colour, and strong nitric acid an orange spot with a blue rim.

*Cyclamine.*—This colouring matter belongs to the eosin class, but contains sulphur as an essential constituent.

Its constitution is probably represented by the formula—



It comes into commerce as a powder which dissolves in water with a magenta colour without fluorescence. Dilute sulphuric acid completely throws down the free acid as a scarlet precipitate. Ammonia has no action. In strong sulphuric acid the dry colouring matter dissolves with an intense orange colouration. Zinc dust and ammonia completely decolourise the aqueous solution, which on exposure to the air gradually becomes pink, with a green fluorescence.

Cyclamine dyes wool or silk in a neutral bath, producing a beautiful, very blue shade of red, resembling that of the blossom of the ordinary wild *Cyclamen* (from which the name of the dye is presumably derived). The colour is not changed much in soaping, but, like most other eosins, it is not fast to light.

Dyed on wool Cyclamine changes to a light pink on boiling with dilute sulphuric acid, and its original colour is restored by ammonia, but, if boiled with the latter reagent, the colour is partially extracted. The fibre is turned orange-red by strong sulphuric acid, and strong nitric acid produces a light orange spot.

(b.) *Adjective Dyes.*—*Cloth brown* (red shade).—This is a claret-brown powder, soluble in water with an orange-red colour. The colouring matter is completely thrown down by dilute sulphuric acid as a scarlet precipitate. Ammonia has no action. Stannite of soda decolourises it. In strong sulphuric acid the dry powder dissolves with an intense reddish-violet colour.

*Cloth brown* dyes wool directly in a bath acidulated with sulphuric acid, but the makers recommend that the material should be previously mordanted with 3 per cent. of bichromate of potash, and 1 per cent. of sulphuric acid, and that, in dyeing, acetic acid should be added to the dye-bath. In this manner bright brick-red shades are obtained.

Dyed on wool with a chrome mordant, *Cloth brown* is turned somewhat lighter on boiling with dilute sulphuric acid. The colour bleeds slightly in dilute sodium carbonate. By strong sulphuric acid it is turned dark violet, and strong nitric acid produces a brown spot with a black rim.

*Cloth brown* (yellow shade) comes into commerce as a black powder, which is soluble in water with a dark brown colour. It is completely precipitated by dilute sulphuric acid. Ammonia has no action, and stannite of soda decolourises it. Bichromate of potash occasions a light brown precipitate. In strong sulphuric acid the dye dissolves with a dull reddish-violet colour.

*Cloth brown* (yellow shade) is dyed on wool like the red shade, and produces on a chrome mordant a snuff-brown.

On wool the colour thus produced is very little affected by boiling with dilute sulphuric acid, and not at all by dilute sodium carbonate. Strong sulphuric acid turns the fibre dark violet, and strong nitric acid produces a brown spot with a violet rim.

*Cloth orange* forms a light chocolate powder, soluble in water with an orange colour. It is completely thrown down from the aqueous solution by dilute sulphuric acid, as an orange precipitate, whilst ammonia has no action. Stannite of soda decolourises the aqueous solution, and bichromate of potash produces in it an orange precipitate. In strong sulphuric acid it dissolves with an intense reddish-violet.

*Cloth orange* is dyed on wool like the *Cloth brown*, and produces on a chrome mordant a dull orange (brighter, however, than Alizarin orange).

The dyed fibre is not changed by boiling with dilute sulphuric acid, but in boiling dilute soda solution it bleeds slightly, with a yellow colour. Strong sulphuric acid colours the fibre violet, while strong nitric acid produces a brown spot with a black rim.

*Cloth orange* and the two shades of *Cloth brown* are all nitrogenous compounds, and judging from their general behaviour belong to that class of coal-tar colours which are generally known as the "benzidine colours," but which might more appropriately be termed "direct cotton dyes." Indeed, although the *Cloth browns* do not give good colours on cotton, the author obtained an exceedingly fine shade of orange on cotton by dyeing it with soap and soda in 5 per cent. of *Cloth orange*. On wool, the colours obtained appeared at first somewhat uneven, but, after soaping well for about 15 minutes in a 1 per cent. soap solution at 60°, they became perfectly even, and possessed a full appearance. This mode of application renders them suitable for remixing with other adjective dyestuffs. Unfortunately, they are not very fast to light.

*Gallamine Blue.*—This colouring matter comes into commerce as a dirty green paste, and is formed by the action of gallaminic acid on nitrosodimethylamine. This paste is slightly soluble in boiling water with a blue colour. In dilute sulphuric acid it is somewhat more soluble with a red colour, while in ammonia it dissolves easily with a violet colour. In strong sulphuric acid the dried and powdered paste dissolves with a red colour, which in thin layers appears dirty green (dichroic).

Gallamine blue produces on wool, mordanted with chrome and tartar, a blue which somewhat resembles in shade that obtained with Galloeyanin, with which Gallamine blue is indeed closely related in its chemical composition. The colour produced is partly discharged by soap.

For printing on calico the following recipe is given:—The colour is made up with 10 litres Gallamine blue paste, 18 litres thickening, 4 litres chromium acetate (15° B.), and 1 litre magnesium acetate (15° B.). After printing, steam 1½ hours and wash.

Gallamine blue dyed on wool gives the following reactions:—When boiled with dilute sulphuric acid it is almost completely removed with a violet colour. Boiling with dilute sodium carbonate also removes the greater part of the colour. Strong sulphuric acid produces no change, while strong nitric acid produces a light brown spot.

It is not probable that Gallamine blue will meet with an extended application as a self-colour, but it may be found useful in mixtures.

### 2. DYES USED PRINCIPALLY FOR COTTON.

(a.) *Substantive Dyes.*—*Diamine Yellow.*—This dye comes into commerce as a dark orange paste, soluble in water with an orange colour. Dilute sulphuric acid produces a brown precipitate, ammonia no change in the aqueous



solution. The dried paste dissolves in strong sulphuric acid with a red-violet colour.

Diamine yellow, like Diamine red and Diamine blue, is a direct cotton dye, and is applied like most of the others, viz., in a concentrated vat rendered alkaline with soap and phosphate of soda, or soap and soda, or some other alkali. It produces a shade of yellow something like Chrysamine, which is rendered duller by boiling with dilute sulphuric acid, whilst sodium carbonate turns it orange. Concentrated sulphuric acid turns the fibre red-violet, and strong nitric acid produces a violet spot, which is lighter in the centre.

*Diamine Red N.* forms a red-brown powder soluble in water with an orange-brown colour. Dilute sulphuric acid produces a violet-black precipitate; ammonia is without action. In strong sulphuric acid the dye dissolves with a pure blue colour.

Diamine red N yields on cotton a full shade of red, which is turned by dilute sulphuric acid to a dull violet. Sodium carbonate restores the colour. Strong sulphuric acid turns the fibre blue, and strong nitric acid produces a light-coloured spot with a drab rim.

*Diamine Blue B.* comes into commerce as a brown-black powder soluble in water with a violet colour. Dilute sulphuric acid produces in the aqueous solution a bright blue precipitate, while ammonia turns it crimson. In concentrated sulphuric acid Diamine blue B. dissolves with a blue colour.

Dyed in the ordinary way on cotton, Diamine blue B. yields a somewhat reddish shade of blue, which remains unchanged when boiled with dilute sulphuric acid. Ammonia dissolves some of the colour off, the solution being light red, while the fibre is turned violet. Strong sulphuric acid turns the fibre blue, and strong nitric acid produces a flesh-coloured spot.

*Sulphone-azurin*, another direct cotton dye, comes into commerce as an indigo-blue powder, which in its reactions differs essentially from those already in the market. Even in boiling water it is only sparingly soluble. Dilute sulphuric acid turns the aqueous solution to a red-blue, which on boiling becomes red-violet. Ammonia produces no change in the cold, but the solution becomes redder on boiling. The aqueous solution is decolourised by boiling with stannite of soda. In strong sulphuric acid it dissolves with a crimson colour.

Sulphone-azurin is dyed on cotton in an alkaline bath, and produces the greenest shade of blue which has hitherto been obtained with the direct dyes.

On the fibre it is turned redder by boiling with dilute sulphuric acid; the original colour is restored by sodium carbonate. Strong sulphuric acid turns the fibre violet, and nitric acid produces a flesh-coloured spot.

(b.) *Adjective Dyes.*—*Indoline.*—This is a derivative of nitrosodimethylamine, and is formed by the action of nitrosodimethylamine on  $\beta$ -naphthol. The commercial product is the zinc chloride double salt with the chloride of the base. The same product has been in the market for some years under the name of *New blue*, or *Meldola's blue*. It is dyed on cotton mordanted with tannin and tartar emetic, or tannin and iron, and produces indigo-blue shades which are remarkable on account of their great fastness. Perhaps the greatest drawback which has attended the use of this colouring matter is the fact that its dust has such a violent action on the mucous membranes, causing sneezing and coughing when inhaled even in minute quantities. This drawback has now been overcome by a continental firm, which produces the colouring matter, not in powder, but in the form of well-defined crystals, under the name of *New blue crystals*.

*Chrome Violet.*—This colouring matter, which is used only in printing, is the ammonium salt of aurin tricarboxylic acid. It forms a reddish chocolate powder, soluble in water with a deep red colour, which is turned light brown by ammonia. Dilute sulphuric acid completely precipitates the colour. In strong sulphuric acid it dissolves with a brown colour. Chrome violet is printed on calico with thickening and chromium acetate, and the colour is developed by steaming. It is useful for mixtures.

## VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

*Oxycellulose.* E. Noeltzing. *Färb. Zeit.* 1889 (2).

By the action of oxidising agents on cellulose (cotton), products of oxidation are formed which have been called oxycellulose. This name is, however, given in common to two products, different in their nature, namely, the oxycellulose of Witz, and that of Cross and Bevan. In the *Bulletin de la Soc. Ind. de Rouen*, Witz has published the results of extensive and very interesting researches on the mode of formation and preparation of his oxycellulose. It is formed by the reaction of almost all oxidising agents: chlorine, acids of chlorine, chromic acid, dilute nitric acid, &c., on cotton. Thus the strength and power of resistance of the fibre is, on the one hand, considerably diminished, especially when the cotton, after oxidation, is treated with alkali or soap, whilst on the other hand the affinity of the fibre to basic colouring matters is increased to some extent, but it is lessened considerably for the sour and direct azo-colouring matters. The colourist is but too frequently disappointed in the results attained with material which has become attacked so as to form oxycellulose.

In the practice of printing, such an attack of the cloth is, however, observed not only under the influence of oxidising agents, but also under that of acids and sour salts. This is owing to the formation of the so-called *hydrocellulose*, the properties of which have been studied by Aimé Girard. Hydrocellulose does not possess the property of fixing basic colouring matters directly. In many instances, such as in the reaction of sour oxidising agents, the formation of oxy- and hydrocellulose possibly takes place simultaneously.

Under the name of oxycellulose Cross and Bevan have described a product that seems not to be identical with that of Witz. This "oxycellulose" is obtained by treating cotton on the water bath with nitric acid of 50 per cent., washing, dissolving in alkali, and reprecipitating with acid, a solution of salt, or with alcohol. The "oxycellulose" thus obtained does not possess the property of being dyed by basic colouring matters, either directly or after solution in alkali and reprecipitation.

The nitrocellulose obtained by the reaction of concentrated nitric acid or nitrosulphuric acid on cotton in the cold has, like the oxycellulose of Witz, the property of attracting basic colouring matters. The same is the case, as might be expected, with celluloid (a compressed mixture of gum-cotton and camphor); also the reaction products of sulphuric acid on cellulose, amyloid, and parchment paper are dyed in a similar manner.

Cellulose is not modified by dissolving in an ammoniacal copper solution and subsequent precipitation; the precipitated substance does not attract basic colouring matters, nor is there any oxycellulose formed in the preparation of pure cellulose by Hugo Müller's method (alternate treatment of the raw fibre with bromine water and ammonia).

Common bleached cotton shows a weak, but imperceptible, attraction to basic colouring matters. This is owing to traces of oxycellulose being formed in the treatment with chloride of lime. Hence cotton which is to be dyed with direct azo-colouring matters is not chlorinated.

*Mercerised* cotton attracts but very little. But if caustic soda, even in a very dilute state, is caused to act on cotton at an elevated temperature, and in the presence of air, the fibre is considerably weakened, and then attracts basic colouring matters. By the reaction of alkali, therefore, oxycellulose is formed, and not hydrocellulose as in the reaction with hydrochloric acid.

*On the Prevention of Stripiness in Woollen Stuffs Dyed in the Piece.* Otto Walther. *Färb. Zeit.* 1889 (2).

It has been proved by practical experience, that in consequence of the various manipulations to be applied to woollen stuffs, such as washing, pressing, wringing, hanging on rods, treatment in the hydro-extractor, &c., all the impurities of an oily or different nature are found in most cases to be unequally distributed, and that, consequently,



when dyeing the pieces, an equal attraction of the colouring matter cannot possibly take place. In fact the fundamental rule is too frequently neglected, namely, that every material intended for dyeing should be carefully cleaned and be subjected to the process of dyeing only in a clear state and without blemish. For wool in the flock, such an exquisite purity is apparently less necessary, at least as far as regards unequal dyeing, because the unequally dyed portions are so thoroughly mixed in the course of manufacture, by spinning and weaving, that at most the intended shade can only suffer a slight deviation. Now, it is the unanimous opinion of all authorities in the woollen industry, that to obtain perfectly pure stuffs a clean washing of the wool is an unconditional requirement, and hence the conclusion is absolute that wool intended for cloth to be dyed in the piece should be submitted to a most thoroughly and carefully conducted process of washing. There is nothing that favours more the occurrence of impurities in yarns and stuffs than the residue of wool fat adhering to the woollen fibre, and yarns and stuffs manufactured; wool badly cleaned of fat will never turn out quite pure, or at least not as pure as is necessary for a uniform appearance of the colours in the piece. It might be objected that very many yarns, and even cloths, are manufactured from unwashed wool, and were afterwards washed perfectly clean. But it should be observed that this is done mostly with inferior materials, and even then never for finer cloths.

A further circumstance, accounting partly for the more frequent occurrence of unequal colouring at the present time, is that a great part, if not the whole, of the wools now used for manufacture show a character different as to many points from that of the wool material of former times. This characteristic difference may be distinctly observed by anyone who, in addition to home-grown wools (formerly used exclusively), also manufactures with colonial wools. Thus the transmarine wools, especially those from the Cape, always have a greasy feel which cannot be removed even by the most thorough washing. This greasiness is either absent altogether in native wools, or possessed by them only to a very small extent. The occurrence of "stripiness" in such dyed wools is doubtless in great measure due to the fact just referred to. The fibre retains this property throughout all the stages of the manufacture, and thus it happens that also the yarns or cloths show little adhesion to water, and imbibe it with difficulty, whilst they as rapidly lose the quantity imbibed. Of course such greasy cloth works badly in the dye-bath, for the dye-liquor fills and penetrates the more wetted parts of the cloth or yarn far more rapidly than the others.

The defective purity of most washed Cape wools is the consequence of irrational treatment in washing, and the peculiarly greasy consistency natural to the fibre of those wools. It should, therefore, be made a rule that washed Cape wools should, before manufacture, be subjected to a thorough and most careful washing, *i.e.*, on the backs of the animals, care being taken as far as possible to avoid felting the wool. The chemical operation of removing the burrs, by means of acid and heat, the so-called "carbonising" applied to at least 50 per cent. of all woollen cloths dyed in the piece, contributes in no small measure to make the intricate and frequently quite unaccountable phenomena in dyeing, milling, and washing still more complicated. After the process of carbonisation a salt or this along with some acid, may be left behind in some cases, adhering to the fibre, and so much the firmer the more this fibre is contaminated with natural grease or other matters of greasy nature.

In consequence of the treatment in boiling water wool loses some of its elasticity and property of felting, and because both these qualities are desirable in spinning and fulling, it is better that the ready and dressed goods be subjected to the process of boiling, a practice which the author in most cases follows. If there be any suspicion about the purity of the goods a small quantity of potash may be added; if necessary this may also be done during the boiling. Where elasticity and faculty of felting are of less consideration it is still advisable to boil the wool before spinning, in order to a more complete purification of the goods from oil, soap, &c. The pieces, or even the yarns,

may before dyeing be effectually subjected to the same treatment. The chief point is that the wool, as well as the fabrics or yarns, shall be thoroughly rinsed immediately after boiling, when still warm, so that the dissolved substances may be removed before they again coagulate on the fibre.

Cloths are brought to the washing machine directly from the vat or the boiler, and rinsed clear after addition of fuller's earth, or they are brought from the boiling vat into a second vat filled with pure water that can be renewed from time to time. This second vat contains two pairs of squeezing rollers—one horizontal, the other vertical—close together, through which pieces sewed together to form an endless band are caused to pass until they are perfectly clean; that is, until they have lost their slippery feel. Instead of the two pairs of rollers a single horizontal pair may be mounted for the whole breadth of the cloth; but in this case the cylinders should be made to fit well, and so that they will not turn awry; one of them should also be covered with caoutchouc. For the same purpose the broad-washing machine, constructed by Hemmer, in Aix la Chappelle, may be employed, with the additional advantage that the washing may be done with fuller's earth, which is preferable to the simple squeezing and rinsing in the vat. Finer stuffs which have to undergo the finishing before the dyeing process, are boiled best before the finishing. This prevents the impurities from being fixed too firmly on the fibre by the finishing. For the preservation of the nap the goods may also be wound loosely on wooden cylinders, but should then, before cooling down on the cylinders, be immediately beamed off and brought still warm on the washing machine. With a view to a thorough cleansing, the boiling in an open boiler is, however, to be preferred. If this operation be carried out correctly, it will be found that the colours obtained are brighter and fuller, and that, besides, there is a saving in dyestuff. With unboiled goods, part of the colouring matter is lost through rinsing, in consequence of its being fixed less firmly on the fibre, so that the shade has frequently to be made somewhat deeper.

The chemicals used for carbonising and neutralising will now adhere to the fibre so much the less, the better the material has become freed from oil, soap, and alkalis. Their deteriorating effect in piece dyeing is, therefore, partially prevented. In consideration of the manifold complications arising from combinations between the residues of the acid or the neutralising substances with the soap applied to the wool, it is advisable to carbonise the cloths after fulling. This, however, is possible only with those kinds of material which, after fulling have still to undergo some mechanical process, such as raising or pressing, &c., as mechanical aid in the removal of the carbonised organic remains of vegetable origin cannot be well dispensed with.

#### *The Recovery of Tin in Dyeing* M. Martinon. *Ann. de la Soc. des Sciences Ind. de Lyon.*

THE weighting of silk with bichloride of tin is effected by immersing it in a solution of variable strength, but generally averaging from 20 to 28 B.

When the silk is well impregnated with this solution, it is wrung out, and, in order to get rid of the greater part of the bichloride, it may either be pressed in a special machine, which extracts only a small quantity of the liquid, or be hydro-extracted, the inside of the hydro-extractor being lined with lead, or covered with gutta-percha or hardened caoutchouc.

The silk is next washed in clean water, passed through carbonate of soda, and either soaped, or put again into the bath of bichloride of tin.

The following results follow from the washing of the silk.

The bichloride absorbed by the silk is decomposed at first partially, and afterwards entirely, and more or less rapidly, according to the proportions of the silk and water brought into contact. A certain quantity of the oxide of tin resulting from this decomposition remains fixed upon the silk, the rest spreads through the bath; it is the latter quantity that has hitherto been lost, and for the recovery of which the method to be described has been recently devised.

The first wash-waters of the silk are run into a large reservoir, and immediately after milk of lime is added to saturate the free hydrochloric acid, and to facilitate the deposition of the oxide of tin, the mixture being meanwhile agitated. Agitation by a current of air with a Koerting injector gives very good results. For the precipitation of this oxide of tin other bodies besides lime may be employed, such as the alkaline carbonates or alkaline earths. Tannin, which is often a residue in dye-houses, sulphuric acid even, and many other bodies can effect this precipitation.

For the proper treatment of these wash-waters there are required at least two reservoirs of suitable capacity. Whilst the liquid of one is being decanted, the other may be filled.

When the deposit has accumulated sufficiently, which takes place quickly enough if sufficient lime be used, and if the stirring be properly performed, the clear liquid is drawn off, and the precipitate thrown on to a series of filter beds.

This filtration of the oxide of tin takes place very slowly, and even after 24 hours only a paste containing 15 per cent. is obtained. This white paste may be treated in several ways.

It may be converted directly into bichloride, or reduced to the metallic state by well-known metallurgical processes. Whatever plan is adopted, the additional outlay is soon covered by the profit realised.

Thirty to 40 per cent. of the tin used may thus be recovered, and already the method is in operation in almost all the dye-houses at Lyons and at several other manufacturing centres.

The soap baths used in the soaping or boiling of the silk weighted with bichloride of tin are treated in exactly the same way; these deposits contain tin partially combined with fatty acids, the oxide of tin absorbed by the silk in its passage through these baths.

The deposits in the baths of catechu and of tin salts used in black dyeing may be treated similarly for the recovery of tin by metallurgical processes. The application of these processes furnishes at Lyons from 6 to 7 kilos. of metallic tin per month.

*The Chroming of Wool.* E. Knecht and A. Ward. Jour. Soc. Dyers and Colourists, 1889, 184-185.

A SERIES of experiments previously carried out by Messrs. P. Kay and E. Bastow with the author was published in a paper entitled "The Fixation of Chromium by the Wool Fibre."

The conclusions drawn from the results obtained were briefly as follows:—

1. The amount of chromium fixed by the wool fibre in mordanting with bichromate of potash varies with the concentration of the solution. When sulphuric acid is used along with the bichromate, the amount fixed is considerably greater. In mordanting with 3 per cent. of bichromate (the amount usually employed on the large scale) only about 1 per cent. is fixed by the wool.

2. In using pure bichromate of soda equivalent to 3 per cent. of bichromate of potash, a larger proportion of chromium is fixed than in the case of the potash salt, which might in some measure account for the different results obtained by practical dyers with these two mordants.

3. The addition of tartar to the mordant solution has no material influence on the amount of chromium fixed.

4. The amount of chromium fixed from chrome alum is about twice as great as that fixed from an equivalent percentage of bichromate. In this case the chromium is probably not so intimately combined with the fibre as when bichromate has been used as a mordant; besides, it here plays the part of a base, while in the latter case it plays the part of an acid.

5. In mordanting with bichromate of potash the amount of chromium fixed by the fibre is directly proportional to the time of immersion and to the temperature of the mordanting bath.

6. The fixation of chromium by wool in mordanting with potassium bichromate is always accompanied by a partial decomposition of the bath into chromate and chromic acid. Although the reaction is not a quantitative one, and is accompanied by a certain amount of mechanical absorption,

it seems probable that the keratine, or substance, of the wool plays here the part of a base, which forms with chromic acid an insoluble or sparingly soluble chromate.

7. In the practical working of a bichromate bath, the amount of chromium contained in the bath must necessarily increase according to the number of times the bath has been used. Thus if 3 per cent. of bichromate are used in the first bath, 1 per cent. is absorbed by the wool, 2 per cent. remaining in solution. On adding 3 per cent. for the second bath, we have a total of 5 per cent. bichromate in solution. If we now assume that 1 per cent. of this is again absorbed in mordanting the second lot of wool, we shall have left in solution after chroming for the second time an equivalent equal to  $5 - \frac{5}{3} = 3\frac{1}{3}$  per cent. bichromate of potash, so that in making up again with 3 per cent. for the third mordanting bath we shall actually have an equivalent to  $6\frac{1}{3}$  per cent. bichromate of potash. In this manner the bath must necessarily become more and more concentrated until it has ultimately to be thrown out, and large quantities of bichromate are thus inevitably lost every day. Theoretically (and this is the most important practical issue of our experiments) the only rational way of chroming on an economical scale would be to use 3 or 4 per cent. of bichromate for the first bath, and then add to each consecutive bath just as much fresh bichromate as has been absorbed by the wool (about 1 per cent.) along with a sufficient quantity of sulphuric acid to convert the chromate formed, into bichloride again.

8. If we take the amount of chromium fixed by the fibre as a guide for the efficiency of the mordant, then by far the best chromium mordant for wool would be chromic acid. Next in value comes bichromate of potash and sulphuric acid, then bichromate of potash alone, and lastly chromate of potash.

#### ECONOMY IN THE USE OF CHROME.

The chief object of the present investigation was to confirm by experiments, carried out on the large scale, to what extent the percentage of chromic acid, as well as the alkalinity of a chrome bath, increased after continued use.

The experiments were performed on liquors which had been used for chroming Botany slubbing. There were nine slots of slubbing chromed before a liquor was run off down the drain.

In the following, No. 1 liquor represents the liquor left in the cistern after the first panful of wool had been chromed; No. 2 liquor, the liquor left in the cistern after the second panful of wool had been chromed; and so on, up to No. 9 liquor. The chrome used was crystallised bichromate of soda.

For the 1st panful 5.6 per cent. of chrome for the weight of wool was used.

For the 2nd panful 4.4 per cent. of chrome for the weight of wool was used.

For the 3rd panful 4.4 per cent. of chrome for the weight of wool was used.

For the 4th panful 4.4 per cent. of chrome for the weight of wool was used.

For the 5th to 9th panfuls 4.0 per cent. of chrome for the weight of wool was used.

First, the total amount of chromic acid was estimated in the nine liquors by means of ferrous ammonium sulphate. In each case three titrations were made, and the figures were calculated from the mean of the three. The following figures were obtained:—

100 LB. WOOL to 630 GALS.

Pounds in 630 Galls. abstr.	Per Cent. $K_2Cr_2O_7$		Grms.	Difference.
4.21	1.36	No. 1 liquor contains 0.6567 $CrO_3$ per litre		..
5.73	2.91	No. 2	0.9168	0.2541
7.27	2.86	No. 3	1.1550	0.2442
8.68	2.89	No. 4	1.3629	0.2079
9.52	3.16	No. 5	1.5114	0.1485
10.31	3.21	No. 6	1.6368	0.1254
11.23	3.08	No. 7	1.7829	0.1452
12.37	2.86	No. 8	1.9635	0.1815
13.6	2.77	No. 9	2.1582	0.1747

Although these figures do not coincide exactly with those obtained by experimenting on the small scale, they are, nevertheless, analogous, and are of sufficient interest to woollen and worsted dyers, inasmuch as they show that in running off of an old chrome bath a considerable quantity of the mordant is lost.

That the bichromate is actually converted into chromate in working on the large scale is proved by the following estimations, made with laemoid as indicator, with the same liquors as those already referred to:—

Liquor required for the conversion of the chromate formed into bichromate.

No. 1 liquor, per litre	0.0980	grms. sulphuric acid.
No. 2     "     "     "	0.2009	"     "     "
No. 3     "     "     "	0.2949	"     "     "
No. 4     "     "     "	0.3773	"     "     "
No. 5     "     "     "	0.4655	"     "     "
No. 6     "     "     "	0.5488	"     "     "
No. 7     "     "     "	0.6027	"     "     "
No. 8     "     "     "	0.6713	"     "     "
No. 9     "     "     "	0.6909	"     "     "

From these latter results it cannot only be shown to what extent the alkalinity of the mordanting solution increases, but also in the following table the relative proportions of chromate and bichromate left in solution after each operation, in grms. per litre:—

—	Total $\text{CrO}_3$ .	$\text{CrO}_3$ as Chromate.	$\text{CrO}_3$ as Bichromate.
No. 1 .....	0.6567	0.2008	0.4559
No. 2 .....	0.9108	0.4116	0.4992
No. 3 .....	1.1550	0.6924	0.5526
No. 4 .....	1.3629	0.7730	0.5899
No. 5 .....	1.5114	0.9338	0.5776
No. 6 .....	1.6368	1.1244	0.5124
No. 7 .....	1.7820	1.2349	0.5471
No. 8 .....	1.9635	1.3754	0.5881
No. 9 .....	2.1582	1.4156	0.7427

This table shows that, whereas the amount of bichromate left in solution remains about constant at 0.5 grm. per litre, the amount of chromate steadily increases until, when the bath has to be let off, it contains an equivalent of no less than about 3.237 grms. of crystallised bichromate of soda per litre. There are thus let off into the drains from a vat of, say, 800 galls. capacity, somewhat less than 26 lb. of chrome, and this must not only mean a loss to the dyer, but must also tend to pollute the watercourse to a very considerable extent.

But now that the disease has been pointed out a remedy must be also suggested, and, if possible, that remedy should be of the simplest kind. Such a remedy is the following:—

By adding to the second "hole-full" just that amount of sulphuric acid necessary to convert the chromate formed into bichromate—viz., 0.098 grm. of sulphuric acid per litre of liquid in the bath, it would be brought back to the same condition as it was in when started; and it would not be necessary to add to the second bath any more chrome than had combined with the fibre, viz., about 1 or  $\frac{1}{2}$  per cent. The same would have to be done with the third and subsequent baths. In this manner the vat could probably be got to work for a greater number of times in succession, and on letting off, say after the ninth time, the loss in chrome would be considerably less than one-half of what it is at present.

Practical experience has shown that a chrome bath works better after it has been used two or three times than when fresh. In this case—that is, if the dyer specially wishes to have his chrome bath alkaline—the same principle might be adopted, and the bath kept on the alkaline side till the end by only commencing to add the necessary amount of sulphuric acid after, say, the third hole-full. The loss in chrome on letting off would, indeed, be somewhat greater than if

sulphuric acid were used from the beginning; but there would, nevertheless, be a very considerable saving as compared with the process referred to.

In proposing this method there was no desire to suggest that every dyer would have to be able to analyse quantitatively the contents of his chrome vat. A much simpler remedy is at hand. He has only to add bichromate to the first three hole-fulls as usual, but for the fourth and subsequent hole-fulls he must use a mordant made up in solution, and containing besides the bichromate of potash or soda the necessary quantity of sulphuric acid to keep his subsequent baths of a standard strength.

Some dyers use sulphuric acid in chroming over and above the amount necessary to neutralise the alkalinity of the water, and thus practically chrome with a solution of chromic acid. To these cases the method of the authors does not apply. They are of opinion, however, that also by using excess of sulphuric acid the loss of chrome in letting off would be very considerably lessened.

*The Theory of Chroming.* E. Knecht. *Jour. Soc. Dyers and Colourists*, 1889, 186.

WHEN wool is boiled in a solution of bichromate of potash or bichromate of soda, it absorbs, as is well known, a certain proportion of chromium and becomes yellow. Now this absorption is not due, as is held by some chemists, to the simple mechanical absorption of unchanged bichromate, but is due, according to the opinion expressed two years ago by the author, to a splitting up of the bichromate molecule into chromate and chromic acid. The chromate remains in solution, while the chromic acid combines chemically with the substance or a constituent of the substance of the wool fibre to form an insoluble or sparingly-soluble yellow chromate. This will be inferred from the following observations:—

1. When wool is boiled in a weak solution of bichromate of potash, the colour turns from orange to yellow.

2. Wool dissolved in strong hydrochloric acid and then diluted with water gives with bichromate of potash a thick yellow precipitate. A similar precipitate is obtained with lanuginic acid, and it is probable that this precipitate is chemically closely allied to the insoluble compound produced on or in the wool-fibre in mordanting.

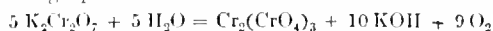
It has been argued that the formation of chromate in mordanting wool with bichromate is not due to the dissociation of the salt, but to the generation of ammonia from the wool-fibre. In order to test the correctness of this assertion, wool was boiled for an hour in a flask with water, and the gases given off were collected in another flask containing cold distilled water. All the ammonia which might have been produced must now be contained in the receiving flask; but on testing the liquid with Nessler's reagent we found it to contain only the merest trace of ammonia, less than one thousandth part of what would have been necessary to effect the chemical change referred to. It might further be argued against our theory that the neutralisation of the bichromate is due to the presence of alkaline salts in the wool-fibre. But this argument must also fall to the ground when the results of the following experiment are taken into consideration:—

Wool was first boiled with excess of dilute hydrochloric acid, for the purpose of removing any salts of the alkalis or alkaline earths. It was then boiled repeatedly in distilled water until no more traces of acid could be detected. Five grms. of the wool thus treated were now mordanted with 4 per cent. of bichromate of potash (0.2 grm.). The bichromate and chromate left in solution were estimated volumetrically, and the chromic acid absorbed by the wool by difference. The following results were noted:—

	Grms.
Total bichromate in solution.....	0.630
" chromate     "     "	0.112
Chromic acid in the wool .....	0.057
	<u>0.199</u>

This shows that the reaction is independent of any alkaline inorganic salts which may be contained in the wool.

Nietzki's assertion that in chroming wool the chromium is fixed as chromate of chromium requires proofs which are as yet not forthcoming. If the bichromate should be reduced to chromium chromate, the reaction would be attended by the formation of caustic alkali, and a very considerable oxidation of the fibre, as will be seen from the following equation:—



It is well known that chromed wool becomes oxidised when exposed to light; this is shown by the change in colour from yellow to green. But that such a powerful oxidation as that shown by the above equation should take place in the ordinary process of chroming seems highly improbable.

#### *A Simple Method of Producing "Green-Mordanted" Wool.* E. Knecht. Jour. Soc. Dyers and Colourists, 1889, 186—190.

In using the alizarin colours, as well as some of the other adjective colours, on a chrome mordant, it is generally desirable, indeed sometimes necessary, that the chromium should not be present on the fibre as chromic acid, but as chromic oxide. This result can be attained by mordanting with chrome alum or chromium fluoride; but the results are not good in the former case, while chromium fluoride, although yielding excellent results as a chrome mordant for wool, it is yet too expensive. The most usual way of obtaining "green-mordanted" wool is to use along with the chrome either tartar or oxalic acid. As the chromic acid becomes fixed on the fibre it is reduced to the green state at the expense of the tartaric or oxalic acid, which in their turn become more or less completely oxidised. This method has been found to give very good results in the hands of practical men, but the quantities of tartar and oxalic acid usually employed (2½ per cent. tartar, or 3 per cent. oxalic acid to 3 per cent. chrome) are not by any means sufficient to completely reduce the chromic acid on the fibre to chromic oxide.

At the same time the method for economising chrome which we mentioned in the first part of our paper could not be used, since the chrome bath becomes partially reduced by the presence of tartar, and this reduction would only be accelerated by the addition of sulphuric acid.

Now if wool mordanted in the usual way, either with bichromate alone, or with bichromate and sulphuric acid, be passed through a solution of bisulphite of soda or some similar reducing agent (but the bisulphite is the cheapest), the change from the yellow to the green very soon takes place. As the result of a number of experiments the author finds that it is best to chrome with 3 per cent. of bichromate and 1 per cent. sulphuric acid, or thereabouts, and reduce in a tepid or warm bath containing not more than about 5 per cent. of commercial bisulphite solution (sp. gr. 1·3).

Treated in this manner the chrome on the wool becomes more completely reduced than if tartar had been used in chroming. There is certainly the trouble of preparing a fresh bath, but the difference in price between the tartar and the bisulphite would compensate for this. Besides, the results obtained in dyeing are much better with a large number of adjective dyes, notably logwood, alizarin blue, cœrulein, and gallein.

In the discussion which followed, the President, Mr. Jas. Sharp, said he thought that 4 or 5 per cent. of "bichrome," referred to by Dr. Knecht, which was certainly a maximum amount, might probably be cut down to one-half and still give all the results necessary. If so, about half the bichrome at present used finds its way down the sewers. To produce brighter or more bloomy shades, a little sulphuric acid was now found an advantageous addition to the bichromate bath. Nevertheless, the use of sulphuric acid must be graduated with caution on the dye on the warp "will go." Sometimes tartar or oxalic acid is used with the bichrome.

Mr. A. H. Allen desired to know the action of normal chromate and bichromate upon laemoid, more especially which of these salts is neutral to laemoid and which is not.

He gathered that the point of strict neutrality to laemoid was at the formation of bichromate, the chromate neutral as to composition being alkaline as to reaction. (Confirmed by Dr. Knecht.) He considered it irrational to add tartarate to bichrome, and that people doing so got their fair results in spite of what they did, and those results would be better under more rational conditions. Was it a fact that so much bichromate was wasted?

Mr. Wilkinson, referring to the author's recommendation to add sulphuric acid after each batch of wool is taken out of the vat, said that when dyers mordant with bichrome, if they get it in the condition of chromic acid through the addition of sulphuric acid, the fibre becomes undoubtedly injured, and slubbing dyers in general preferred to keep on the side of the chromate than on that of the free chromic acid. That acid is also injurious to the colours, and its activity in their direction causes those colours to be taken up so greedily that uneven shades are the result. Not only is its action energetic on the fibre, but it is also exceedingly active on the colouring matter. In the loose wool trade, such as that of Huddersfield, it is a very different matter. A large amount of sulphuric acid is used there simply to neutralise the amount of alkali that is brought forward from the scouring, because less attention is paid to the scouring process. Here again he thought it would be well worth the attention of some chemist to obtain a knowledge of the amount of alkali which is usually left in the slubbing. The amount of alkali left behind in different works will be found to vary, and consequently the chromate or bichromate will be found to vary in the mordant bath. He thought it better indeed to keep nearer to chromate than bichromate. A good deal of trouble had been caused in some kinds of alizarin dyeing through too active bichrome baths.

Mr. Rawson said that in many dyehouses, to his knowledge, the slubbing or yarn is taken direct from the scouring bowls, without any washing with water, into the chrome bath. The consequence is that the alkali in the wool at once reduces the bichromate of potash to normal chromate, and it would therefore be impossible to calculate the amount of acid necessary to be added without taking this factor into consideration. The amount of alkali thus introduced into the chrome bath would often be much more than that formed by the boiling of the wool itself. In the experiments on the large scale it appears that about 3 per cent. of potassium bichromate was removed by each successive lot of wool. This includes the bichrome removed by washing; but he would very much like to know what amount was actually fixed on the fibre. It was scarcely likely that as much as 1 per cent. was removed by washing, so that in all probability, accepting the figures given, more than 2 per cent. was fixed. This was, comparatively, a large amount. The quantity of chromium fixed on the fibre was a most important matter in connexion with the fastness to light of logwood blues and blacks. If more than a certain amount was present, the black, in a short time, was most likely to turn green. In all cases of logwood blacks which had turned green an excess of chrome had been found according to his experience. It was difficult to state a limit, since much depended upon the condition in which it exists; but, in his opinion, not more than 1·5 per cent. of potassium bichromate should be fixed upon the fibre. Where the goods are chromed after dyeing as well as before (with the object of making the wool clean), there was no doubt that part of the chromium was often fixed on the fibre as chromate, or chromic acid, and such being the condition, the black was very liable to turn green. It was most probable that in after-chroming or finishing by the use of too much bichromate of potash, the logwood black was rendered liable to turn green. If chromic acid is deposited on the fibre in the first chroming, it is in all probability reduced to chromic oxide when it comes into the logwood bath. Both the author and speaker had made many experiments in connexion with the action of sodium bisulphite on chromed wool, and in all cases had found that the shades produced by logwood were not only deeper when the chromed wool had been thus reduced, but very much brighter. The effect was especially noticeable in light shades. The patterns produced were each mordanted with 3 per cent. of potassium bichromate and 1 per

cent. of sulphuric acid, and dyed with 10 per cent. of logwood. Of course, this amount of bichrome was too large for practical operations, but it was taken in order to show the effect of chromic acid and chromic oxide respectively with greater distinctness. One of the patterns, after mordanting, was treated with 5 per cent. of sodium bisulphite solution in the manner described by Dr. Knecht. The difference was very striking; the hank treated with bisulphite was of a bright, bloomy blue shade, whereas the other was a dull, dirty grey. This latter effect can only be attributed to the colouring matter of logwood being partially destroyed by the chromic acid on the fibre.

Mr. Frusher desired to know whether Dr. Knecht had made any experiments with wool previously bottomed with indigo. In chroming on indigo-bottomed wool they had a certain loss of indigo in the ordinary mordanting bath with bichromate, and with the addition of sulphuric acid there was an increased loss of indigo. He desired to know whether there would be any greater effect with the use of sulphuric acid than without?

In reply, Dr. Knecht said that the amount of chrome used for his experiments was between 4 and 5 per cent. The material to be chromed was Botany slubbing, a substance which absorbed considerably more than woven or spun material. Wool of that quality also absorbed more chrome than any other wool—English wools, for instance. He had shown that after they had mordanted wool in potassium bichromate the bath became alkaline. If they mordanted another lot of wool in the same bath it became still more alkaline, and if they mordanted further lots the alkalinity became increasingly great. He suggested the use of sulphuric acid to counteract that growing alkalinity, but did not either use or suggest the use of that amount of sulphuric acid necessary to render the bath acid. He kept it on the alkaline side. He only added a sufficient amount of sulphuric acid to bring the chromate back to bichromate. He had never tried to completely exhaust a bichrome bath, but thought it might be done. The author thought that in the case of, indigo-bottomed wool, chroming with sulphuric acid would be much more injurious than chrome alone.

Mr. Rawson considered that much chromium existed on the fibre after chroming, in the form of chromic acid, but that it was reduced to chromic oxide in the dye-bath at the expense of the colouring matter of the logwood. A portion of the colouring matter was not only destroyed and lost, but the decomposition products were apparently fixed to a certain extent on the material, and a dull grey instead of a bright blue was obtained. With an excess of colouring matter, no doubt this grey became merged and lost in the excess of deep black obtained. When indigo-dyed cloth was chromed, the shade became duller and greener, and certainly appeared to have lost indigo.

#### PATENTS.

*Improvements in Dyeing Cotton or other Vegetable Fibres with Blended Colours.* J. Imray, London. From La Société Anonyme des Matières Colorantes et Produits Chimiques de St. Denis, Paris, France. Eng. Pat. 18,010, December 10, 1888. 4d.

To dispense with the usual preliminary mordanting in dyeing cotton with alizarin and analogous dyestuffs, the material is dyed at once in a bath containing the colour stuffs "along with the substances necessary to make them take on to the texture." As example, for dyeing mauve, 100 kilos. of cotton are immersed cold in a bath containing 10 kilos. of the following mixture diluted with 2,500 litres of water: 2 parts of Alizarin No. 1; 1 part of "silicate of soda, 45°"; 2 parts of pyrolignite of iron (specific gravity not stated). The dye-bath is gradually heated to the boiling point in half or three-quarters of an hour. Similarly a rose colour is produced from a mixture of alizarin, acetate of alumina, oil, and a salt of tin; green by substituting ceruleine and bisulphite of soda for the alizarin in the last mixture; and other shades from similar suitable mixtures.

—W. E. K.

*A Process for Dyeing with Yellow, Orange, and Red Azoic Colouring Matters.* J. Imray, London. From La Société Anonyme des Matières Colorantes et Produits Chimiques de St. Denis, Paris, France. Eng. Pat. 197, January 4, 1889. 4d.

Two colouring matters described in Eng. Pats. 9315 and 11,976 of 1887 (this Journal, 1888, 499 and 563), were there stated to dye unmordanted cotton in an alkaline bath. This patent relates to a process of dyeing found to give the best results, and is especially applicable to colouring matters derived from phenol, resorcinol, naphthol, and their sulphonic and carboxylic acids. The colouring matters are dissolved in water rendered strongly alkaline by an excess of caustic soda, and sodium chloride is then added up to the point at which the colouring matter is precipitated at a low temperature. In such a bath, when heated, cotton becomes perfectly dyed until the bath is almost exhausted; whereas if the addition of sodium chloride be omitted cotton can only be dyed in presence of great excess of the colouring matters. The sodium chloride may be substituted by other salts having a similar precipitating action. After dyeing (and without rinsing) the cloth is passed through acidulated water, which fixes the colouring matter. An example is given of the method of applying this process in the case of the red colouring matter obtained "by combining the diazoic derivative of an azoxytoluidine with one of the sulpho-naphthols."—W. E. K.

#### VII.—ACIDS, ALKALIS, AND SALTS.

*The Fixation of Atmospheric Nitrogen.* A. A. Breneman. Amer. Chem. Soc. J. 11, 1—45.

**PART I.—Historical Summary showing the Progress and Development of Processes for the Manufacture of Cyanogen and its Derivatives.**—Prussian blue was discovered about 1710 by Diesbach, who happened, in handling a sample of Dippel's animal oil made from blood, to add to it a solution of potash and obtained a blue colour. (Richardson and Watts' Chem. Tech., Vol. 1, p. 1 *et seq.*) Woodward, in 1724 (Phil. Trans. 1724, 33—34, 15—25) deflagrated saltpetre and argols, and fused the product with dried blood; the mass was lixiviated with water and the solution treated with copperas and alum, which gave a green precipitate, becoming Prussian blue when heated with hydrochloric acid. Brown, in the same year (*loc. cit.*) proposed the use of flesh, and Geoffrey in 1725 that of wool and charred horn.

Potassium ferrocyanide was first made by Macquer (1750—1760?) who treated Prussian blue with potash. Beaumé, in 1773, proved the presence of iron in it, and Gay Lussac, in 1814, first showed the existence of cyanogen in Prussian blue and its derivatives. Liebig's investigation led to the theory at present held as to the chemistry of the process, still the only one in use, for making yellow prussiate. From 1820 to 1837 Muspratt, Mackintosh, Gautier (J. de Pharm. 1827, 11), Gentele (Dingl. Polyt. J. 129, 362), and Naumann improved the manufacture; but up to this time (1837) at least two-thirds of the nitrogen had been lost during the fusion, and the first attempt to recover this was made when Kuhlmann, in 1838 (J. prakt. Chem. 26, 410), discovered that cyanogen and ammonium cyanide were formed by passing ammonia over red-hot charcoal, a discovery which Jacquemyn applied to the waste gases from the prussiate pots; the process was patented by Berry (Eng. Pat. January 21, 1840).

*Cyanogen from Atmospheric Nitrogen.*—Zinken, in 1813 (J. prakt. Chem. 25, 246), and Koeb, in 1819, noticed a fused salt in the lower parts of some furnaces; Berthier, in 1826 (Jsb. Chem. Tech. 1879, 28), found potassium carbonate in a similar product, while Dawes, in 1835, announced the existence of potassium cyanide in such deposits. In 1837 Neilson first introduced the hot blast at the Clyde iron furnaces, and an exudation of fused salt

soon afterwards occurred on the walls of the hoshes; Clark showed this to contain 43.4 per cent. of KCN and 45.8 per cent. of  $K_2CO_3$  (Pogg. Ann. 40, 315; Dingl. Polyt. J. 65, 466). This furnace went on producing potassium cyanide for three years. Zinken and Bromels (J. prakt. Chem. 25, 246) and Redtenbacher also noticed the production of potassium cyanide in blast furnaces.

Bunsen and Playfair (Brit. Ass. Report, 1845) drew gases from a blast furnace at Alfreton at a point 2 ft. 9 in. above the tyeres, and found that they consisted of N, 58.65 per cent.; CO, 37.43 per cent.; H, 3.18 per cent.; C<sub>2</sub>N, 1.34 per cent. Potassium cyanide was also condensed from the gases on cooling. Above the hoshes, and as low as the tyeres, no cyanides were found, the conditions for their production being evidently a reducing atmosphere and a high temperature; the latter, Bunsen and Playfair put at not lower than that at which potassium oxide is reduced by carbon. Smith (Chem. News 1865; Jsb. Chem. Tech. 11, 54), and Kirpely (Jsb. Chem. Tech. 1879, 44) also describe fused furnace salts, and the former found one to contain potassium cyanate, 21.45 per cent.; potassium cyanide, 47.73 per cent.; and potassium carbonate, 10.13 per cent.

Lewis Thomson, in 1839 (Dingl. Polyt. J. 73, 281), proved that potassium cyanide is produced when coke, potash, and iron filings are heated together to a bright red in air, and Fownes and Young (J. prakt. Chem. 26, 407), in 1841, confirmed this statement, using carbon from cane sugar and pure potash. In the following year Erdmann and Marchand (J. prakt. Chem. 26, 412) repeated Fownes' experiments, with uncertain results; they asserted that no cyanogen was formed when the materials were dry; and Berzelius (Jsb. d. fortschr. d. Chem. 1844, 23) pointed out that this result confirmed the earlier result of Wöhler, and suggested that the water first formed ammonia, which was then converted into cyanogen. Langlois (Ann. Chim. Phys. [3], 1, 177) proved that cyanogen is formed whether the air be dry or wet.

Bunsen and Playfair (*loc. cit.*) passed nitrogen over a mixture of carbon and potash heated to bright redness, and obtained potassium cyanide. Rieken (Dingl. Polyt. J. 121, 286) found that a white heat was necessary. Diebrich (Jsb. Chem. 1, 473) showed that related reactions also cause combination of C and N:—thus nitric oxide and "potassium carbonic oxide" produced cyanogen when heated together; carbon dioxide, when mixed with ammonia or nitrogen, and passed over hot potassium, or ammonium carbonate kept in contact with fused potassium, yielded cyanogen.

A. V. Newton first patented a process for obtaining cyanides by passing N from any source, but free from "oxidating substances," through a layer of small pieces of charcoal saturated with potashes and heated to redness, the vapours containing cyanides to be absorbed by suitable liquids (Dingl. Polyt. J. 95, 293; Eng. Pat. 9985, December 13, 1843). He recommends the use of the waste gases from sulphuric acid chambers after passing through ferrous sulphate and lime water, to absorb oxides of nitrogen. The best proportion of potash to charcoal is from 25:100 to 100:100, according to the density of the latter; the nitrogen should always be in excess, and is best under pressure, much friction with the charcoal being advantageous.

Possoz and Boissiere (Jsb. Chem. Tech. 1855, 83; 1858, 191; Dingl. Polyt. J. 104, 446; 107, 444; 129, 361; 149, 56), before the date of Newton's patent, had a factory at Grenelle, Paris, which turned out 15,000 kilos. of yellow prussiate per annum manufactured on this principle. In 1844 they removed to Newcastle-on-Tyne, where they received the co-operation of Bramwell and Hughes, and in 1845 the output was more than a ton per day. These works were closed in 1847, after considerable loss. The cost of production was said to be less than 2 francs per kilo., and the salt of fine quality. In their latest form the retorts for this process were cylinders of firebrick 10 ft. long and 2 ft. in diameter, with walls 9 in. thick; lateral slits in the walls served for admission of air and stirring of the contents. The alkalis charcoal, which contained 30 per cent. of potash, was prepared by saturating the charcoal with potash solution, and was fed into the top of the cylinder, being thus dried by waste heat. The cylinders were kept at a white

heat and the gases pumped through them. The cyanised charcoal was withdrawn at the bottom and thrown into water containing spathic iron ore. The production of cyanide for a given weight of potash was said to be greater than in the ordinary prussiate process; soda was inferior to potash, and even small quantities of water hindered the production of cyanides. Failure was due to cost of retorts and volatilisation and other loss of alkali.

The patents of R. Laming, Swindell, and J. Laming (Eng. Pats. 9832, July 13, 1843; June 12, 1844; 10,955, November 18, 1845) describe methods similar to the above, but using other sources of nitrogen. Bunsen (Brit. Ass. Rep. 1845) proposed a furnace on the plan of the blast furnace, fed with alternate layers of alkali and charcoal.

Armengand (Dingl. Polyt. J. 120, 111; Genie Industriel, 1853, 315) and Eitel (Dingl. Polyt. J. 120, 77; French Pat. November 16, 1846) patented processes which differed from that of Possoz and Boissiere in the use of steam and a comparatively low temperature.

Marguerite and Sourdeval (Jsb. Chem. Tech. 1873, 361; Ber. 6, 79), in 1862, found that a hot mixture of baryta and carbon absorbed N from the air, forming barium cyanide, which yields ammonia in presence of steam at 300°. Messrs. Brin, in 1883 (Eng. Pat. 5802, December 18, 1883; also this Journal, 1884, 173), patented a process for making ammonia by passing N over heated coke containing baryta.

In 1868 Berthelot discovered the synthesis of HCN from  $C_2H_2$  and N; he concluded that potassium acetylene,  $C_2K_2$ , is first formed in the above cyanide processes, and that this then combines directly with N.

In 1878, Blair (Scientific American, 1878, 21; Dingl. Polyt. J. 230, 93) patented an apparatus, and in 1879 Weldon (Jsb. Chem. Tech. 1879, 472) a rotary furnace for the manufacture of cyanides by the charcoal-potash-nitrogen method. The latter found a bright red heat sufficient.

Alder, in 1881 (J. Chem. Tech. 1881; D.R. Pat. 1881, 12,351, and 1882, 509) claimed that the presence of finely-divided iron is beneficial, and used lime together with sulphides and sulphates of the alkali metals instead of potash.

Ludwig Mond (U.S. Pat. 269,309, December 19, 1882; compare this Journal, 1889, 506) in 1882 patented a process similar to that of Marguerite and Sourdeval (see above. Compare also Readman, this Journal, 1889, 758).

Fogarty, in 1883 (U.S. Pats. 288,323 and 402,324, November 13, 1883), dropped a mixture of powdered carbon and alkali into superheated furnace or generator gases, whereby cyanides were produced and subsequently decomposed by the steam present, yielding ammonia. This process has been tried on a large scale without commercial success.

Young, in 1884 (Eng. Pat. 16,046, December 6, 1884; also this Journal, 1886, 34), patented the use of magnesium limestone for constructing furnaces for cyanide processes.

Siepermann (Jsb. Chem. Tech. 1887, 675) obtained cyanates by passing ammonia over a mixture of barium carbonate with alkaline carbonate, adding coal if cyanides are wanted. He finds a moderate red heat best. Dickson (U.S. Pat. 370,768, October 4, 1887) injects a mixture of air, steam, coal-dust, and powdered alkali into a chamber heated by the combustion of the injected fuel, thus producing cyanides and ammonia.

**Formation of Metallic Nitrides.**—Wagner (Jsb. Chem. Tech. 1857, 121) suggested the nitrides of boron and silicon in the interior of the earth as the source of volcanic ammonia; but Bunsen and Rainer regard this ammonia as of organic origin.

Briegleb and Geuther, in 1862 (J. prakt. Chem. 123, 228), investigated metallic nitrides, obtaining those of magnesium, aluminium, and chromium by heating the metals in pure N. Magnesium nitride yields cyanogen when heated in CO or CO<sub>2</sub>. Tessie de Motay (Jsb. Chem. Tech. 1873, 279; Ber. 5, 395) found that titanium nitride yields ammonia when heated in steam, and can be regenerated by being heated in N.

**Cyanides from Oxides of Nitrogen.**—Blinks (J. prakt. Chem. 101, 359; 103, 230; 105, 69; 110, 248) passed hydrocarbon vapours mixed with nitric acid vapour or oxides of nitrogen over highly-heated firebrick. With hydro-



carbons in excess, cyanides are the chief products; with nitrogen compounds in excess, ammonia is mainly produced. Firman proposed to use gases from sulphuric acid chambers.

Roussin, in 1858 (*Compt. Rend.* **67**, 875; *J. prakt. Chem.* **78**, 375), suggested the deflagration of sodium nitrate and acetate as a source of cyanide.

*Production of Cyanides from Ammonia.*—Clouet, in 1791 (*Ann. Chim. Phys.* **11**, 30; *J. prakt. Chem.* **26**, 408; *Crell's Annalen*, 1796), mentioned that ammonia passed over hot charcoal yields hydrocyanic acid. Tromsdorff (*J. prakt. Chem.* **26**, 409) showed that the product was ammonium cyanide. Kuhlmann (*loc. cit.*; *Liebig's Annalen*, **38**, 62; *J. prakt. Chem.* **16**, 482) found that nitrogen compounds mixed with hydrogen or hydrocarbon vapours yield ammonia when passed, together with carbonic oxide, over red-hot platinum sponge.

Graeger (*Jsb. Chem. Tech.* 1858, 184) passed vapour of ammonium carbonate over alkaline charcoal in iron cylinders, 1½ in. diameter, and obtained 93–95 per cent. of the theoretical yield of ferrocyanide. The narrow cylinders were necessary. Brunquill, in 1856 (*Preuss. Verhandlungen*, 1856, 30; *Jsb. Chem. Tech.* 1856, 102; *Dingl. Polyt. J.* **140**, 374, 452), applied this principle by passing ammonia, or the vapours from distillation of bones, over hot firebrick and then over hot wood charcoal in pieces of the size of a chestnut. The ammonium cyanide was absorbed by solution of ferrous sulphate and the cyanides of iron converted by potash or soda into prussiates. Only moderate temperatures were employed.

Levoir, in 1859 (*J. prakt. Chem.* **76**, 447; *Dingl. Polyt. J.* **153**, 466), noticed that a flame of ammoniacal gas yielded cyanogen (ammonium cyanide), a statement questioned by Fischer (*Dingl. Polyt. J.* **157**, 466), and verified by Romily in 1867, who passed illuminating gas through ammonia and burned it, causing the flame to impinge on a surface of water containing potash (*Compt. Rend.* **64**, 320; **65**, 865; *Jsb. Chem. Tech.* 1867, 761); potassium cyanide was found in solution if the flame was smoky or luminous. The process was tried commercially.

Johnson (*Dingl. Polyt. J.* **156**, 212; *Jsb. Chem. Tech.* 1860, 221) and Webster (*Eng. Pat.* 1913, August 8, 1860) patented processes for making prussiate by passing ammonia over alkali charcoal.

Fleck, in 1863 (*Polyt. Centrbl.* 1863, 717; *Dingl. Polyt. J.* **169**, 209; *Jsb. Chem. Tech.* 1863, 323), proposed treating a hot mixture of charcoal or coal, sulphur, and potash with a definite quantity of ammonium sulphate; potassium sulphocyanate is formed and converted into potassium cyanide by metallic iron; 94.42 per cent. of the ammonium salts are claimed to be ultimately obtained as cyanides. Meyer, in 1874, asserted that the process was not practically successful.

Gelis (*Jsb. Chem. Tech.* 1862, 283) made prussiates by mixing cold solutions of ammonium sulphhydrate with carbon disulphide; the ammonium sulphocarbonate formed was converted by potassium sulphide into the potassium sulphocyanate, which yields prussiate when treated with iron. The cost was said by Payen to be 1.6 francs per kilo. of potassium ferrocyanide; but the process was condemned as wasteful by Meyer, and abandoned.

Schwarz (*Bull. Soc. Chim.* 1869, 167; *Dingl. Polyt. J.* **191**, 399; *Jsb. Chem. Tech.* 1869, 269) obtained ammonium cyanide by passing vapour of carbon disulphide mixed with ammonia over hot iron, or copper, in an iron tube. The cyanide was converted into Prussian blue and then into prussiate.

E. Meyer, in his review of the cyanogen industry up to 1873 (*Jsb. Chem. Tech.* 1874, 442), found that no method proposed up till that time had succeeded as well as that of Possoz and Boissiere, which, however, had failed to replace the original fusion method.

Maxwell-Lyte (*U.S. Pat.* 161,137, March 23, 1875) patented a process for making ammonia by bringing nitrogen in contact with nascent hydrogen, liberated in presence of a triad or pentad element.

Tscherniak and Gunsberg patented, in 1878 (*D.R. Pat.* 3199, April 9, 1878; *Ber.* **12**, 140; *Dingl. Polyt. J.* **232**, 80; *Jsb. Chem. Tech.* 1879, 471; 1882, 570), a method for making cyanides, similar to that of Schwarz.

Graham Young (1880) suggested the use of electricity for combining nitrogen and hydrogen.

*Decomposition of Alkaline Hydrates by Metals in Presence of Air to produce Ammonia.*—Dufréné, in 1880 (*Eng. Pat.* 5478, December 29, 1880), patented a process for making ammonia by combustion of zinc in air in presence of an alkaline hydrate; hydrogen and nitrogen are both set free and combine as ammonia in presence of hot iron, or platinum sponge. Twineh (*Eng. Pat.* 3712, August 25, 1881) removes oxygen from the air by nitric oxide, and brings the nitrogen in contact with nascent hydrogen from the decomposition of steam, or of alkaline hydrate, by metals, to produce ammonia.

**PART II.—An Inquiry in Regard to the Conditions under which Cyanogen is produced by Direct Union of Nitrogen and Carbon.**—The condition of free nitrogen is that of two atoms joined to one another to form a molecule and requiring the expenditure of a certain quantity of heat to tear them apart and leave each atom free to enter into new combination.

The most prominent source, and perhaps the only true source, of nitrogenous compounds in nature is found in the union of nitrogen and oxygen of the air under the influence of electric discharge.

Nitrogenous compounds have heretofore only been obtainable from substances already containing nitrogen, and the discovery that the conditions of the blast furnace are at times those necessary for the fixation of atmospheric nitrogen by combining it with carbon, gave rise to the hope that the problem as to how atmospheric nitrogen could be directly pressed into service as a source of nitrogenous compounds was at last solved. Such has not however proved to be the case, notwithstanding the many experiments that have been tried, which have been summarised in Part I. of this treatise. We have now to inquire what are the essential and what the non-essential conditions for the fixation of free nitrogen as cyanogen? The study of the blast furnace shows that a high temperature, a reducing atmosphere, an alkaline flux, and an excess of nitrogen are conditions favourable to the process of fixation, but as yet the conditions most suitable for the process remain undefined.

The author proceeds to discuss, by the light of the experiments summarised in Part I., the influence which temperature, the presence of oxygen and of water, the time of contact between the nitrogen and carbon, the degree of subdivision of the carbon, and the alkalis or other bases, has upon the formation of cyanogen from carbon and nitrogen; also the nature of the base to be employed in the process; the relation which ammonia bears to cyanogen and the conditions which determine the formation of the one or the other; the influence of other gases upon the absorption of nitrogen by carbon; the influence of hydrocarbons in the preparation of cyanogen or ammonia; finally, the influence of pressure upon the process. The results of the discussion are fully summarised as follows:—

**SUMMARY** of the present aspect of the question:—

The manufacture of cyanides and ammonia from the nitrogen of the air, while quite possible on the small scale, is beset with difficulties in its economical and commercial application, which, up to this time, have been insurmountable. These difficulties are probably matters relating mainly to construction and the endurance of materials.

At the same time there is still much uncertainty in regard to the chemical and physical conditions most favourable to the fixation of nitrogen.

The following conclusions are the results of the forgoing inquiry:—

1. The temperature necessary for formation of cyanogen may be lower than at first supposed, but certainly not below a high red heat. For ammonia a low red heat is sufficient.
2. The presence of free oxygen is deleterious in all cases.
3. Water in very small quantities is not prejudicial to production of cyanogen, and an excess is probably necessary in the synthesis of ammonia.
4. The long-continued contact of carbon and nitrogen is of less importance than thorough commingling of all reacting materials.
5. The presence of a strong base is essential, but under certain conditions ammonia, produced synthetically, may



serve the purpose of a base and effect the synthesis of ammonium cyanide.

6. Alkaline bases are preferable, potash especially, and probably because of the intermediate action of metallic potassium, which is reduced at a lower temperature than sodium. Baryta is the best of the alkaline earths for the purpose.

7. Ammonia and cyanogen are probably formed under conditions differing in respect to temperature and moisture, but otherwise similar, as already indicated.

8. The presence of carbonic oxide and of reducing gases in general, is probably favourable, while oxygen, carbonic acid and steam in excess (the latter only in the case of cyanogen) are probably injurious. Nothing is known as to the influence of sulphurous acid or hydrogen sulphide, but they are probably undesirable.

9. The presence of hydrocarbon vapour is favourable, and may, under certain conditions, permit the formation of both cyanogen and ammonia without the presence of a base.

10. There are no direct data for estimating the influence of pressure upon the reaction in question. The application of pressure is at least worthy of trial.

In the discussion which followed this paper, Dr. Waller referred to the formation of calcium chromate by heating chrome iron ore with lime, as an example of the formation of a salt aided by the presence of a base. Dr. Alberg remarked that the addition of lime would in some cases contribute only physical conditions which would aid in the reaction, such as porosity and infusibility; he added, as his opinion, that even the momentary formation of some compounds does not take place unless a base is present to fix them as soon as formed. [Professor Breneman in his paper having expressed his opinion that the base has no predisposing effect in inducing the formation of cyanogen, only serving to remove it from the sphere of action as soon as formed.] Dr. Doremus referred to the action of  $MnO_2$  on  $KClO_3$ . Mr. Sabin said the escape of nitrogen as ammonia in the old prussiate process was often very great; he doubted if any cyanide manufacturers in this country, with the exception of a firm at Newark, who are using an improved pear-shaped retort heated only at the central wide portion, are making it pay. Dr. Alberg referred to the increased manufacture of cyanides and ferrocyanides in Germany from gas purifier waste; at present the only practical process for making cyanides from sulphocyanides is by heating the latter with finely divided iron under pressure at  $120^{\circ}$ – $140^{\circ}$  C., whereby ferrocyanide and ferrous sulphide are formed. Dr. Doremus said that the Wilkinson process for manufacturing wood gas produced little cyanogen and no ammonia. Professor Breneman thought that the amount of ammonia and cyanide produced in gas retorts is possibly larger than that due to the nitrogen of the coal alone, though the access of atmospheric nitrogen cannot be very free.—A. G. B.

(See also pages 30–34.)

#### On Ammonia-Soda Processes. II. Schreib. Zeit. f. angew. Chem. 1889, 445–448 and 486–489.

IN a previous communication (this Journal, 1888, 434) the author indicated the line of work which he has followed up in the present article, and upon which he has founded a patented process for treating the mother-liquor of the sodium bicarbonate so as to fit it for use again in the carbonating towers. The process is based upon three reactions: (1) sodium chloride will precipitate ammonium chloride from concentrated solutions until the two salts are contained in the solution in equivalent proportions; (2) the solution so constituted will take up a large quantity of ammonium carbonate, whilst a further quantity of ammonium chloride becomes precipitated; (3) a low temperature will cause a further quantity of ammonium chloride to separate out and the resulting solution will dissolve sodium chloride. The three reactions are used simultaneously in actual work, but in order to ascertain their respective influences the author has examined them singly. By treating a saturated solution of ammonium chloride with solid sodium chloride and continual agitation, ammonium chloride separates out in flakes. Although the resulting solution contained the two

salts in approximately equivalent proportions, the relation was never quite constant. The results of several experiments averaged:—

Specific gravity.....	1.1758
Grms. per 100 cc.	
Total salts.....	37.17
NaCl.....	20.05
$NH_4Cl$ .....	17.12
Temperature of experiment.....	$20^{\circ}$

A similar but more imperfect result is given by the inverse reaction, in which a saturated solution of sodium chloride is agitated with solid ammonium chloride. The results of several experiments averaged:—

Specific gravity.....	1.18195
Grms. per 100 cc.	
Total salts.....	35.63
NaCl.....	23.71
$NH_4Cl$ .....	11.92

By evaporating a solution containing the two salts in exactly equivalent proportions, the salts crystallised out in the same proportions and the boiling mother-liquor contained the salts in the same relative quantities, but on cooling this mother-liquor to  $2^{\circ}$  and  $-5^{\circ}$ , only ammonium chloride crystallised out. On passing ammonia gas into a solution of the two salts, both chlorides separate out, but on passing a current of carbonic just sufficient to form normal ammonium carbonate, only ammonium chloride separates out.

The effect of these successive reactions on a pure ammonium chloride solution is shown in a tabular form:—

	$NH_4Cl$	NaCl	$NH_3$	$(NH_4)_2CO_3$
	Per Cent.	Per Cent.	Per Cent.	Per Cent.
I. Solution of $NH_4Cl$ .....	28.72	..	..	..
II. On treatment with NaCl at $20^{\circ}$ .....	17.12	20.05	..	..
III. On treatment with $NH_3$ gas .....	15.93	16.75	6.71	..
IV. On treatment with $CO_2$ gas .....	13.04	17.78	..	18.80
V. On cooling to $-5^{\circ}$ and shaking with NaCl .....	8.05	23.40	..	18.80
VI. After treatment of solution II. with $NH_3$ , $CO_2$ , and NaCl at $45^{\circ}$ , and subsequent cooling to $2^{\circ}$ and shaking with NaCl .....	4.10	25.50	..	19.50

On treating solution VI. with  $CO_2$  gas to saturation, sodium bicarbonate was precipitated, leaving a solution containing—

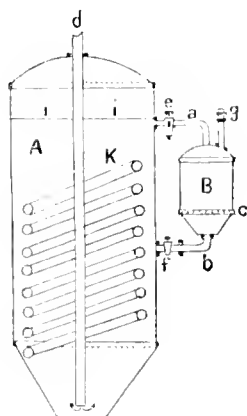
	Per Cent.
$NH_4Cl$ .....	21.0
NaCl.....	9.0

It is not advantageous to use commercial ammonium carbonate for these experiments, owing to the presence of the acid salt, which would precipitate sodium bicarbonate along with the desired ammonium chloride.  $NH_3$  and  $CO_2$  are best obtained in the right proportions by distilling a mixture of calcium carbonate with sal-ammoniac.

In order to adapt the process to the ammonia-soda manufacture, the apparatus shown in Fig. 1 is employed. The liquor leaving the sodium bicarbonate filters which contains the mixed chlorides of sodium and ammonium is pumped into the vessel A, which is connected with B by pipes *a* and *b*. B is filled with lumps of sodium chloride which rest on the perforated plate *c*. When the two vessels are filled with liquor to the level *i*, the salt in B becomes dissolved, and the liquor, by reason of its increased density, flows downward through *b* into A, whilst fresh liquor flows in through *a*. At the same time ammonia and carbonic acid are passed in through *d*, care being taken to have always an excess of ammonia present. The rise of temperature caused by the reaction should be allowed to reach  $50^{\circ}$ , in order to dissolve the maximum quantity of salt. *g* is a pipe for

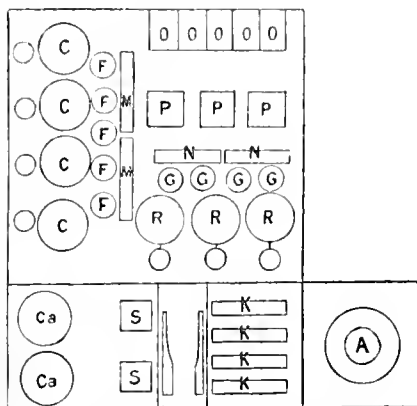
applying air pressure to B to drive out its contained liquor when it requires refilling with salt. When the liquor in A has been sufficiently charged with ammonium carbonate, it is cooled by the passage of cold water through the coil K. The temperature should be lowered to  $5^{\circ}$  if convenient, and

Fig. 1.



in any case to  $10^{\circ}$ . When sufficiently cold water is not available, artificial cooling must be resorted to. When the liquor is cool enough, it is allowed to settle and the clear liquor is drawn off, the rest being separated from the precipitate by filtration and compression. Sal-ammoniac as thus obtained consisted of 98 per cent.  $\text{NH}_4\text{Cl}$  and 2 per cent.  $\text{NaCl}$ . The clear liquor is sent into the carbonating towers. Fig. 2 shows the arrangement of plant in an

Fig. 2.



Ca = Calcining furnaces.  
S = Soda-ash mills.  
K = Boiler house.  
A = Lime kiln.

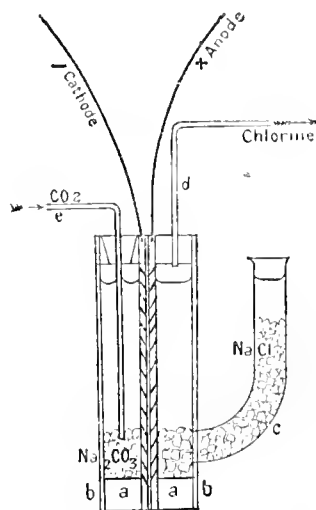
ammonia-soda factory in which the recovered ammonium chloride is decomposed in the dry way by heating it with powdered chalk in retorts. C are carbonating towers in which the ammoniacal salt solution is saturated with carbonic acid; these towers have vessels for holding salt like the apparatus shown in Fig. 1. The contents of the towers are brought upon the filters F, which retain the sodium bicarbonate and allow the liquor to flow into the montejes M, from which it is pumped into the regenerators R, described under Fig. 1. Here the liquor receives ammonium carbonate evolved from the retorts O, and is subjected to the special treatment under notice. The finished charge is sent into the filters G, from which the liquor goes to the montejes N,

to be pumped into the carbonating towers again. The sal-ammoniac left on the filters is sent into the presses P for the more complete removal of adhering mother-liquor. The recovered sal-ammoniac may be worked up in the wet way by distillation with milk of lime, with the advantage of making the liquors of any desired strength, and of obtaining finally a solution of calcium chloride nearly free from sodium salt. It might also be possible to decompose the strong solution of ammonium chloride by some form of calcium carbonate (compare this Journal, ). But the simplest method of treating the recovered ammonium chloride is to mix it with powdered chalk and to heat the mixture in retorts, by which ammonia and carbonic acid are obtained together, and a residue of solid calcium chloride is left in a condition which may be especially suitable to a process for obtaining chlorine. The author refers to the loss of sodium bicarbonate arising from the use of water for washing out the mother-liquor from the mass on the filters, and thinks that mineral oil might be used for expelling the mother-liquor and avoiding loss of product.—G. H. B.

*The Electrolysis of Sodium Chloride Solution with Direct Production of Chlorine and Crystallised Soda.*  
W. Hempel. Ber. 22, 2475—2478.

THE author is studying with J. Fogh the electrolysis of chlorides under such conditions that the ions are prevented from entering into secondary reaction. The simplest method of securing this complete separation of the ions is by conversion into relatively insoluble compounds. In the case of sodium chloride, advantage is taken of the insolubility of sodium carbonate and bicarbonate in the saturated solution of sodium chloride, i.e., the sodium hydrate is converted into these compounds by the action of carbonic acid at the cathode. The apparatus employed by the author is shown in section in the annexed drawings. The electrodes are circular (Fig. 1). For the cathode iron plate is used; for

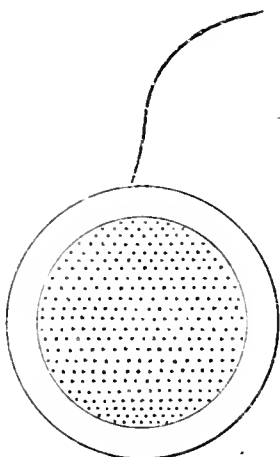
Fig. 1.



the anode, retort carbon. They are perforated at numerous points, as shown (Fig. 2), the perforations being so made as to permit the escape of gas bubbles upwards. Between the electrodes a fine sheet of asbestos paper is secured, the distance between the electrodes being about 1 mm. At a, a, chambers are constructed on either side of the electrodes by means of porcelain rings, that in the cathode chamber being perforated to allow the entrance of the tube conveying the carbonic acid, also the withdrawal of the sodium carbonate produced and there collected. The only detail of construction especially noteworthy is the insertion of the asbestos between the electrodes. This the

author contrasts with and finds superior to the arrangement patented by J. Marx (D.R. Pat. 46,318), who claims to secure the isolation of the products of electrolysis by means

Fig. 2.



of diaphragms of gauze. He also notes that his investigation was commenced at a period prior to the publication of the specification of the patent in question, which claims the conduct of the electrolysis under conditions otherwise similar in every respect. The results obtained by the author are as follows:—

The E.M.F. required is made up of 3.2 volts for the decomposition of the chloride, plus 2.5 volts for the back E.M.F. With a current of 1.73 amperes at this voltage the yield of chlorine amounted to 0.930 grm. per hour. Taking the equivalent of 1 h.p. at 680 volt-amperes, the total production would be 64.5 grms. of chlorine and 259.8 grms. of  $\text{Na}_2\text{CO}_3 \cdot 10 \text{H}_2\text{O}$  per horse-power per hour. (See also Eng. Pats. 13,306, 14,199, and 14,239 of 1888; this Journal, 1889, 778.)—C. F. C.

*The Distribution of the Liquid in Gay-Lussac-, Glover-, and Hydrochloric Acid Towers, &c.* G. Lunge. Zeits. f. angew. Chem. 1889, 603—605.

THE author remarks that he has found the old method of distributing the liquid in coke towers by means of "reaction distributors" entirely replaced by an overflow arrangement in many of the works in London, Lancashire, on the Tyne, and in the North of France. The following figures show the method adopted, Fig. 1 being a plan, Fig. 2 a through section, and Fig. 3 a general view of the apparatus, which

Fig. 1.

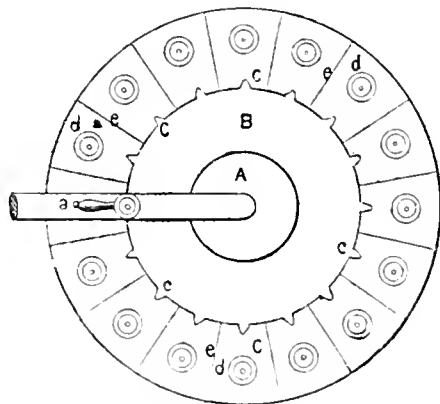


Fig. 2.

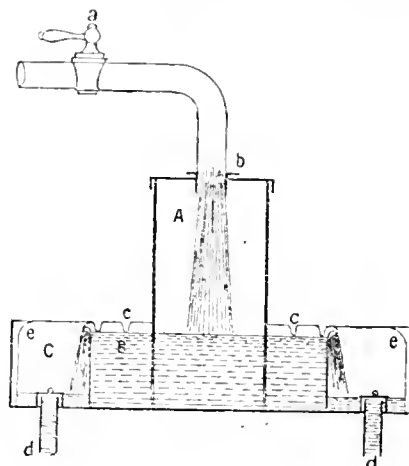
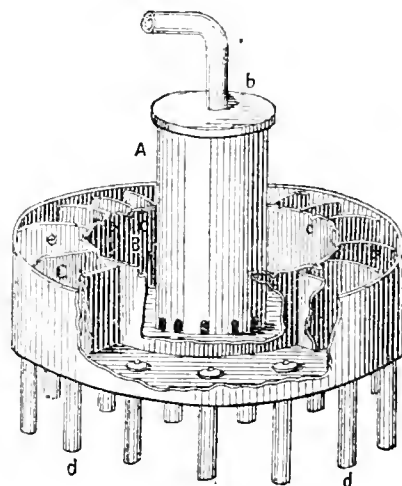
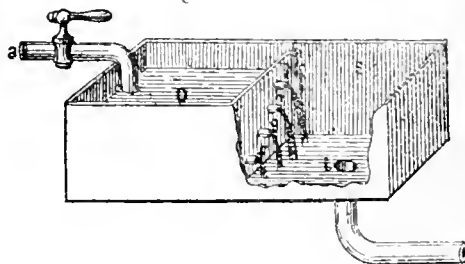


Fig. 3.



is generally made of lead. The liquid to be distributed enters through the cock *a*; where a mixture of two liquids is fed into the tower (e.g., in the Glover tower) two cocks are attached to the pipe. The pipe conveying the liquid is fixed into the circular trough *A*, provided with a cover *b*. Holes are cut in the base of *A*, through which the liquid has free access to the shallower trough *B*. *C* is a wider trough of the same depth as *B*, and is divided up into a number of compartments *e* (16 in this case), each of which is separately connected with *B* by the small spouts *c* shown in the figure. Each partition *e* has a pipe *d*, sealed by means of a hydraulic float, attached to it, and through these

Fig. 4.



the liquid passes down the tower. The spouts in the divisions *e* are best so arranged that, should a stoppage occur in any one of them, the excess of liquid runs over into the adjacent compartments, and not outside the trough C. In order to regulate and to control the supply of liquid, a leaden vessel, shown in Fig. 4, is used in many works, and is placed between the supply pipe and the trough A. The two compartments D and E communicate by means of the tubes *f, g, h, i*, and the spout *k*, so that the workman has only to be told how many of the pipes he is to have working to ensure the passing of the right quantity of liquid.—C. A. K.

*Adulteration of Chili Saltpetre with Stassfurt Potassium Salts.* O. Böttcher. *Sächs. landw. Zeitschrift*. 1889, **37**, 192–193.

Two samples of Chili saltpetre showed by their high percentage of potash, chlorine, sulphuric acid, and magnesia, coupled with a low percentage, 8.57–8.71, of nitrogen, that they consisted of about half of potash salts, and could not be regarded as saltpetre. Unadulterated Chili saltpetre is composed about as follows:—nitrogen, 15.5 per cent.; chlorine, 1.7; sulphuric acid, 0.7; soda, 35.0; water, 2.6; sand, 1.5, &c.—D. A. L.

*The Life of Sulphuric Acid Chambers.* A. Burgemeister. *Chem. Zeit.* **13**, 1633–1634.

In order to gauge the rate at which the walls of sulphuric acid chambers are corroded by the action of the chamber gases and acid, a number of measurements were made of the thickness of the walls of two chambers, built respectively in 1865 and 1876.

The chamber built in 1865 is a No. 2 chamber, and during the 285 months between December 1865 and end of September 1889, was working for 210 months, and idle for the remaining 75 months. Its capacity is 1,559 cubic metres, and it was originally constructed of lead 2.57 mm. thick. The average of many measurements showed that the thickness in October 1889 was 1.88 mm., a decrease of 0.69 mm. This is equal to a loss of 7.86 kilos. per square metre, or 26.8 per cent.

The other chamber belongs to the same set as the last-mentioned one, and has been used as No. 1 chamber in that set. It has a capacity of 257 cubic metres. It was rebuilt in 1876, of lead 3.40 mm. thick. Owing to its position in the set it has been worked at a higher temperature than the No. 2 chamber (at from 65° to 90° C.). It has been at work 120½ months in all. Measurements taken in October last showed the average thickness of its walls to be 2.80 mm., a reduction of 0.60 mm., equal to a loss of 6.84 kilos. per square metre, or 17.65 per cent.

The action on the walls of these chambers was of course not uniform. Those parts of the walls in contact with the acid lying in the chambers were found to be most acted upon, and in some parts of this region the lead was as thin as paper, whilst in places, during many years past, holes have been eaten through. Next, those parts were most acted upon where the lead overlapped and where the wooden framework prevented its cooling. When the seams are burnt from the inside, an overlapping strip is left outside, and in such places the inner thickness of lead is soon eaten through, and the seams leak. When, on the contrary, the seams are burnt from the outside, they do not leak until the outer sheet of lead is also eaten through. The floor is the part of the chamber least acted upon, as the acid covering it at the relatively low temperature of 40°–60° C. does not corrode the lead so vigorously as do the warmer gaseous contents of the chamber.

Lunge (*Sulphuric Acid and Alkali*, Vol. I. 281) says: "Under normal circumstances a chamber will generally last from eight to 10 years, but with many repairs during the latter years." The above instance shows that a much greater age may be reached.—H. S. P.

## PATENTS.

*Improvements in the Manufacture of Cyanogen Compounds from Sulpho-cyanides.* T. Parker and A. E. Robinson, Wolverhampton. Eng. Pat. 17,447, November 30, 1888. 4d.

A SOLUTION containing about 20 per cent. of the sulpho-cyanide and acidified with sulphuric acid is subjected to the action of an electric current at a density of from 10 to 30 ampères per square foot of anode. The resulting gaseous hydrocyanic acid may be led off and condensed in water, leaving a sulphate in the original solution.—B. T.

*Improvements in Salt-cake Pots.* D. Y. Cliff, C. Cliff, P. Cliff, and B. Cliff, Leeds. Eng. Pat. 17,459, November 30, 1888. 4d.

THE improvements consist in making "salt-cake pots" from fire-clay and other clay or cement.—F. S.

*Improvements in Flat or Prismatic Evaporating Pans for the Treatment of Salt.* M. and H. Fläsehdreger, Arnshall, Germany. Eng. Pat. 17,541, December 1, 1888. 6d.

THE evaporating pan is provided with a heating boiler which extends nearly the whole length of the pan, and is provided with vertical sides with water or flue tubes. The boiler is furnished with a lid, having "bent-over bevelled edges" so as to prevent the grains of salt solution in the pan and those settling at the bottom from entering the boiler. By this arrangement a better evaporation, as well as a rational consumption of the fuel, is claimed.—F. S.

*Improvements in Evaporating Brine and other Solutions.* Sir Lowthian Bell, Northallerton. Eng. Pat. 17,564, December 1, 1888. 6d.

THE invention consists in utilising the waste heat of slag derived from iron furnaces and furnaces employed in other metallurgical operations. The slag may be received into iron vessels mounted on wheels and capable of containing several tons. These vessels when charged are caused to be passed down an incline into a chamber, over which an evaporating pan of large area is set. When the slag blocks are in the chamber they are sprinkled with water to harden the exterior. Steam is thereby generated, and this steam coming in contact with the bottom of the pan imparts its heat thereto, and so heats the brine or solution which the pan contains. Or the steam thus generated may be passed through pipes immersed in the solution. In the lower part of the chamber also there is water which the blocks of slag, as they progress slowly down the incline, and when they are sufficiently hardened, ultimately reach, and so the water in the chamber is kept at a boiling heat. When the blocks are cooled down to the temperature of the water the carriages on which the vessels containing them are mounted, are passed on up another incline, and so out of the chamber.

In place of using water a current of air may be caused to pass through the chamber in the reverse direction to that in which the blocks travel through it, and the air thus heated may be made to pass beneath the pans containing the brine or solution to be evaporated, the air being led back again to the exit end.—F. S.

*Improvements in or relating to the Treatment of Salt, and in the Means employed therefor.* J. W. Hewson, Stockton-on-Tees. Eng. Pat. 17,807, December 6, 1888. 8d.

AN improvement in the manufacture of blocks of salt, enabling this to be done without regard to when and where the process of evaporating the brine has been carried out, or—what amounts to the same thing—to the amount of heat and moisture contained in the salt at the commencement of this process (except that the proportion of water should not be below that which salt in the free state naturally retains).

The salt is pressed into asbestos-lined iron or tin moulds, and heated in a suitable oven to about 170° F., for about

half an hour. It is then removed from the moulds and heated to about 220° F. for from one to two days.

By this means salt may be shipped in bulk and subsequently converted into blocks, from the state in which it is delivered.

A further important result claimed is the facility afforded for reducing coarse grained to fine table salt, by substituting a cheaper and more convenient grinding process for the boiling process now in use.—F. S.

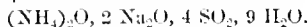
*A New and Improved Method of Manufacturing Glazing, Antiseptic, and Washing Compounds.* J. Ascough, Handsworth. Eng. Pat. 1430, January 26, 1889. 6d.

THIS improved method consists in manufacturing these compounds by friction, heat, fusing, or fluxing the equivalent or approximate component parts of such compounds together until they become intimately mixed and combined.

It "is primarily associated with boron and sodium compounds, but can be applied to the manufacture of many other chemicals." Thus to make borax, boric acid and sodium carbonate are "frictionized" by grinding or other means, or fused together. The product may be used for purposes for which borax is now used.—F. S.

ERRATUM.

In the November issue for 1889 of this Journal, page 882, col. 2, the formula of the Acid Bisulphite of sodium and potassium should be  $2 \text{Na}_2\text{O}$ ,  $\text{K}_2\text{O}$ ,  $4 \text{SO}_2$ ,  $9 \text{H}_2\text{O}$ , and that of the Acid Bisulphite of sodium and ammonium—



## VIII.—GLASS, POTTERY, AND EARTHENWARE.

*Leadless Glazes.* Seger. Thonind. Zeit. 1889, 13, 524.

THE author has already proposed and practised the substitution of barium glazes containing boric acid for the lead glazes common on stoneware, and has now turned his attention to the production of glazes free from lead capable of receiving the rich and varied colours needed for artistic purposes. He has confined his work to glazes melting at temperatures below the fusing point of gold, that being the limit usually adopted, and starting with a normal calcium glass of the formula  $0.5 \text{K}_2\text{O}$ ,  $0.5 \text{CaO}$ ,  $2.5-3.0 \text{SiO}_2$ , has investigated the effects produced by the addition of alumina, silica and boric acid in various proportions, with the following results: Glazes of the composition  $0.6 \text{K}_2\text{O}$ ,  $0.4 \text{CaO}$ ,  $2.5 \text{SiO}_2$ ,  $0.5 \text{B}_2\text{O}_3$ , with and without the partial or complete substitution of  $\text{Na}_2\text{O}$  for  $\text{K}_2\text{O}$ , fuse below the melting point of silver, and are clear at a little above that of gold; those of the composition  $0.6 \text{K}_2\text{O}$ ,  $0.4 \text{CaO}$ ,  $0.1-0.3 \text{Al}_2\text{O}_3$ ,  $2.5 \text{SiO}_2$ ,  $0.5 \text{B}_2\text{O}_3$ , tend to become opaque as the proportion of alumina increases unless rapidly cooled; on the other hand those poor in alumina have the greater tendency to "crackle." On raising the proportion of silica to  $3.5 \text{SiO}_2$  in the case of the glaze containing  $0.2 \text{Al}_2\text{O}_3$ , the "crackling" was inevitable, and simultaneous increase in the quantity of the boric acid gave a glaze which became opaque on slow cooling; notwithstanding this a glaze of the formula  $0.6 \text{K}_2\text{O}$ ,  $0.4 \text{CaO}$ ,  $0.8 \text{Al}_2\text{O}_3$ ,  $5 \text{SiO}_2$ ,  $1 \text{B}_2\text{O}_3$  proved of general utility.

Glazes containing  $0.5 \text{K}_2\text{O}$  and  $0.5 \text{CaO}$  behaved similarly, and proved useless when the contents of alumina was low. On increasing the quantity of alumina transparent glazes were obtained, the reverse being true of boric acid; thus the glaze  $0.3 \text{K}_2\text{O}$ ,  $0.7 \text{CaO}$ ,  $0.5 \text{Al}_2\text{O}_3$ ,  $4 \text{SiO}_2$ ,  $0.5 \text{B}_2\text{O}_3$  was clear, while  $0.3 \text{K}_2\text{O}$ ,  $0.7 \text{CaO}$ ,  $0.5 \text{Al}_2\text{O}_3$ ,  $4 \text{SiO}_2$ ,  $1 \text{B}_2\text{O}_3$  was opalescent, and  $0.3 \text{K}_2\text{O}$ ,  $0.7 \text{CaO}$ ,  $0.5 \text{Al}_2\text{O}_3$ ,  $4 \text{SiO}_2$ ,  $2 \text{B}_2\text{O}_3$  was like milk-glass; increase in the proportion of alumina, though giving clear glazes, raised their melting points. With regard to the bases in the glazes, the limit  $0.2 \text{K}_2\text{O}$ ,  $0.8 \text{CaO}$  must not be passed, as more lime causes opalescence in all cases.—B. B.

*On Copper-Red.* Lauth and Dutailly. Mon. Céram. et Verr. 19, 237.

NUMEROUS experimenters have endeavoured to arrive at the true Chinese copper glaze, a sample of which, analysed by Ebelman and Salvétat in 1832, had the composition:  $\text{SiO}_2$  73.9,  $\text{Al}_2\text{O}_3$  6.0,  $\text{Fe}_2\text{O}_3$  2.1,  $\text{CaO}$  7.3,  $\text{CuO}$  4.6,  $\text{K}_2\text{O}$  3.0,  $\text{Na}_2\text{O}$  3.1, parts per cent. The authors find that the most successful imitations have a composition of which the main constituents vary within the following limits:—

	Per Cent.		Per Cent.
Alumina.....	10.56	to	5.4
Silica.....	66.00	to	46.5
Alkalis.....	6.29	to	28.0

They lay down the following rules:

(1) The glaze must not be too aluminous. (2) Alkalis rather than lime should be used as basic material. (3) Borax prevents the "crackling" which is apt to be produced by the alumina and alkalis present, and favours the formation of the red colour. (4) Stannic oxide in quantity smaller than that of the copper oxide employed is useful, aiding the reduction of the latter. (5) The use of lead gives bad results. Two good recipes are:—

	I.	II.
Pegmatite.....	40	10
Sand.....	40	44
Chalk.....	18	12
Borax (dried).....	12	..
Soda.....	..	21.5
Cupric oxide.....	6	6
Stannic oxide.....	6	3

	Percentage after deducting of CuO and SnO <sub>2</sub> .	
	I.	II.
Silica.....	67.62	68.92
Alumina.....	7.06	6.85
Alkalis.....	7.83	17.83
Lime.....	9.87	6.39
Boric anhydride.....	8.23	..

The formation of the red colour takes place best in open vessels; during the firing strong reduction occurs, followed by oxidation and the development of blue or green tones if air be allowed access at the end of the operation. It is thought that the red colour is due, not to a cuprous silicate, but to the diffusion and solution in the glaze, of metallic copper, similar to that obtaining in the case of gold. If the ware be cooled rapidly, only a colourless glaze is produced, slow cooling being necessary to bring out the full colour.

—B. B.

*Manufacture of "Porcelaine Craquelée."* Lauth and Dutailly. Mon. Céram. et Verr. 19, 284.

THIS method of decoration is very much used by the Chinese. It may be brought about in two ways: (a) by conducting the burning at such a temperature that the expansion of paste and glaze is different; (b) by altering the composition of either. The simplest way is to modify the composition of the glaze. By increasing the quantity of alkalis the glaze becomes more easily fusible; by increasing the quantity of silica, less easily fusible; it is found practically better to increase the quantity of silica and so make the glaze more difficultly fusible. If more silica be added, the alumina must be decreased in proportion; the same result may be attained, however, by increasing the

alumina alone, although in this latter case the markings are not so regular. The authors use the following glazes:—

	"Craquelée" Glaze (Increase in Silica).	Normal Glaze.
SiO <sub>2</sub> .....	79.42	66.18
Al <sub>2</sub> O <sub>3</sub> .....	11.80	14.55
KNaO .....	5.51	3.55
CaO .....	2.88	15.90

They use a paste consisting of 66 per cent. of SiO<sub>2</sub>, 27 per cent. of Al<sub>2</sub>O<sub>3</sub>, and 7 per cent. of KNaO. The "craquelée" glaze is made from 51.5 parts of pegmatite, 38 parts of sand, 6 parts of kaolin, and 5 parts of marble. By increasing the alumina alone the following glaze may be used:—

SiO <sub>2</sub> ..... 69.92	} corresponding to	85.70 parts of felspar.
Al <sub>2</sub> O <sub>3</sub> .... 18.14		14.18 " " sand.
KNaO.... 11.95		1.42 " " kaolin.

—T. L. B.

#### PATENT.

*Improvements in and Relating to the Construction of Glass Melting Pots.* A. G. Neville, Lazeurville, U.S.A. Eng. Pat. 13,128, August 20, 1889. 6d.

The glass melting pots described in this specification are provided with a "glory-hole," an opening for the entrance of the flame, a series of air-supplying passages, a series of escape flues communicating with the pot at different levels, and a hot blast flue having communication with the pot at several points.—E. G. C.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

*Hydraulic Cements.* Dingl. Polyt. J. 273, 471—477, 551—563, and 587—600.

NEARLY the whole of the first part of this article is occupied by an account of *Candlot's* work, for an abstract of which see this Journal, 1889, 543 and 544. The rest deals with sundry alterations in the methods of testing prescribed in Germany which are of purely local interest, save that which does away with the "firing test" (*Darrprobe*, this Journal, 1889, 781) as fallacious, seeing that it may both condemn a good cement and allow a bad one to pass.

The committee of the Association of German Cement Makers points out that the rules for testing Portland cement are only applicable to that material, and will not serve to compare other kinds of hydraulic cement with each other.

R. Dyckerhoff, at the general meeting of the Association of German Cement Makers, discussed the various methods of preparing test-pieces. When these were universally made by hand both tension and compression specimens had the same density; but when they began to be prepared by the Böhme hammer apparatus, 150 blows with a 2-kilo. hammer being used in each case, it was plain that as the compression specimens attained the same density as those prepared by hand, the tension test-pieces must have a greater density, as their bulk was only one-fifth as great. If fewer blows were used (*e.g.* 30) so as to get the right density, the tensile strength fell considerably short of the hand-gauged test-pieces. These facts are well shown by the following table:—

1 Cement : 3 Normal Sand.

Mark.	Method of Preparation.	Water used.	Specific Gravity.		Strength, 28 days.	
			Tension.	Compression.	Tension.	Compression.
		Per Cent.				
A.	Hammer apparatus, 150 blows .....	10	2.304	2.226	22.1	224.0
A.	By hand.....	10	2.228	..	22.4	..
A.	Hammer apparatus, 30 blows .....	10	2.232	..	18.6	..
B.	Hammer apparatus, 150 blows.....	10	2.304	2.236	20.5	193.6
B.	By hand.....	10	2.235	..	20.6	..
B.	Hammer apparatus, 30 blows .....	10	2.228	..	15.6	..

From this it appears that the density of the test-pieces is a gauge of their right preparation only when the specimens compared have been made by the same means, and in the same manner.

Böhme has experimented with his own apparatus on the influence of the number of blows used, on the density and tensile strength of briquettes:—

1 Cement : 3 Normal Sand.

No.	Mode of Preparation.	Average Weight.	Corresponding Density.	Mean Tensile Strength at Seven Days.
I.	By hand .....	160.6	.. .. .	Kilos, per Sq. Cm. 16.06
II.	Hammer apparatus, 75 blows.....	158.0	2.225	12.75
III.	" " 100 " .....	159.5	2.246	13.25
IV.	" " 125 " .....	159.5	2.246	14.56
V.	" " 150 " .....	159.0	2.239	15.56

The apparently greater tensile strength of the hand-gauged briquettes, when compared even with those prepared by 150 strokes in the hammer apparatus, may be due to some subsidiary factor, such as the trimming and smoothing off of the cement in the mould, and not to any innate superiority in the process.

Schumann has noticed that cubical test-pieces for compression tests exhibit a somewhat greater strength when allowed to harden in the mould than when taken out earlier, the difference amounting to 17–34 kilos. per sq. cm., but following no discernible law. Böhme observed a maximum difference of 11 kilos., while Schott attributes the phenomenon to the variation in the quantity of moisture present, and points out that with soft cement mortars true cubes are not obtained, but in their place slightly rhombohedral masses. The Association accordingly resolved that the test-pieces shall not be withdrawn from the moulds until 24 hours after gauging have elapsed.

Schiffner discoursed on the testing of quick-setting cements by the standard rules. Such cement gives a lower tensile strength than it actually possesses on account of its setting to some extent during gauging. He counselled the use of a temperature not greater than 15°–18° C. (compare Candlot's researches in this direction, this Journal 1889, 544), and proposed in the case of cements setting too quickly, even at this temperature, to abandon the use of the hammer apparatus in favour of hand-gauging and the use of quantities of material only sufficient for one or two briquettes at a time. The neglect of these precautions leads to the tensile strength of test-pieces prepared by the machine to be about two-thirds only of that of the specimens prepared by hand.

Some means of checking the predetermined composition of the mortar on the spot where it is to be used as a building material is very desirable, and P. Goloubiatnikow has devised the following plan:—100 grms. of the neat cement are passed through a sieve having 900 holes per sq. cm., and the quantity and therefore percentage of the portion that passes through, determined: this is called *a*.

The sand with which it is to be mixed is similarly treated and the result termed *b*. One hundred grammes of the mixed sand and cement awaiting use at the works are then dealt with in the same way, and the portion which passes through the sieve weighed and called *M*. Then calling the unknown quantities of cement and sand in the mixture *C* and *Z* respectively, we have—

$$C + Z = 100$$

and

$$\frac{a}{100} C + \frac{b}{100} Z = M.$$

Hence—

$$C = \frac{100(M-b)}{a-b} \text{ and } Z = \frac{100(M-a)}{b-a}.$$

By this simple method results of a considerable degree of exactitude can be obtained, the total maximum experimental error being 4.5 per cent.

If  $a = b$ , a sieve with a different mesh must be used. The process is said to be applicable to mortar that has been wetted for use; more water is added until the consistency is that of a thin paste, which is then evaporated to dryness with constant stirring over a naked flame, and dealt with as above; it is asserted that the size of the cement particles is unaltered by this treatment.

The sintering points and relative fusibility of cements have been studied by M. Meyer, who experimented with six samples of slurry and found them all run down to a slag below the temperature at which Seger's standard cone No. 15 melts.

By using definite quantities of fuel to produce given temperatures it was found that the consumption of 1,100 grms. of coke (in a Senfström furnace) yielded a temperature high enough to give a porous appearance to all the test-pieces, while when 1,150 grms. were burned, complete fusion took place. The six samples of slurry,

ranged from the most to the least fusible, had the following percentages of calcium carbonate:—

No. 1.....	76.6
No. 2.....	76.2
No. 3.....	75.2
No. 4.....	76.3
No. 5.....	77.4
No. 6.....	78.8

Nos. 1 and 2 contained much ferric oxide, No. 3 much magnesia.

In order to gain some knowledge of the influence of the percentage of lime on the heat of saturation of a cement by hydrochloric acid, a calorimetric investigation was instituted. An Erlenmeyer flask was well protected from the reception or emission of heat in the ordinary way, 20 grms. of normal sand previously washed with acid introduced, and 250 cc. of 10 per cent. hydrochloric acid added. The whole apparatus was brought to the temperature of the room, the thermometer it contained (graduated to 0.05° C.) read off, and the cement to be tested (1 gm.) reduced to the finest possible powder thrown in, the whole well mixed and the thermometer again read when it ceased to rise. The water-value of the apparatus as well as the correction for loss by radiation, were determined by performing an experiment with warm water only, and also with pure lime. The six cements ranged in the order of their fusibility gave the following figures:—

Order of Fusibility.	Rise of Temperature.	Lime.
	° C.	Per Cent.
1	1.76	60.3
2	1.77	61.1
3	1.78	61.1
4	1.82	63.8
5	1.80	65.3
6	1.80	67.7

It appears therefore that the rise increases with the percentage of lime. It has been further established that the harder a cement has been burnt the less heat it evolves in its reaction with acid: e.g., an irregularly burnt piece of clinker was divided into three parts according to the temperature it had been locally subjected to; the under-burnt portion gave a rise of temperature of 2° C. for 1 gm., the next, one of 1.94° C., and the thoroughly burnt, 1.82° C. As the final products of the reaction of the cement and acid are the same in all three cases, it is plain that an evolution of heat by the clinker occurs in the kiln when it is burnt at a higher temperature.

Blast furnace slag tested by the same method gave, before granulation, a rise of temperature of 0.8° C., and when granulated, 1.8° C. This, therefore, may be used as a method for detecting sophistication of Portland cement with slag.

#### *Action of Air, Fresh Water, Sea-Water, and Frost on Cement.*

(a) *Before use.*—Tomäi has come to the following conclusions:—

- (1.) Test-pieces of neat cement are best for the study of the action of the air.
- (2.) Dry carbonic anhydride is not sensibly detrimental, and may be advantageous in some cases.
- (3.) The prolonged action of moist air is certainly deleterious. During the experiments from which these deductions are drawn it was incidentally determined that the so-called "Nachfeinen" did not take place, as no difference in the fineness of the cement before and after exposure could be detected; it was also found that the rise of temperature decreased in proportion to the extent to which moist air had acted on the cement. The practical upshot of all this is that aeration is harmless or even beneficial if rationally conducted, but most injurious in the contrary case.



(b) *After use.*—It is of importance to ascertain the behaviour of cement under various conditions of hardening, considering its bearing upon actual work. R. Dyckerhoff has found that test-pieces put under water less than 24 hours after gauging show but little deviation in strength in compression from others treated in accordance with the standard rules. The time limit is fixed by the rapidity with which the cement sets; quick-setting cements can be immersed after the lapse of six hours or even less.

The following experiment serves to show the influence of water pressure on cement while it is hardening:—A tank, 2 m. + 50 cm. + 2 of brick, half a brick thick, was built, using a mortar consisting of one of Portland cement to three of sand. After a few days it was filled with water and found to leak at the lower part of the sides and bottom; the water was then drawn off and one side lined with a mixture of one of cement to one of sand, covered with neat cement, when it was found to be water-tight. An attempt was made to coat the three other sides while under water, but without

success, as the cement only stuck to the upper third of the tank. At a certain pressure the cement had no tendency to harden at all. It is therefore best to keep water off cement while setting, but if this be impracticable, recourse may be had to a quick-setting cement (1:1), or a mixture of the usual composition (1:3), to which has been added one-fourth part of slaked lime, thus decreasing the time of setting to a quarter of an hour. The results of a series of experiments made with a cement of ordinary strength, which set in five hours, and of which the test-pieces had been prepared in the standard way, demonstrated that the highest tensile strength at the end of a year is attained by exposure in the open air after the first 24 hours.

If either kept under water or in a dry atmosphere the figures are considerably lower. Thus Portland cement may be looked upon as pre-eminently an aerial mortar.

The appended table shows the effect of sea-water on cement both gauged with it and allowed to harden in it:—

Mark.	Time of Setting.	Tensile Strength. Kilos. per Sq. Cm.							
		Gauged with, and kept in, Fresh Water.		Gauged with Fresh Water, kept in Sea-water.		Gauged with Sea-water, kept in Fresh Water.		Gauged with, and kept in, Sea-Water.	
		1 Week.	4 Weeks.	1 Week.	4 Weeks.	1 Week.	4 Weeks.	1 Week.	4 Weeks.
A	8	20.9	26.7	18.6	25.1	17.1	24.8	16.9	23.2
B	6	..	25.6	17.5	22.4	..	22.8	15.5	20.6
C	6	18.1	23.6	16.7	21.2	14.9	20.5	15.3	19.3
D	6	15.6	21.1	13.8	19.3	11.4	18.0	11.0	17.7
E	$\frac{1}{2}$	15.4	20.4	14.9	18.6	12.9	17.1	12.0	17.0

The general effect of the sea-water is to lower the tensile strength; if used for gauging the cement it makes this effect most felt; the use of fresh water in practice should therefore be insisted on. A quick-setting cement suffers less, as the infiltration of the sea-water and the destructive double decomposition taking place between the lime in the cement and the magnesium salts in the sea-water, is sooner stopped.

The comparative harmlessness of sea-water acting on cement from without appears contradicted by the experiments of Professor Brazier, of Aberdeen, and by the failure of the breakwater at that place. The former were, however, conducted on a scale and under conditions which prevent their bearing directly on actual work, while the latter cannot reasonably be attributed to any specific and inevitable action of sea-water on Portland cement. The breakwater was constructed as follows:—Bags of concrete (1 part of cement, 2½ parts of sand, and 3½ parts of gravel), weighing about 100,000 kilos., were deposited at the bottom of the sea and levelled by divers. On these were built concrete blocks (1 part of cement, 4 parts of sand, and 5 parts of gravel), previously made and allowed to harden in the air, and heavy stones. The blocks weighed in the first instance 7,500—18,000 kilos., and were afterwards increased to 10,000—24,000 kilos.; they were built up with open joints to a height of 8 m., 30 cm. above low-water mark. Above these was built a layer of concrete run in between temporary retaining boards *in situ*, consisting of 1 part of cement, 3 parts of sand, and 4 parts of gravel. The mean thickness of the whole structure was 12 m.; all the materials were of good quality. After some years signs of destructive action were observed. In heavy weather single blocks of concrete were torn away, and through the gaps thus left the sea entered and did further mischief. A creamy paste appeared in various cracks and crannies, which was found by Brazier to consist of magnesia and calcium carbonate.

German manufacturers assign this failure to mechanical causes. They consider that the concrete blocks were too small, and that the cement was used too sparingly. Schumann advocates a mixture not poorer than 1 part of cement, 2 parts of sand, and 3 parts of gravel, where

work is exposed to the direct action of the sea. Portland cement has been freely used in Germany for harbour work, and failures are quite exceptional, and can then be traced to the use of bad material.

With regard to the action of frost on cement, Froideville and Schott remark on the fact that cement gauged with excess of water exfoliates when exposed to frost, the solidification of the water in excess of that necessary for the setting of the cement being the cause. Other observers, and notably Bohme (for an abstract of whose work see this Journal 1889, 393), agree that frost has little permanent ill effect on Portland cement used with reasonable care.

Dyckerhoff, Seger, and Delbrück are agreed that the addition of salt to Portland cement is baneful, but consider it requisite for Roman cement and lime mortar. Goerke states that the use of sand containing salt is prohibited at Norden on the Baltic.

Dyckerhoff has experimented on the behaviour of various hydraulic cements under different conditions of hardening, and records his results in the appended table:— (See top of page 72.) Normal sand was used throughout, and the samples were, without exception, gauged by means of the Bohme hammer apparatus, using 150 strokes.

Schumann found, in comparing Portland cement with puzzolana cement, that the latter suffered more from the action of frost than the former, and that it was altogether a weaker material. Like trass cement it consists of a mixture of acid silicates and lime, which combine to form cementitious basic silicates when brought into contact with water, thus offering a contrast to Portland cement, which has been brought to such a composition in the kiln that it combines directly with the water added to it. This accounts for the fact that puzzolana and trass cements harden better under water than they do in air.

According to L. Tetmaier, cements free from any tendency to alter in volume after setting do not exist; they all expand in water and contract in air. It is essential to note the above facts in testing this property, and to eschew such crude tests as that consisting in filling a thick-walled

Nature and Composition of Sample	Standard Tests.			Tensile Strength. Kilos. per Sq. Cm.											
	Time of Setting (hours).	Strength; 28 Days. Kilos. per Sq. Cm.		Kept under Water. Tested after Weeks				Kept 24 Hours in the Open Air. Tested after Weeks				Kept one Week in Water, then in the Open Air. Tested after Weeks			
		Tension.	Compression.	1.	4.	13.	26.	1.	4.	13.	26.	1.	4.	13.	26.
Portland cement, A. 1:3 sand.	8.	21.4	208.8	17.4	21.4	28.2	28.4	16.8	29.1	28.3	33.5	18.1	24.0	33.3	32.7
Puzzolana I. 1:3 " "	12	20.8	105.6	12.5	20.8	21.8	23.9	4.9	11.0	12.2	16.3	12.2	14.1	20.2	19.3
Portland " A. } 1:6 " "	..	..	..	8.6	13.0	16.9	19.8	8.9	15.5	18.4	22.3	9.0	14.8	23.3	26.4
+ 1/2 slaked lime }	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..
Trass 1, slaked lime 1, } ...by vol.	..	..	..	1.8	10.7	14.8	19.3	1.8	6.5	12.1	14.8	2.0	7.1	12.4	13.9
sand 1 .....	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..
Portland cement, B. 1:3 sand.	3	21.0	211.2	17.5	21.0	22.7	28.2	16.1	27.6	25.5	35.4	17.6	22.1	30.3	33.9
Puzzolana " II. 1:3 " "	2.5	20.7	136.8	14.4	20.7	22.3	23.4	6.1	12.2	10.6	15.9	13.7	12.6	17.6	19.0

glass vessel with the cement, and observing whether it burst or no. Abnormal and mischievous expansion of cement may be classed under two heads: (i) expansion in air; (ii) expansion under water. The analyses of three cements that showed expansion of the first kind are:—

	A.	B.	C.	D.*
SiO <sub>2</sub> .....	19.73	20.16	21.85	21.47
Al <sub>2</sub> O <sub>3</sub> .....	8.49	6.19	7.20	6.97
Fe <sub>2</sub> O <sub>3</sub> .....	3.42	2.90	2.82	2.73
CaO .....	61.63	62.28	60.42	54.38
CaCO <sub>3</sub> .....	Trace.	..	1.34	7.52
CaSO <sub>4</sub> .....	3.16	..	1.93	1.85
MgO .....	1.95	3.76	0.83	0.81
H <sub>2</sub> O .....	1.63	3.05†	2.13	2.66
SO <sub>3</sub> .....	..	0.75	..	..

\* Analysis of sample C after remaining three months in sacks.  
† H<sub>2</sub>O and CO<sub>2</sub> = 3.05.

A became brittle and friable in two years; it then contained 8.75 per cent. of CO<sub>2</sub>. B had a tensile strength of 22 kilos. per sq. cm. after 84 days, and disintegrated in a year and a half. C failed in three months, but if previously exposed in sacks for three months (see note to D) it was permanent in air; it was also permanent in water without previous aëration. It therefore appears that the ultimate composition is not the reason for the expansion of the samples, but it must be ascribed to a want of homogeneity in the raw materials, together with imperfect firing, causing the formation of unstable bodies of little cementitious value, and readily decomposed by CO<sub>2</sub> to form CaCO<sub>3</sub>, thereby setting up a gradual expansion as the air acts from the outside upon the cement.

Cements which fail by expansion under water fall into three classes:—(a) Those containing easily oxidisable bodies, e.g., CaS, which expand on oxidation. One per cent. of CaS is injurious in Portland cement, but cement from blast-furnace slag may apparently contain as much as 5 per cent. without detriment. (b) Those containing free lime on account of imperfection of mixing. (c) Those, which although properly mixed and burned, contain too much free lime, magnesia, or calcium sulphate. The "firing test" ("Darrprobe," this Journal, 1889, 781) will detect the free lime, but not the calcium sulphate.

An account is given of the failure of the cement on *Stephan's Dome* in Vienna, for an abstract of which see this Journal, 1889, 781.

*Influence of Foreign Matters on Cement.*—Coloured plates of Portland cement made by mixing pigment with the cement, have sometimes failed from the action of the pigment on them; thus a red pigment containing 22 per cent. of sulphuric acid (2 free) caused the disintegration of a plate into the composition of which it entered to the extent of 17 per cent., owing to the formation of calcium sulphate. Magnesia, if present in such quantities as 16 to 18 per cent., brings about the destruction of cement by expansion.

For *Dyckerhoff's* researches in this direction see this Journal, 1889, 781.

*Candlot* has investigated the action of solutions of calcium chloride of various strengths on the setting of Portland cement, with the following results:—

CaCl <sub>2</sub> per litre (grms.)...	0	2	5	10	20	40	60	160	200	300
Time of setting (hours)...	25'	1	10	10	12	80	6	20'	9	8'

Thus the first effect is to retard it greatly, but a maximum is reached, varying with the cement used, between 10 and 40 grms. per litre. At a concentration below 60 grms. per litre, the normal formation of hexagonal plates of Ca(OH)<sub>2</sub> takes place, and the CaCl<sub>2</sub> is inert chemically. Above 100 grms. long needles of calcium oxychloride form, and the time of setting drops abruptly from six hours to 20 minutes. Bearing on this phenomenon is the solubility of Ca(OH)<sub>2</sub> in solutions of CaCl<sub>2</sub> of various strengths.

CaCl <sub>2</sub> per litre (grms.) .....	0	15	36	61	100
CaO dissolved per litre (grms.) ..	1.298	1.003	1.032	1.121	1.312

The fluctuation is quite comparable with that of the time of setting.

*Rinne* disputes *Candlot's* conclusions, and considers his deduction that calcium chloride in concentrated solutions retards the setting to be based on an error; he opines that *Candlot* used too little of the solution to allow of the proper hydration of the cement, having regard also to the avidity of CaCl<sub>2</sub> for water.

W. Michaelis advocates the judicious addition of finely-ground slag to Portland cement. Comparative experiments, extending over five years, on a cement of high quality, with and without the addition of 25 per cent. of slag, showed that the mixture possessed a higher strength, both in tension and compression, than the pure cement.—B. B.

*Hardening of Portland Cement.* Schöffner. Deutsche Töpfer u. Ziegler Zeit. 20, 597.

The author considers that the chief cause of the occasional failure of this mortar to harden is either presence of an excess of water during the mixing, or a want of moisture through premature desiccation.

From a series of experiments, the author concludes that mortar which has been kept damp for 24 hours after mixing, hardens perfectly when placed under water. On the other

hand, if left without any such precaution, the mortar becomes more or less tender.

It is therefore essential to keep the mortar damp for the first few days after preparation.—S. B. A. A.

*The Action of Air and other Gases on Cement.* Tomäi. Deutsche Töpfer u. Ziegler Zeit. 20, 697.

FRESHLY-GROUND cement was found to have a specific gravity of 3.162, and a weight per litre of 1,250 grms. After 28 days' storage these numbers were 3.154 and 1,242 grms. respectively, and after 90 days' exposure to air, 3.128 and 1,225 grms. Dry air, free from carbonic acid, dry carbonic acid, and especially moist carbonic acid free air, diminish the weight of cement. As regards the strength of cement, dry carbonic acid was found to exert a favourable influence, both the tensile and compressive breaking stress being increased. Dry air has a similar effect. Moisture weakens cement; a cement which tested at first 17.8 kilos. tensile breaking stress, and 201 kilos. compressive breaking stress, required, after 28 days' exposure to moist air, only 13.5 kilos. and 143 kilos. respectively. Sulphuretted hydrogen and calcium sulphide have but little influence on the specific gravity and weight per litre of cement, but the heat produced on mixing with water is diminished, and the time required for the cement to set is increased. The surface of fracture was found to have a greenish tint, due, no doubt, to the formation of sulphide of iron. The strength diminished from 17.8 kilos. tensile stress and 201 kilos. compressive stress to 12.9 kilos. and 137 kilos. respectively, after 28 days' exposure to sulphuretted hydrogen, and to 14.3 kilos. and 158 kilos. respectively, by the addition of 1 per cent. of calcium sulphide. The author is of opinion that the degree of heating and the time of setting of a cement depend on the aluminates and ferrates present in it, since these are the only bodies likely to be affected by the addition of calcium sulphide.—H. T. P.

*The Permeability of Roofing Tiles.* R. Boute. Thonind. Zeit. 1889, 13, 611.

THE natural porosity of roofing tiles, although useful in favouring the adherence of mortar, is objectionable inasmuch as it allows the percolation of rain-water, especially on slightly-sloping roofs. Glazed tiles are little better than the common kind, as the least flaw lets in water, which, once in, saturates the tile, and cannot freely evaporate because of its glazed surface. A better plan consists in giving the tiles, before burning, a clayey coating, by which their porosity is unimpaired while their permeability is much diminished. The same effect may be produced by steaming the tiles, and filling their pores with graphite. Roofs already existing may be protected by coal-tar or beet molasses. Besides the direct action of the latter, due to its gelatinous character, is the aid afforded by the dead mycelia of moulds which flourish during its change to acetic acid.—B. B.

*Portland Cement Concrete.* V. Brausewetter. Thonind. Zeit. 1889, 13, 624.

THE failure of concrete cast *in situ* (leaving out of consideration those cases in which a cement with a natural tendency to expand and disintegrate has been used) may be attributed either to error in its preparation or to the use of hydraulic lime in place of Portland cement. With regard to the former, it must be remembered that the least possible quantity of water should be used, as a needless excess causes abnormal expansion while setting, followed by considerable contraction. Mixtures not too rich in cement should be employed, a lesson to this effect being taught by the case of "Stephansdom" (this Journal, 1889, 781). Milk of lime is preferable to plain water, as capillary cracks are thus avoided. A sand of low specific gravity is desirable, to obviate the risk of separation from the rest of the concrete by subsidence, owing to its being necessarily of somewhat thin consistency.—B. B.

*Damaged Asphalt Paving.* W. Dehnhardt. Centralbl. f. Bauverwalt. 9, 94.

AN asphalt street pavement which had been laid down 2½ years previously, was found to be much damaged. Soon after being repaired, faulty places again appeared. The damaged places were soft, and saturated with a kind of tar, and exhibited on the surface a number of radiating fissures 2—3 cm. deep. This peculiar appearance was found to be due to an escape of illuminating gas from pipes laid underneath the pavement. The heavy hydrocarbons, and especially the benzene, contained in the gas, were absorbed by the asphalt, rendering the pitch contained in the latter soft, and thus destroying the coherence of the mineral portion of the pavement.—H. T. P.

PATENTS.

*An Improved Process for Hardening, Preserving, and Preparing Oolitic and other Limestones.* G. J. Randall, Tooting Graveney. Eng. Pat. 15,041, October 19, 1888. 4d.

THE limestone is heated and then immersed in a solution of milk of lime, acetic acid, and cane sugar molasses. The stone so treated is next rubbed to a face with fine grit, and allowed to dry in the atmosphere, after which it is dried at a moderate temperature, and allowed to cool slowly.

—E. G. C.

*Process for the Crystallisation of Silica in a Compacted Mass.* A. C. Ponton, Parkstone, B. L. Mosely, Hastings, and C. Chambers, Hastings. Eng. Pat. 15,256, October 23, 1888. 6d.

"LIGHT silicas" are powdered, mixed with water, and in this condition compressed into moulds and exposed to the heat of a porcelain kiln.—E. G. C.

*Improvements in Apparatus for Calcining Cement Materials, Hydraulic Lime, Plaster of Paris, and like Substances.* J. W. H. James, Westminster. Eng. Pat. 16,234, November 9, 1888. 8d.

THE inventor claims a rotating drum, so constructed that the flames and products of combustion, and substances to be calcined, all travel in the same direction; also an annular fuel supply way, and central feed way for substances to be calcined. Other claims are detailed at length in the specification.—E. G. C.

*Improvements in Artificial Stones or Blocks applicable for Grinding, Crushing, Granulating, Polishing, and other similar Purposes.* C. O. Weber and F. W. Follows, Manchester. Eng. Pat. 16,770, November 19, 1888. 6d.

THE base or bulk of the stone or block is formed of ground or granulated flint, or granite, while the face or grinding surface is preferably made of granulated emery, corundum, or other hard abrasive material. The cementing mixture contains oxide of magnesium, hydrochloric acid, sulphate of iron, and chloride of barium.—E. G. C.

*Improvements in Colouring and Ornamenting Portland Cement for the Manufacture of Imitation Grained or Figured Marble, Mosaics, Ornamental Slabs, Tiles, and other Forms, either Plain or in Mixed Colours.* W. Cussans, London. Eng. Pat. 17,257, November 27, 1888. 6d.

AMONG the materials used in admixture with the Portland cement are the oxides of iron, copper, manganese and chromium, also chalk, barytes, brass turnings, copper borings and tin filings.—E. G. C.

*Improvements in the Manufacture of Cement and in Apparatus employed therein.* J. Hargreaves, T. Robinson, and J. Hargreaves, Widnes. Eng. Pat. 17,363, November 29, 1888. 8d.

THE following claims are made:—

- (1.) For mixing slurry by giving a "circular rotating or return motion" to the raw materials ("lime, clay, and water") by means of air or other suitable gas. For this an apparatus is used consisting of a closed vessel with a conical bottom, and capable of withstanding considerable pressure, through the cover of which passes a pipe conveying the agitating gases, and which is provided with an exit a little above the bottom for the fine slurry, and one at the extreme bottom for the coarser particles.
- (2.) For a filter for slurry, consisting of two concentric vessels, the inner of which is perforated and covered with filter cloth. The slurry is forced into the annular space between them, and the water flows through the filter cloth into the inner vessel, and so away.
- (3.) For a continuous filter for slurry, consisting of a closed conical vessel, having perforated walls covered with filter cloth set in any convenient position, into the larger end of which the slurry is forced, emerging at the smaller end, the water passing away through the sides of the vessel. The semi-dry slurry is cut into briks as it comes out, and is carried by an endless band through a drying oven.
- (4.) For drying slurry when it is intended to be burnt by the cylinder process, by means of a tower containing a vertical shaft bearing scrapers which pass over the surface of annular trays or shelves concentric with it. The slurry is fed in at the top, and falls from shelf to shelf, being stirred by the scrapers while exposed to a current of hot gases, preferably the waste product of the burning process,—excess of oxygen being avoided when it is desired to eliminate free sulphur from the slurry.
- (5.) For "weathering" ("aërating") the finished cement by means of an artificial atmosphere of carbon dioxide or that gas together with aqueous vapour, and for an apparatus therefor essentially similar to that described under (4). The carbon dioxide may be obtained from the waste gases of kilns, &c., cooled and freed from soot by passage through a scrubber.—B. B.

*Improvements in Apparatus for Use in the Preparation of Slurry for the Manufacture of Cement and in Mixing Slurry with Fuel.* W. Joy, Northfleet. Eng. Pat. 18,491, December 18, 1888. 1s. 1d.

THIS invention relates to improvements in the arrangements of the bars of the grids or gratings of the exits of the wash mills, and to means by which the flow of slurry to the mill-stones is automatically controlled. For details the specification with its nine sheets of drawings must be consulted.—E. G. C.

*A Weather- and Acid-Proof Enamel for Building Materials and the like, and a Method of Producing the Enamel.* J. Stiel, Cologne, Germany. Eng. Pat. 8597, May 23, 1889. 4d.

THE following ingredients are used:—Potash, soda, cryolite, apatite, fluorspar, and minium. Oxides are added according to the colour required, and the mixture is melted in a suitable furnace.—E. G. C.

*An Improved Composition for Covering Walls and the like.* G. F. Redfern, London. From J. G. Maardt, Copenhagen, Denmark. Eng. Pat. 11,922, July 26, 1889. 4d.

A Mixture of 75 parts of carbonate of lime and 25 parts of kaolin is made into a stiff paste with water, calcined, pulverised, and combined with 5 parts of calcined magnesite.—E. G. C.

*Improvements in Utilising the Waste Product from Sewage Works for the Manufacture of Bricks, Tiles, Quarries, Building Blocks, Slabs, and the like.* T. Shaw, Manchester. Eng. Pat. 12,623, August 10, 1889. 4d.

THE sludge is mixed with an equal quantity of clay. The plastic compound formed is dried and burnt.—E. G. C.

*Improvements in the Process of Treating Cements and Hydraulic Limes.* S. Jørgensen, Faxe Ladeplads, Denmark. Eng. Pat. 14,281, September 10, 1889. 4d.

BLAST-FURNACE slag moderately low in lime, or natural products of similar composition, are treated with sulphuric acid in proportion to be determined by trial, 40 lb. of slag to 25 lb. of sulphuric acid being suitable normally. A dry grey powder is thus obtained, consisting of free silica and metallic sulphates which may be added to the extent of 1·7 per cent. to good Portland cement, 3·3 per cent. to inferior qualities, and 3·5 to 7·0 per cent. to slag cement, increasing, it is alleged, their hardening properties.—B. B.

*Improvements in Waterproof Slabs and Blocks for Structural Purposes.* D. Nicoll, London. Eng. Pat. 14,733, September 18, 1889. 4d.

ONE or more of the bituminous substances described by the patentee in previous patents of dates ranging from February 5th, 1866, to May 11th, 1887, are mixed with grit, shingle, sand, or chalk, and the compound cast into moulds shaped so as to form tenons and mortises; the blocks thus produced are placed one above the other so that the tenons and mortises correspond, a space being left sufficient to allow a flame from such a source as a spray of mineral oil to play between them, and fuse them together. By this means large blocks may be built up, which may be used for sea walls, tunnels, &c.—B. B.

*Improvements in the Manufacture of Artificial Asphalt.* J. Erslev, Copenhagen, Denmark. Eng. Pat. 15,669, October 5, 1889. 4d.

EIGHTY to 90 parts of resin ("Resina pini flava") are melted together with 20 to 10 parts of coal-tar ("Pyrooleum lithanthracis") and 100 parts of sand or clay, and 200 to 400 of rough gravel added. The product may be used in the same way as natural asphalt, than which it is said to be better and cheaper.—B. B.

## X.—METALLURGY.

*Preparation of Manganese from Manganese Chloride and Magnesium.* E. Glatzel. Ber. 22, 2857—2859.

CRYSTALLISED manganese chloride is first dehydrated in a porcelain basin, powdered and 100 grms. of the powder mixed with 200 grms. of dry powdered potassium chloride.

This mixture is brought into a Hessian crucible stamped down, closed with a loose lid and melted down in a wind furnace. A moderate heat only is requisite. The crucible is now withdrawn and 15 grms. of magnesium thrown in in pieces of 3 to 4 grms. at intervals of two to three minutes.

The fused mass should not be heated to too high a temperature before the addition of the magnesium, otherwise the reaction is so violent that the contents of the crucible are thrown about. A dull red heat necessary to keep the mass molten is sufficient.

The crucible is now covered with the lid and returned to the furnace, heated strongly again, and allowed to cool in the furnace. On opening the crucible a lump of manganese is found weighing from 20 to 25 grms.

The metal thus obtained has a metallic fracture, which does not oxidise in dry air, is non-magnetic, and has a specific gravity of 7·3921 at 22°.—J. B. C.

*On the Hardening and Tempering of Steel.* W. C. Roberts-Austen. A Lecture before the British Association, Newcastle-on-Tyne, 1889.

This lecture is as perfect and comprehensive a consideration of the subject as it is possible to obtain in the space at the disposal of the author. The first part is devoted to the opinions expressed by the various writers from the earliest periods down to the present day. Each branch of the subject is treated historically, and full references are given to all the works of the writers quoted. After this extensive retrospect, the author proceeds to discuss in more detail the more recent researches and theories. In referring to the confusion at present existing in the use of the words "hardening," "tempering," and "annealing," he gives the following definitions:—"Hardening" is the result of rapidly cooling a strongly heated mass of steel; "tempering" consists in re-heating the hardened steel to a temperature far short of that to which it was raised before hardening—this heating may or may not be followed by rapid cooling; "annealing" consists in heating the mass to a temperature higher than that used for tempering, and allowing it to cool slowly.

The hardening and tempering of steel is largely the result of the form which the carbon therein is made to assume, but the main facts are not fully explained by the change in the mode of existence of the carbon.

It is possible to obtain a perfectly soft steel by rapid cooling from a certain temperature, in which the carbon is shown by analysis to exist as "hardening carbon." Further explanations are forthcoming by the assumption of the existence of allotropic forms of iron. The consideration of this important branch is entered into at some length.

Chernoff showed that steel could not be hardened by rapid cooling until it had been heated to a definite temperature—to a degree of redness which he called  $\alpha$ .

Gore found that a molecular change could be produced by heating an iron wire, which exhibited a momentary elongation during cooling.

Barrett discovered that an iron wire suddenly glows at a certain stage during cooling, a phenomenon which he designated "recalescence."

In 1879 Wrightson called attention to the abnormal expansion of carburised iron at high temperatures. Pionchon showed that at 700° the specific heat of iron is altogether exceptional, while Le Chatelier detected that at the same temperature a change occurs in the curve representing the electro-motive force of iron; both of these latter experimenters concluding that they had obtained evidence of the passage of iron into an allotropic state. Osmond in his quite recent researches has studied by the aid of a chronograph the molecular changes which take place in pieces of iron and steel during slow heating and slow cooling, using as a thermometer a thermo-electric couple of platinum and of platinum containing 10 per cent. of rhodium, converting the indications of the galvanometer into temperatures by Tait's formulae. A mass of electro-deposited iron in cooling from 2,000° to 870° falls uniformly at the rate of about 2.2° a second, but at 858° there is a break and 26 seconds are absorbed while the temperature falls through an interval which previously would have occupied six seconds, after which the temperature again decreases uniformly. In the case of mild steel there are two such breaks, each of which occurs at a temperature different to that in the case of pure iron. With higher carbon steels the two breaks gradually get nearer to one another until they coalesce in the case of steel containing 1.25 per cent. of carbon, the single break in this instance lasting 76 seconds at a temperature of about 680°.

"Now, it may be urged, evidently the presence of carbon has an influence on the cooling of steel when left to itself; may it not affect molecular behaviour during the rapid cooling which is essential to the operation of hardening? We know that carbon, during rapid cooling, passes from the state in which it is combined with the iron into a state in which it is dissolved in the iron; we also know that during slow cooling, this dissolved carbon can re-enter into combination with the iron, so as to assume the form in which it occurs in soft steel. Osmond claims that this

second arrestation in the fall of the thermometer corresponds to the recalescence of Barrett, and is caused by the reheating of the wire by the heat evolved when carbon leaves its state of solution and truly combines with the iron." If it is desired to harden steel the metal must be rapidly cooled before its temperature has fallen to 650°, otherwise the presence of carbon will be unavailing. A break occurs, however, in electro-deposited iron containing no carbon, a break indicating an evolution of heat with which carbon has nothing to do, and which must indicate therefore some molecular change in the iron itself. "Osmond argues thus: There are two kinds of iron, the atoms of which are respectively arranged in the molecules so as to constitute hard and soft iron, quite apart from the presence or absence of carbon. In red-hot iron the mass may be soft, but the molecules are hard, let us call this  $\beta$ -iron; cool such red-hot pure iron, whether quickly or slowly, and it becomes soft; it passes to the  $\alpha$  soft modification, there is nothing to prevent its doing so. It appears, however, that if carbon is present, and the metal be rapidly cooled, the following result is obtained: a certain proportion of the molecules are retained in the form in which they existed at a high temperature—the hard form, the  $\beta$  modification—and hard steel is the result."

"This molecular change from  $\beta$ -iron to  $\alpha$ -iron during the slow cooling of a mass of iron or steel is, according to Osmond's theory, indicated by the first break in the curve, representing the slow cooling of iron, as is proved by the fact that it occurs alone in electro-iron. A second break, usually one of much longer duration, marks the point at which carbon itself changes from the dissolved or hardening carbon to the combined carbide-carbon. It follows that if steel be quickly cooled after the change from  $\beta$  to  $\alpha$  has taken place, but before the carbon has altered its state—that is, before the change indicated by the second break in the curve has been reached—then the iron should be soft, but the carbon, hardening carbon; and as such, the action of a solvent should show that it cannot be released from iron in the black carbide form. This proves to be the case, and affords strong incidental proof of the correctness of the view that two modifications of iron can exist. It will be seen, therefore, that although the presence of carbon is essential to the hardening of steel, the change in the mode of existence of the carbon is less important than has hitherto been supposed."

The experiment was then shown of heating a bar of high-carbon steel to redness, fixing it in a horizontal position at one end and hanging on to the other a weight insufficient to bend it when at its hottest. The bar remained straight as it cooled until a certain point was reached, when it commenced to bend slightly before becoming permanently rigid. This point has been found to coincide with that at which, according to Osmond's theory, molecular change takes place, and in this instance, being a high-carbon steel, at that point also at which the hardening carbon changes into carbide-carbon. Advantage has been taken of this fact to straighten distorted steel axes.

An experiment was also shown to demonstrate the weldability of steel in the Bunsen burner. The two ends of a newly fractured steel rod were placed together, covered with platinum foil to exclude air, and heated in a Bunsen flame. The metal welded without pressure, so that it was difficult to break with the fingers, although the temperature had not attained a red heat.

As regards the behaviour of other metals on iron, Osmond has shown that manganese enables steel to harden very energetically, and that when 12 to 20 per cent. is present there is no break whatever in the curve representing slow cooling. The iron shows no sign of any change such as occurs in other cooling masses of iron, and it may be assumed that it remains in the  $\beta$  condition. If this be so then manganese-iron alloys should be hard whether they are cooled rapidly or slowly, and this is found to be the case. Again "red-hot iron is non-magnetic. Hopkinson has shown that the temperature of recalescence is that at which iron ceases to be magnetic. It may be urged that  $\beta$  iron cannot therefore be magnetised. Steel containing much manganese cannot be magnetised, and it is therefore fair to assume that the iron present is in the  $\beta$  form." When small

quantities of manganese are present the change of form of the iron is not altogether prevented but is retarded.

"Tungsten possesses the same property as manganese, but in a more marked degree. Chromium has exactly the reverse effect, as it enables the change of hard  $\beta$  iron to a soft iron to take place at a higher temperature than would otherwise be the case, and this may explain the extreme hardness of chromium steels when hardened in the same way as ordinary steels."

The points *a* and *b* of Chernoff change in position with the degree of carburisation of the metal, and in a highly carburised steel the point *a* corresponds to that point at which Osmond considers that iron in slow cooling changes from the  $\beta$  to the  $\alpha$  modification. It is impossible to harden steel by rapid cooling after the temperature has fallen below *a*. If steel be heated to a temperature above *a* but below *b*, it remains fine-grained however slowly it is cooled. If it be heated above *b* it assumes a crystalline granular structure whatever may be the rate of cooling. The crystalline structure is destroyed by forging. The quenching in oil of large masses of steel also seems to break up the crystalline structure. In such cases the metal is hardened only at and near the surface, but the cooling of the interior is more rapid than otherwise would be the case and the formation of large crystals is arrested. This prevention of crystallisation the author believes to be the great merit of oil quenching, which, as regards large masses of metal, is certainly not a true hardening process.

The metal of a complicated steel casting probably contains much hardening carbon, and the mass is crystalline. The effect of annealing is to permit the change of carbon from the hardening to the carbide form and incidentally to break up the crystalline structure, with the result that a stronger and more extensible casting is obtained.

"Looking back over all the facts, it will be evident that two sets of considerations are of special importance: (1) those which belong to the relations of carbon and iron; and (2) those which contemplate molecular change in the iron itself. The first of these has been deliberately subordinated to the second," not from any want of abundant facts relating to it but because the author believes "that the possibility of molecular change in the iron itself, which results in its passage into a distinctive form of iron, is at present the more important subject for consideration, not merely in relation to iron, but as regards the wider question of allotropy in metals generally." Chemical elements act on each other in accordance with the law of Mendeléeff, and it may be that the relation between small quantities of elements and the mass of metal containing them may be in some way or other connected with the same law. It has already been proved in the case of gold, where the tenacity is the physical property affected.

In the case of iron it is difficult to say in what direction the law will show itself. "Possibly the direct connexion with the periodic law will be traced by the effect of a given element in retarding or promoting the passage of ordinary iron to an allotropic state; but the future of steel will depend on the care with which we investigate the nature of the influence exerted by various elements on iron, and the thermal treatment to which it may most suitably be subjected."—A. W.

#### *The Manufacture of Galvanised Iron.* B. Preu. Oesterr. Zeits. Berg. u. Hütten. **37**, 447.

THE zincing tanks are made of wrought iron  $\frac{5}{8}$  in.— $2\frac{1}{2}$  in. thick, they are heated from the sides, but only on the lower four-fifths of their height. Three processes are in use.

(1.) Winiwarter's. To guard against the formation of hard zinc (a ferri-ferrous zinc containing not more than 5 per cent. of iron), and the consequent brittleness of the plates, the tank is nearly filled with lead and covered with a layer of zinc about 1 in. thick, 2 or 3 per cent. of tin being added to the zinc to promote crystallisation. The plates are heated, cleaned, and dipped into a mixture of two parts of a solution of zinc chloride (50° B.) and three parts of a solution of ammonium chloride (5° B.), diluted with water to 8° or 9° B. The sides of the tank are protected from the corrosive action of the zinc by a refractory lining

extending just down to the level of the lead. The disadvantages are that the zinc must be continually renewed, and the surface always kept bright and free from oxide.

(2.) The most general method is to fill the bath with zinc only, and protect it from oxidation by a coat of ammonium chloride. The zinc adheres firmly to the finished plates, but the fumes are objectionable.

(3.) Küller's patented process merely consists in the removal of the superficial layer of zinc from the galvanised plates by means of steel wire brushes. There is no economy of metal, as the patentee claims, but the appearance is greatly improved, and the plates are more suitable for better class goods.—S. B. A. A.

#### *Roasting Zinc Blende.* W. Minor. Chem. Zeit. **13**, 1602—1603.

IN practice the time required for the elimination of sulphur from different varieties of blende varies from two to six hours. To determine the cause of this variation, the author roasted carefully selected samples of blende, and analysed them at intervals. The following results were obtained:—

(1.) Composition of the ores before roasting.

No. of Sample.	Zn Total.	Zn not combined with S or SiO <sub>2</sub> .	S.	Fe.
I.	37.81	6.65	21.05	2.33
II.	31.92	4.75	27.94	13.12
III.	49.29	5.83	22.11	2.06
IV.	32.76	5.03	30.24	15.98

(2.) Percentage of S after roasting.

No. of Sample.	One Hour.	Two Hours.	Three Hours.	Four Hours.	Five Hours.	Six Hours.
I.	10.32	2.83	1.80	1.71	0.95	0.08
II.	24.33	21.95	20.65	17.02	2.15	0.18
III.	14.69	6.27	1.23	0.22	0.16	0.16
IV.	27.25	24.30	18.25	11.94	4.27	0.04

The author infers that the elimination of sulphur is retarded to a much greater degree by iron pyrites than by even considerable quantities of zinc silicate.—S. B. A. A.

#### *The Refining of Silver and the Separation of the Bismuth contained in it.* C. Rössler. Berg. u. Hüttenm. Zeit. **48**, 387.

IN Rössler's method, which is chiefly employed for the refining of silver containing bismuth, at first the greater part of the lead and then the bismuth pass into the slag. The slag is ground up fine and fused with soda and charcoal. The metal thus obtained is granulated and boiled with strong sulphuric acid in a small iron kettle till completely converted into sulphate, excess of acid being avoided. On cooling the mixture is extracted in a wooden vessel, lined with lead, and of 250 litres capacity, with 200 litres of sulphuric acid of 20° B., the temperature not being allowed to exceed 10° C. At this low temperature the bismuth sulphate is dissolved (about 5 kilos. of bismuth in each charge). The solution is drawn off the insoluble lead sulphate, and the silver dissolved in it precipitated by metallic copper. The bismuth remaining in the solution is removed by heating the latter to 50° C.; the solution is then cooled and again used for dissolving. The lead, as well as the separated silver, contain bismuth, which is removed by digestion with hydrochloric acid. The solution is added to the washed bismuth sulphate, and the bismuth is then precipitated by metallic iron.—H. T. P.



*New Furnaces for Quicksilver in Spain.* Eng. and Min. J.  
1889, 430—431.

THE *Revista Minera* of Madrid gives the following description of two new continuous furnaces recently introduced by "El Porvenir" Company in Asturias for the treatment of its quicksilver and arsenical ore.

**Double Retort Furnace.**—The Figures 1, 2 and 3 of Plate I. indicate sufficiently its arrangement. The circumstance which gives novelty to this furnace is the fact of the discharge mouth being partially open for the entrance of air, which can be done without inconvenience or danger of escape of the fumes, owing to the artificial draft which is produced by a hydraulic ventilator similar, except as to size, to that for the shaft furnace. The retorts are of cast iron, and have a common hearth in which is burned small coal.

Every hour and a half, 50 kilos, of finely crushed ore are fed through the hopper. When the ore is very rich, it may be fed in small pieces, lime being mixed with the ore when it contains 15 per cent. of quicksilver. In this manner 1,600 kilos. of rich ore are treated in 24 hours.

For the condensation there are two larger chambers, other two smaller, a wooden compartment with water on the bottom, and afterward the ventilator and the tubing which carries the fumes to the chimney, in which no signs of quicksilver are to be seen.

From the first condensing chambers 90 per cent. of the quicksilver produced is recovered, and from the second nearly all of the remainder, for from the wooden compartment scarcely one kilo. of metal is produced after a year's work. A great part of the quicksilver runs out during the operation through small holes pierced in the chambers near the floor.

According to Rodriguez, the loss in this furnace does not amount to 1 per cent., and the cost of treatment of one ton of ore is 1 dol., which cost can be materially reduced if, in place of two, six retorts be placed in the furnace. The hearth consumes each 24 hours  $3\frac{1}{2}$  metrical hundredweights of fine coal, worth 37 cents. With this furnace not only is the treatment very perfect, but the great advantage of doing away with bricking the ore is gained, which costs more than 20 cents for each ton of ore.

**Stack Furnace.**—The form of this is slightly conical; it is 2 metres in diameter above the grate and 1·85 metres at the height of the throat, being terminated by a round arch, which has the charging hopper in its upper part. The height of the furnace may be considered as divided into two parts: the lower or hearth chamber being 2·85 metres and the upper portion 2·85 metres. See Plate II.

The hearth forms a chamber of less diameter than the rest of the furnace. At 85 cm. from the floor is situated the grate which is 1·10 metres in diameter, and with this dimension the walls continue vertically 80 cm. higher (Fig. 4a); above this a conical portion of 50 cm. which at the top has a diameter of 60 cm., then a cylinder 60 cm. high, covered by a round arch, pierced with holes, through which pass the products of combustion of the hearth. In order to give to this part of the furnace sufficient strength to support the weight of the ore, there are constructed over the grate, as is shown in Fig. 4a, two low arches, which sustain five courses of bricks, which in their turn support the upper portions of the furnace. The central opening O is protected by a cast-iron cap in such a way that the exit of the fumes can proceed without the admission of ore to the hearth. With this same object the other openings (a) below the central one have the angular section shown in Fig. 4a. The calcined ore is withdrawn through the doors P. The furnace chamber has externally a conical form, and is covered with iron.

All the interior of the furnace is constructed of fire-brick, of a thickness of 30 cm.; surrounding this lining there are four roony hollow spaces of 10 cm. in width and 80 cm. in length, which rise to the top of the body of the furnace and communicate below with the hearth at the level of the grate. A tower of ordinary material, 60 cm. thick, encloses the whole furnace. The throat is a pipe of cast iron 50 cm. in diameter and about 4 metres in length, placed with a slope toward the condensation chambers, where it branches off so as to communicate separately with the two series.

The condenser is composed of four chambers, each of some 10 metres capacity, placed two on the right and two on the

left of the pipe conducting the fumes from the furnace, each branch communicating with the upper part of the first on each side. The communication between the chambers is made through two little openings of rectangular section near the floor, sloping towards each of the sides of the chamber and into a conduit through which is running a stream of water. As is shown in Fig. 3a, the sooty precipitate, which formed, consisting almost exclusively of arsenious acid mixed with quicksilver, can be driven down immediately to the very foot of the chambers, where the quicksilver which is produced is run off through the channel *q* (Fig. 1a) to the receptacles for it. These chambers communicate by means of sloping iron pipes with four other smaller ones of similar construction, and placed in line at a higher level. The chimney, placed at 100 metres distance, is constructed so as to take advantage of steep ground, and its mouth is partially closed with loose bricks, over which coke is spread to the thickness of 30 cm., a very simple arrangement, which diminishes the smoke to such an extent that from a distance the furnace does not seem to be working.

This furnace calcines  $8\frac{1}{2}$  tons of ore in 24 hours in pieces from 800 to 600 cc. and larger. The charges are fed every 75 minutes, each charge weighing 443 kilos., coke amounting to 42½ kilos. in 24 hours being mixed with the ore. The calcination is very good, the ore when discharged not containing more than 0·01 to 0·02 per cent. of quicksilver.

—O. H.

For Plates I. and II. see pages 78 and 79.

## PATENTS.

*Improvements in or connected with the Coating of certain Metals with other Metals or Alloys.* W. Charnock, Warrington. Eng. Pat. 17,476, November 30, 1888. 6d.

In coating iron and steel with zinc tin or terne, an alloy harder and less fusible than the coating metal itself, and of less value, as a covering agent, collects at the bottom of the tank in which the process is conducted. The patentee subjects the articles to be coated to a bath of this hard alloy in a separate tank and finishes them in one of the pure metal, thus effecting a saving in the latter.—B. B.

*Manufacture or Production of Metallic Alloys.* P. M. Parsons, Blackheath. Eng. Pat. 187, January 4, 1889. 4d.

ONE HUNDRED parts by weight of iron, 50—105 of tin, 5—40 of manganese, 3—50 of aluminium, are melted together and cast into ingots. The mixture is used for adding to copper, brass, gun-metal, or bronze in the proportion of 1—5 parts by weight for every 100 of the copper or copper alloy. For adding to bronze or gun-metal the quantity of tin mentioned above may be increased, but should be kept within the limits there defined for brass or yellow metal. Similarly a larger quantity of manganese (which may be used as rich ferro-manganese) may be added to the former than to the latter. The function of the aluminium is to lessen the corrodibility, increase the fluidity, and raise the strength of the product. —B. B.

*A New Fluxing or Protecting Material for Use in the Welding, Brazing, and Coating of Metals.* P. P. Craven, Forest Gate. Eng. Pat. 15,887, October 9, 1889. 4d.

A SUBSTANCE found near Port Augusta, S. Australia, as yet unnamed, and having an average composition of "oxide of iron" 2·90, alumina 4·78, calcium carbonate 0·95, potash 0·15, soda 0·21, magnesia 0·35, lead sulphide 0·12, water 0·58, silica 89·69 parts per cent. respectively, is used by the patentee as a brazing and welding flux, and as a covering for a bath of one metal (e.g. copper) with which another (e.g. iron) is to be coated. By its means he claims to be able "to weld iron to iron, copper to copper, steel to steel (even cast steel to cast steel), or any one of these metals to either of the others with great facility."—B. B.



## QUICKSILVER FURNACE.—PLATE I.

Fig. 1.

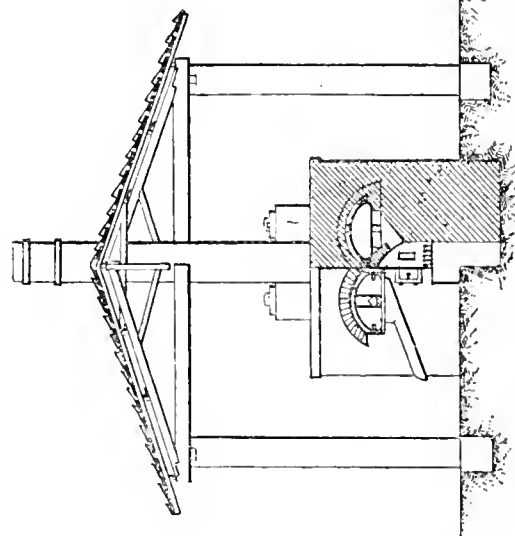


Fig. 2.

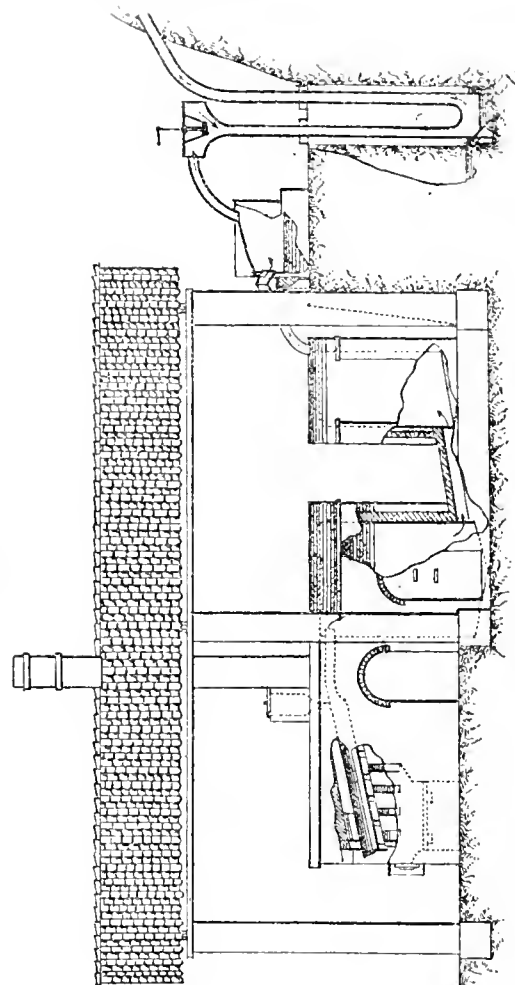
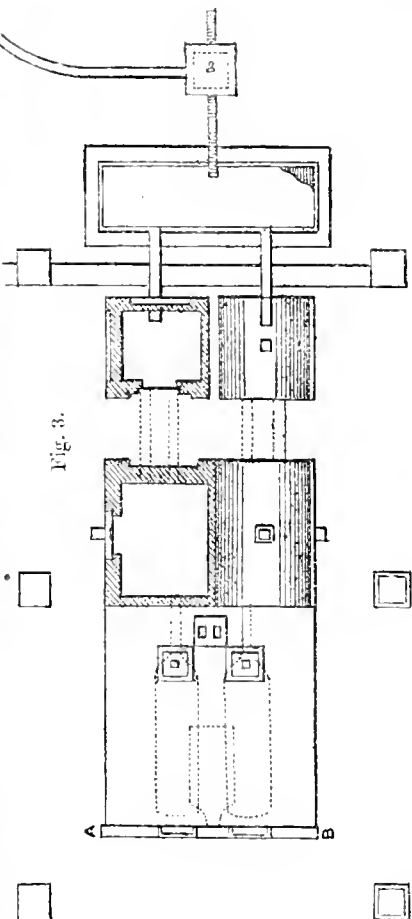


Fig. 3.



Sectional View of First Chamber.

Fig. 1a.

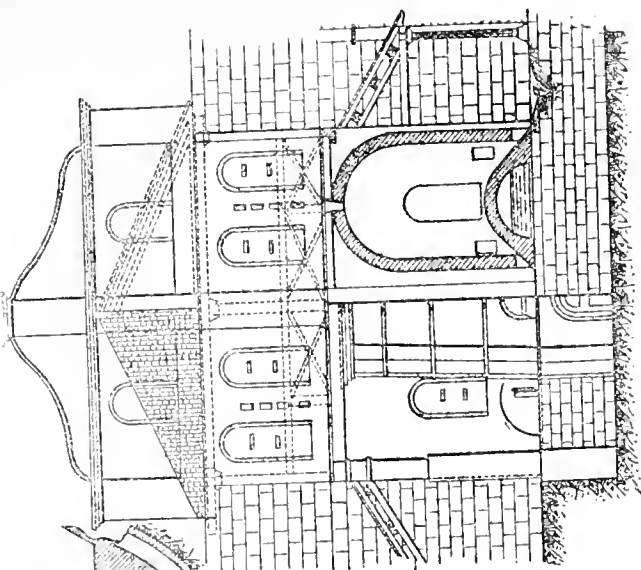
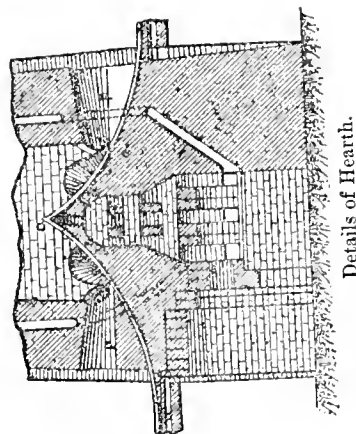


Fig. 4a.



QUICKSILVER FURNACE.—PLATE II.

Fig. 3a.

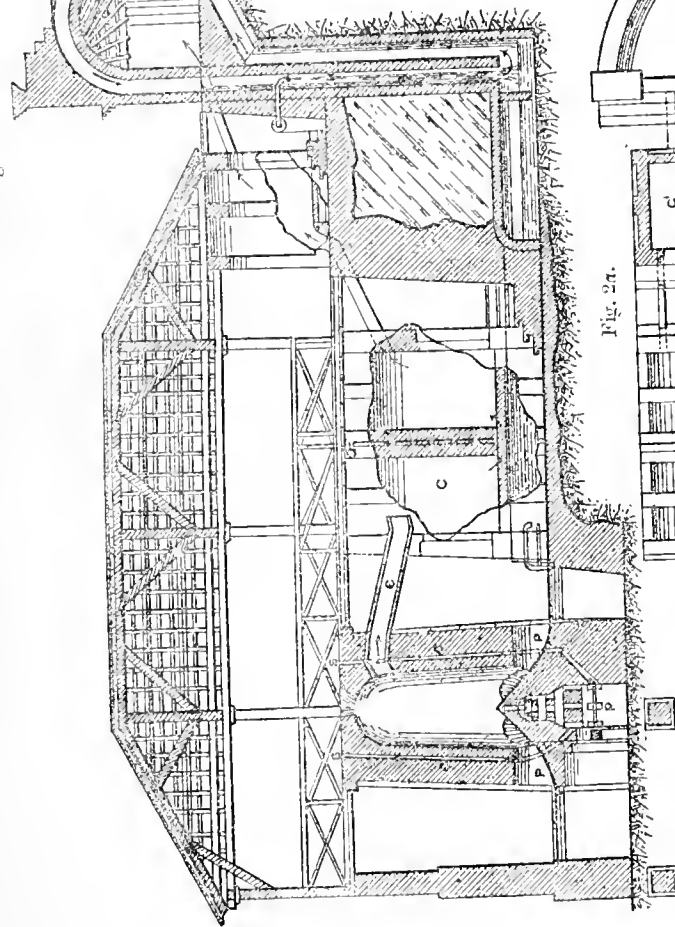
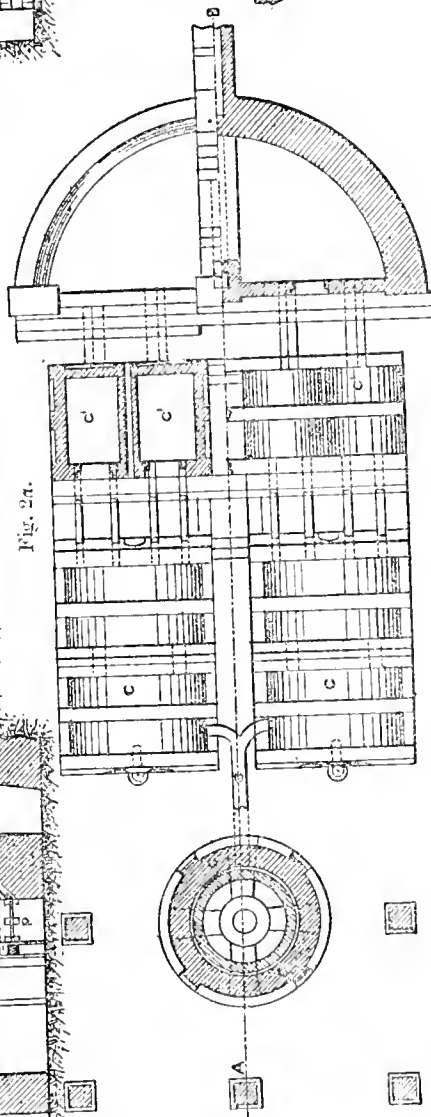


Fig. 2a.



## XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

*The Electrolysis of Sodium Chloride Solution with Direct Production of Chlorine and Crystallised Soda.* W. Hempel. Ber. 22, 2475—2478.

See under VII., page 65.

*Electrolytic Separation of Cadmium from Copper.* Smith and Frankel. J. Franklin Inst. 1889.

See under XXIII., page 108.

*Action of an Electric Current on Sulphocyanides.* Smith and Frankel. J. Franklin Inst. August 1889.

It is found that when an excess of potassium sulphocyanide is present in a solution of nitrate or sulphate of manganese a greyish white compact deposit of metallic manganese is obtained at the cathode when a feeble current is used. Without the sulphocyanide oxide of manganese is produced at the anode. Nickel, cobalt, iron, and several other metals separate easily from cold sulphocyanide solutions when a weak current is used.—W. W. H. G.

### PATENTS.

*An Improved Galvanic Cell or Battery for Electric Lighting or for other Purposes.* S. Miller, London. Eng. Pat. 14,322, October 5, 1888. 8d.

THE outer cell is lined with carbon, corrugated on its inner surface. A porous pot, inside this, is preferably divided into two compartments, a larger and a smaller, the former containing water and a zinc rod, the latter acid to acidulate the water by diffusion through the partition. The depolarising solution preferred is described in Eng. Pat. 14,323 (following abstract). Supply, overflow, and discharge pipes may be provided if desired.—E. T.

*An Improved Electrolyte or Depolarising Solution for Galvanic Batteries.* S. Miller, London. Eng. Pat. 14,323, October 5, 1888. 4d.

A SOLUTION is made up in the following proportions, which may, however, be slightly varied. To water, 40 oz., add sulphuric or hydrochloric acids, or a mixture of the two, 20 oz.; then sodium nitrate, 6 oz., potassium bichromate, 1½ oz., both finely powdered; lastly, sodium chloride, 1 oz. Proportions and methods of preparation may be slightly varied.—E. T.

*Improved Method of and Apparatus for the Formation of Organic Oxides and the Oxidation of Matter Suspended or Dissolved in Liquids.* J. G. Lorrain, London. Eng. Pat. 16,093, November 7, 1888. 6d.

See under XVIII. B., page 97.

*Improvements in Storage or Secondary Batteries.* H. H. Lake, London. From H. Walter, Cincinnati, U.S.A. Eng. Pat. 16,479, November 13, 1888. 8d.

THE electrodes are formed of carbon or of some base metal coated with one of the noble metals, such as gold or platinum, the electrodes being surrounded by closely-packed granulated carbon, kept in position either by bags of indestructible material or by insulating partitions. The solution employed consists of acetate of lead dissolved in vinegar, to which some red lead is added.—B. T.

*Improvements in the Manufacture of Cyanogen Compounds from Sulpho-cyanides.* T. Parker and A. E. Robinson, Wolverhampton. Eng. Pat. 17,447, Nov. 30, 1888. 4d.

See under VII., page 67.

*An Improved Separator for the Plates of Primary and Secondary Galvanic Batteries.* J. Mosceley, Manchester. Eng. Pat. 17,642, December 4, 1888. 6d.

A STRIP of vulcanite, celluloid or similar material, of a width greater than its length, has a shape given to it, such as might be obtained by the following operations:—It is first bent over the plate at its middle, so that the two halves or limbs lie with their width parallel to the faces of the plate. Each limb is then twisted round its own axis to 90° from its former position, so as to bring the width of the limb perpendicular to the surface of the plate. In practice, the above shape may be given by moulding or by bending while hot.—E. T.

*Improvements in Voltaic Batteries.* H. J. Harris and J. Garford, London. Eng. Pat. 17,694, December 4, 1888. 11d.

THE carbon plates are formed with a stud to them, this stud passing through the bottom of the cell to serve as a contact piece. Separated from the carbon chamber by a porous partition is the zinc chamber, the end of the zinc also passing through the bottom of the cell. These studs are then connected up as desired with suitable metal conductors, and they are buried in a non-conducting cement, which will withstand the action of the fluids used in the battery. A cam-shaped moveable handle at the top is arranged for jamming the lid hard down, and also for ease of transport. A solution of sulphuric acid, nitrate of soda, and bichromate of soda in water is used in the carbon cells, and water slightly acidulated with sulphuric acid in the zinc cells.—B. T.

*Improvements relating to Secondary Batteries.* J. L. Huber, Hamburg, Germany. Eng. Pat. 18,589, December 19, 1888. 8d.

A NETWORK, grating, or screen of suitable material is embedded in the active mass of the plate, and perforations are made in the active mass to allow of circulation of the electrolyte, and also to give greater surface to the plate.—B. T.

*Improvements in Electrical Accumulators.* J. C. and G. Fuller, London. Eng. Pat. 18,636, December 20, 1888. 4d.

THE plates are separated by porous partitions or porous pots.—E. T.

*New or Improved Apparatus for enabling Temperatures to be Ascertained at a Distance.* J. G. Lorrain, London. Eng. Pat. 18,752, December 22, 1888. 8d.

AT the station whose temperature is to be measured a high resistance wire or coil of wire of suitable material dips into a tube, in the lower portion of which is mercury or other suitable fluid. Wires lead from the two ends of the coil to the measuring station, where the resistance of the coil can be taken at any time. As the temperature at the first station rises, the level of the fluid in the tube rises, thus diminishing the resistance measured at the second station, and so enabling the temperature of the first place to be ascertained at any moment. Various appliances are described for use at the first station.—B. T.

*An Oblique-Meshed Grid Plate for Storage Batteries.* T. W. Bush, Brosceley. Eng. Pat. 18,978, December 29, 1888. 4d.

EACH perforation consists of a hole passing obliquely downwards (by preference) from each face of the plate into its substance, and either joining in the middle with its fellow from the other side, or being separated from it by a thin partition.—E. T.

*An Improved Plate for Electrical Storage Batteries.* M. Doubleday, Nottingham. Eng. Pat. 18,981, December 29, 1888. 4d.

GRIDS are used constructed with any number of larger holes on one face, and a greater number of smaller holes on the other face, so arranged that one or more of the smaller holes opens into each of the larger ones. Two of these grids are then fastened together to form each plate, the larger holes being brought face to face so that a number of chambers are formed inside, with small openings to the two outer faces.—B. T.

*Improvements in Electric Batteries.* J. Y. Johnson, London. From F. Gendron, Paris, France. Eng. Pat. 19,058, December 31, 1888. 8d.

A POROUS pot is employed which resembles those used in Grove cells, except that, instead of going straight across the cell, it pursues a deeply zig-zagged course. The outer cell has its walls lined with carbon, with leaves of carbon passing from the walls into each bend of the zig-zag. Inside the porous cell, in each bend, is a zinc plate made up of several thicknesses of thin zinc held in a framing of conducting material. A small compartment communicates with the outer chamber by a hole near the bottom of the latter. Into this compartment the spent liquor flows as fresh liquor is admitted, and passes off by an overflow pipe. The porous pot is provided with an arrangement on the same principle.—E. T.

*Improvements in Nickel Plating Engraved Rollers.* J. Parkinson, Lancaster. Eng. Pat. 702, January 14, 1889. 8d.

THE rollers are continuously or intermittently "scratch-brushed" in the nickel-plating baths themselves. Mechanism is described for carrying this into effect automatically. The rollers may be transferred from one bath to another, wrapped in india-rubber cloth or such material. The roller itself is kept in continuous rotation against a "scratcher," consisting of an elongated brush, to which a reciprocating motion is given; or of a belt with bristles on the inside, kept stationary while the roller revolves against it; or of a scratch-brush of the usual shape, kept revolving, and slowly travelling from end to end of the roller.—E. T.

*Improved Process of Treating Plates and the like for Facilitating the Removal of Galeano-plastic Deposits contained thereon.* A. J. Boult, London. From A. R. Reinfeld, Vienna, Austria. Eng. Pat. 3222, February 22, 1889. 6d.

TO facilitate the removal of galvano-plastic deposits, the metal on which the deposit is to be made is coated with a thin covering of nickel, and this nickel is then either oxidised or treated with some soapy or fatty substance to make it smooth. Very thin foils of deposited metal can then easily be removed from this surface.—B. T.

*Improvements in connection with Electric Fuses.* W. H. Scott and Lawrence, Paris, and Scott, Norwich. Eng. Pat. 6332, April 13, 1889. 6d.

INSTEAD of ordinary lead or tin fuse wire, a fine wire of high conductivity such as copper or silver is employed, and is enclosed in small glass tubes with suitable contact pieces at the end for fitting into the fuse holders.—B. T.

*Improvements in Signalling Apparatus for Indicating the Presence of Explosive Gases in the Atmosphere.* E. Stern and M. Kaufmann, Cologne, Prussia. Eng. Pat. 13,668, August 30, 1889. 6d.

A DEEPLY corrugated tube of thin metal, closed at the top, has its open end closed by a porous diaphragm or membrane. If brought into the neighbourhood of light gases, these diffuse through more rapidly than the heavier gas inside can diffuse out. The pressure rises therefore, and the tube by becoming elongated, makes an electric contact with a point fixed above it, and so gives warning by ringing an electric bell.—E. T.

*Apparatus for Automatically Charging Secondary Batteries or Accumulators.* S. C. C. Currie, Philadelphia, U.S.A. Eng. Pat. 13,864, September 3, 1889. 8d.

WHEN the battery has been sufficiently charged, spray is given off, which settling on two metal pieces, placed close together in the top of one of the cells, makes contact between them and so completes a circuit through an electro-magnet and a signal bell. The armature of the magnet is at once attracted, and breaks circuit between two contact pieces, so disconnecting the charging dynamo from the cells. Immediately afterwards, the armature establishes a contact through a second circuit, containing a resistance coil, so that a continuous small current passes round the same electro-magnet, and enables it just to hold up its armature in position. If, however, the E.M.F. of the cells fall owing to the approach of complete discharge, the magnet becomes too weak to retain its armature, which drops and re-establishes the charging circuit. If a short circuit should have occurred in one of the cells, the E.M.F. of the whole battery will be too small to enable the armature to be retained, and it will consequently continue to vibrate up and down, the signal bell ringing at the same time till attention has been attracted.—E. T.

*Improved Electrodes for Secondary Batteries.* G. E. Heye, Charlottenburg, Germany. Eng. Pat. 15,383, October 1, 1889. 4d.

IN the construction of accumulator plates metals should be used which have a "high powder of oxidising," and for this reason the inventor uses plates of alloys of chromium, manganese, wolfram, and lead, and also plates of oxides of chromium and wolfram.—B. T.

*Improvements in the Method of and Apparatus for "Forming" or Charging Plates for Electrical Accumulators.* S. C. C. Currie, Philadelphia, U.S.A. Eng. Pat. 15,446, October 1, 1889. 8d.

IN order to make a strong, compact plate, and therefore one which is but little liable to expansion, the active material, in the form of a plate surrounded by a rim or bead, is held in a strong frame, and wedged up by keys against the sides and ends of the containing cell, so that its expansive efforts during formation are exerted upon itself. Suitable perforated and corrugated plates of insulating material are inserted between the positive and negative plates, and the connexions are so arranged that each plate can be conveniently withdrawn at any time.—B. T.

*Improved Methods of Producing Aluminium and its Alloys by Means of Electricity.* L. Q. Brin, Paris, France, and A. Brin, London. Eng. Pat. 15,508, October 29, 1889. 6d.

THIS is an extension of the methods mentioned in Eng. Pats. 3547, 3548, 3549, 8746, and 8747 of 1888 (this Journal, 1889, 199, 399, and 548). The flux used consists of bauxite 100 parts, salt 125, borax 25, and these are fused in a furnace so constructed that an inert gas can be introduced when required, to carry the metallic vapours to any desired chamber, or to bring them into contact with the metal with which it is desired to make an alloy. Two methods of using the electric current for the reduction of aluminium from the

fused flux are described, one in which potentials of from 50 to 100 volts are used, and an arc is formed at the surface of the fused mass, and another, in which potentials of from 2 volts upwards are used, the fused mass being used as an electrolyte. In the first case the arc acts partly by increasing the temperature and partly electrolytically; in the second method an alloy of sodium and aluminium is formed, and falls to the bottom of the crucible as a button.—B. T.

## XII.—FATS, OILS, AND SOAP MANUFACTURE.

*The French Stearin Industry, and this Section of the Paris Exhibition.* B. Lach. Chem. Zeit. **13**, 1157, 1218—1219, 1335—1336, 1374.

THE author, in a series of articles, gives an account of this industry and its exhibits. Great studies have been made in stearin production in France during the last 10 years.

The Exhibition shows this not only to be so with regard to products, but to machinery and apparatus, of which multifarious forms are to be seen, and the sight of which must give an impulse to the industry in other countries. The French, he admits, entirely surpass his own countrymen in this branch. The machinery exhibit of J. Morane, Jeune, alone, which is first described, he estimates as worth about one million francs.

*Saponification.*—The autoclave machines are of two kinds, those for lime saponification and those for saponification by means of high-pressure steam alone.

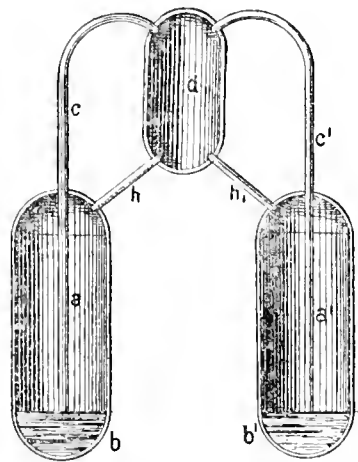
Of the latter, three systems are described.

1. That of G. Levy (the inner construction of which is very complicated) is of sheet copper 30 mm. thick. Opinions of it differ. The work is very good.

2. Thermo-dynamical system of L. Hugues, from the stearin works of L'Etoile, Paris. This was seen in operation by the author. The saponification takes seven to eight hours, and the yield of glycerin, of 28° B., is 9 to 10 per cent. The tallow fatty acids are as clear as palm oil. The fatty acids can be directly distilled with production of 50 per cent. of stearin, m.p. 53°. By careful previous treatment with sulphuric acid, a further yield of 8 to 10 per cent. of stearin can be obtained.

3. A. Michel's apparatus, copper 33 mm. thick. It is brought directly in contact with the fire. See Fig. 1, where

Fig. 1.



*a, a*, are the autoclaves, which being fired, the water *b, b*, is converted into steam, which forces the fatty acids through *c, c*, into *d*, where they are returned, through *h, h*, to *a, a*, and thus kept in intimate mixture.

*Lime saponification.*—

4. System of L. Droux. Spherical, with stirring apparatus.

5. System of J. and B., not exhibited, but seen at work by the writer.

6. That of Morane, Ainc, also with mechanical stirrer.

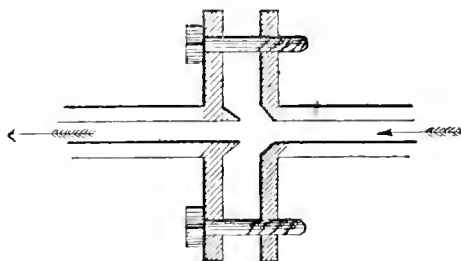
*Glycerin.*—For the treatment of crude glycerin several processes are in use, mostly by use of exhaust steam.

*Distillation.*—Most attention is paid to this form of apparatus in France, which is also shown to advantage by the firm of J. Morane, Jeune.

The condensing apparatus is good, no smell being observable in even the large Paris works, where 8,000 kilos. of fatty acid per 24 hours were distilled. Three forms are described, viz., those of Michl, Lewy, and Marix.

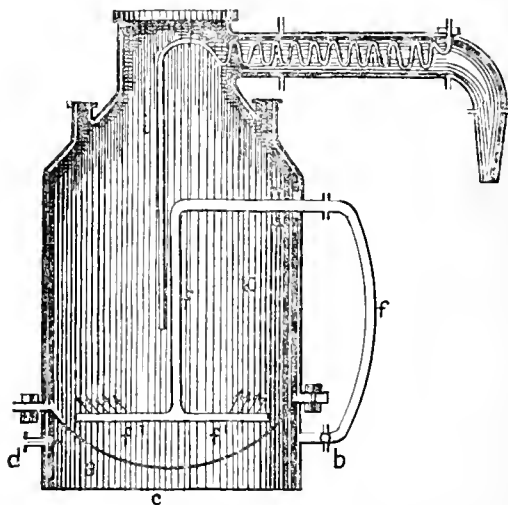
A simple effective flange, Fig. 2, is shown, which on being screwed up forms a perfectly tight joint.

Fig. 2.



Michel's apparatus is of copper. Lewy's is constructed of cast iron. It is shown in Fig. 3; *c* is the fire, at *d* steam

Fig. 3.



is admitted, which after being superheated in *a*, passes through *b*, and thence is distributed by the T-pipe *f*<sup>1</sup>. The fatty acids are heated so far by entering through the spiral *i*.

The author, however, prefers the arrangement of Marix, of the form used in petroleum refineries, in which the distillation takes place at a lower temperature, as low as 250° to 255° where an air-pump is used. Its capacity is 1,500 kilos. and can distil 4,000 to 5,000 per 24 hours. The loss in tar is only  $\frac{1}{4}$  to  $\frac{1}{2}$  per cent. The fatty acids are very white, colourless, and crystalline, they press well, giving 50 to 60 per cent. of homogeneous stearin, melting at 53° to 54°. The principal advantage is the low temperature, 250°—300°. The price, 13,500 francs, is very cheap. It can be worked without the air-pump, and will distil (in still of 1,600 kilos. capacity) 8,000 kilos. per 24 hours. The tar is about 4 per cent., and a good yield of 53° stearin is obtained.

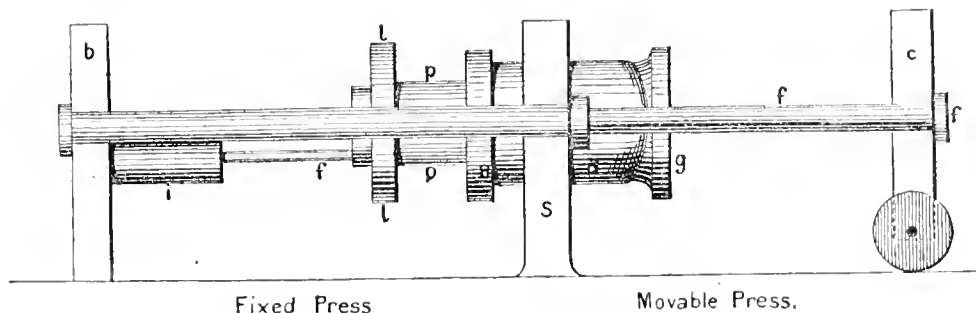
The fatty acids are in thicker cakes than used in Italy, Austria, or Germany. Morane, Jenne, has a simple form of press which is easily manipulated, and requires few fittings and little space.

*Tar.*—Many works distil to thin consistency and treat with English sulphuric acid 66° B. and distil to dryness.

Other works distil to elastic consistency and redistil the last fraction.

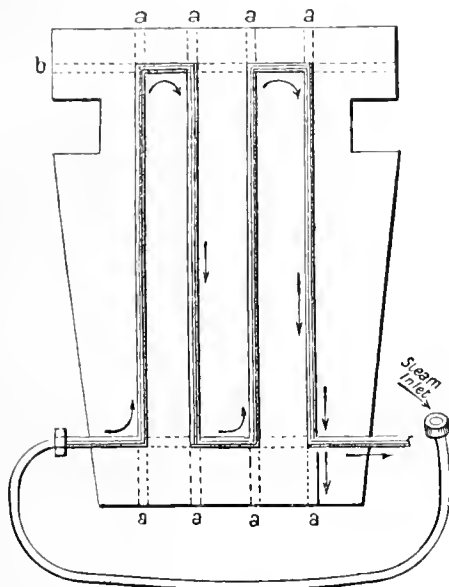
*Pumps and Presses.*—Many varieties of pumps are shown. Of hydraulic presses the newest types of hot or cold are all horizontal. The main feature is that these presses are double. The press cylinder *a*, Fig. 4, is in

Fig. 4.



the middle of the press. In working, the piston carries the moveable wrought-iron plate *l* against the fixed end *b*, while the drawing rod *f* (fixed to both presses) presses the moveable plate *c* against the fixed end *g*. The press cylinder is of cast steel and is supported at *s*. The pistons are of strong brass. The presses are constructed for 30, 50, or more cakes. The heating arrangement, which is good, equable, and exceptionally durable, is shown, Fig. 5.

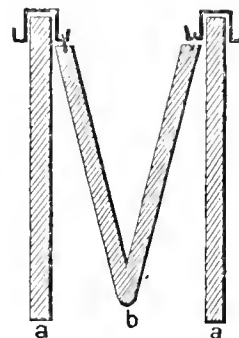
Fig. 5.



Screws are at *a, a*, lead plugs at *b, b*. In Fig. 4 a smaller cylinder *i* will be seen; this is for use in opening of press, which it pushes back when required by turning a three-way valve and opening the return water valve at the large cylinder.

The cold presses are similar. In Fig. 6 is the method of fixing the bags, *a* being plates and *b* bag.

Fig. 6.



The author concludes his notice of the exhibit of Morane, Jenne, with some remarks on an ingenious but complicated triple press, and a few notes as to candle machines.—D. A. S.

*On Wax Investigation.* H. Röttger. Chem. Zeit. **13**, 1375.

G. BUCHNER (Chem. Zeit. **12**, 1276, this Journal, 1888, 871) found with what he termed "absolutely pure" beeswax considerable difference between the acid ether and saponification equivalents of yellow, air-bleached, and chemically-bleached wax. This is not in accordance with results of other investigators such as Helfenberg or Allen, who do not find much difference. The author's experiments confirm these latter results, and he accordingly questions the purity of Buchner's samples.—D. A. S.

*Solidifying Points of Viscous Oils.* Holde. Mitt. König. tech. Versuchs. **1889**, **3**, 119—126.

See under XXIII., page 112.

*Process for the Saponification of Fats.* J. Kaster. Les Corps gras, ind. **1889**, **16**, 148.

THE fat is subjected to the joint action of zinc chloride (4—6 per cent.) and superheated steam (at 200°—220° C.). —B. B.

### XIII.—PAINTS, PIGMENTS, VARNISHES, AND RESINS.

#### PATENTS.

*Improvements in Lacquers and Varnishes.* S. Challows, London. Eng. Pat. 16,035, November 6, 1888. 4d.

CRUDE or refined camphor oil is used in addition to, or instead of, the solvents ordinarily employed.—E. G. C.

*An Improved Apparatus for Dressing White Lead and other Colours.* A. J. Smith, Battersea. Eng. Pat. 16,185, November 8, 1888. 8d.

This apparatus consists essentially of a slightly inclined cylinder, composed of a series of ribs or bars, over which silk or some other textile material is stretched. The ends of the cylinder are left open, so that the stream of colour can be led through the apparatus, the finer particles passing through the textile fabric, while the coarser portions are discharged into a tank.—E. G. C.

*A Cleaning and Polishing Powder for Metals.* J. E. Storer, Sheffield. Eng. Pat. 17,291, November 28, 1888. 4d.

A mixture of lime (140 parts) with rouge (1 part). —E. G. C.

*An Improved Anti-Corrosive Composition for Protecting Iron Vessels or the like.* W. P. Thompson, Liverpool. From P. Leonardi and P. Zen, Venice, Italy. Eng. Pat. 13,345, August 24, 1889. 4d.

The varnish is applied in two coats, the first consisting of a solution of tar in essence of turpentine or benzene, with the addition of 10 per cent. of oxide of iron. The second coat is composed of 10 per cent. of ammoniacal chloride of mercury, 10 per cent. of white arsenic or metallic arsenic, 20–30 per cent. of oxide of iron, “the whole mixed with 100 parts of the above-described solution of tar and turpentine or other varnish.”—E. G. C.

### XIV.—TANNING, LEATHER, GLUE, AND SIZE.

*The Introduction of Hydrogen Peroxide in Tanning.* C. F. Gohring, Gerber Zeit. 1889, 44.

COW-HIDES are often obtained spotted or of too dark a colour. If such leather be washed with a solution of neutral Marseilles soap, and then at once painted with a solution of hydrogen peroxide which has been neutralised with ammonia, and be then dried by gentle warmth or exposure to a current of air, the objectionable colouring matter will be permanently removed without injury to the leather. The author recommends the use of hydrogen peroxide to disinfect hides which have been long stored, and also to preserve extracts of tanning materials.—H. T. P.

*Application of Pine Bark to Tanning.* A. Frank. Ber. des Ver. z. Förder. d. Gewerbe. 1889, 171.

THE author draws attention to the fact that in the manufacture of cellulose from pine wood the bark is entirely wasted. He has established that this refuse has a value for tanning purposes, which would effect an economy of a shilling a hundredweight on the cellulose.

Leather treated with pine bark alone is inferior in quality, colour, and weight, to that prepared with oak bark, but the author quotes Eitner to prove that the extract of pine bark would form a perfect complement to certain imperfect tanning materials such as chestnut wood, quebracho, &c.

—S. B. A. A.

*Benzoyl tannin.* C. Bottinger. Ber. 22, 2706–2709.

By the action of benzoyl chloride upon the aqueous solution of tannin, made strongly alkaline with sodium hydrate (Baumann, Ber. 19, 3218), a benzoyl derivative of characteristic properties is obtained. It is freed from the by-products of the reaction by exhaustive treatment with ether, and is obtained as a semi-crystalline yellowish powder. It softens to a glutinous mass on boiling with water, but does not dissolve; it is not sensibly attacked by water at 165°. It dissolves in sulphuric acid on gently warming. On diluting the solution the original molecule is found to be completely resolved into gallic and benzoic acids. Benzoyl tannin dissolves in phenylhydrazine on warming; the product of the reaction is resolved on treatment with boiling ether into two bodies: (a) crystallising in long colourless needles; (b) bright yellow coloured and amorphous.

The author has commenced a methodical comparison of the benzoyl derivatives of “tannins” from various sources, more particularly in relation to their reactions with phenylhydrazine.

The molecule of quercitanic acid is characterised by the presence of a ketone group, contained in a carbohydrate residue, probably allied to levulose (Ann. 238, 366), and easily split off.—C. F. C.

#### PATENTS.

*Improvements in Machinery or Tumblers for Treating Hides, Skins, and Leather.* P. A. Jesson, Paris, France. Eng. Pat. 17,782, December 5, 1888. 8d.

THE tumblers are formed by taking two hollow pyramids of metal or other suitable material, and arranging them base to base on a common axis. The pyramids may be either equal or unequal in size, and may have any convenient number of sides, and may be attached directly to each other at their bases, or may be separated by a polygonal casing of any desired width. One of the tumblers is hollow, and serves for the introduction of water or other liquid during the rotation of the machine. A door is provided for charging or emptying the tumbler.—B. H.

*Improvements in Extracts applicable for Use in Fermenting and in the Preparation of Leather.* M. P. Hatschek, London. Eng. Pat. 18,399, December 17, 1888. 4d.

THIS invention is an addition to Eng. Pat. 3363 of 1888 (this Journal, 1888, 859). The extract called “Maltopeptine” which forms the subject of the former patent can be used to replace egg-yolk, animal brains or other albuminous matter for fermenting kips, skins, and hides and for dressing them, producing “the same or very similar and frequently better results” than the aforesaid substances. The maltopeptine can be used as a dressing for leather after mixing with oil. The peptonising of the raw material used can also be accomplished by using the juice or tissue of the agave.—B. H.

*Improvements in Machinery or Apparatus for the Treatment of Hides and Skins.* H. E. Freudenberg, Weinheim, Germany. Eng. Pat. 645, January 12, 1889. 8d.

THIS invention is for apparatus for the softening, stretching, fleshing, and cleansing of hides and skins. According to this invention knives for fleshing are dispensed with and replaced by a rotating grinding stone, against which the flesh sides of the hides are pressed under an abundant and continuous water jet. The periphery or face of the grindstone is formed with deep narrow grooves diverging from the centre, which answer the purpose of the angular position of the fleshing knife as held by the workman. Drawings are given, and for further particulars the specification must be consulted.—B. H.



*Improvements in the Process for Freeing Kips, Skins, and Hides from Lime, and for Causing the same to Swell.* J. Hauff, Fuerbach, Germany. Eng. Pat. 12,521, August 7, 1889. 4d.

As cresotinic acid has been found to have too powerful an action on light skins, oxynaphthoic acids (hydroxynaphthoic acids), by which should be understood the carbon-containing acids formed by the action of carbonic acid on the alkaline compounds of  $\alpha$ - and  $\beta$ -naphthols, have been tried and found suitable. It has been found also that mixtures of salicylic acid and cresotinic acid, of salicylic acid and oxynaphthoic acid, of cresotinic acid and oxynaphthoic acid, or of all three, are suitable for heavy skins. It has also been found that a less quantity of these acids is required for the removal of lime from the skins or hides than corresponds to the neutralisation of the lime. This is explained by the property possessed by these acids of forming soluble basic lime salts.—B. II.

*Improvements in and relating to a New and Useful Process of Chrome-Tanning Leather, and in and to the Article produced thereby.* P. Jensen, London. From D. Pierson, Wilmington, Del., U.S.A. Eng. Pat. 13,126, August 20, 1889. 6d.

By this process tanned leather and skins are made permanently soft and flexible without the aid of oils or grease. The "chemical preparation" used is prepared as follows:—Chromic hydrate is prepared by precipitating a solution of chrome alum with carbonate of soda. The chromic hydrate so obtained is dissolved in sulphuric acid of 16°–18° B., and reprecipitated. A vat is provided of suitable size, and of a depth exceeding by 2 ft. the length of the hides, and filled with water. Sulphuric, nitric or other suitable acid is then added till the liquor is of a gravity of 4° B. When cold, excess of the pure chromic hydrate is added, and the solution agitated till all the acid is neutralised. The hides are then suspended vertically in the liquor for from 12 to 18 hours. The hides are then removed and subjected to the action of a warm solution of logwood. The contents of the vat are agitated as before, and are then ready for another lot of hides. This is repeated until the chromic hydrate is entirely consumed.—B. II.

*Improved Means and Process for Preparing Skins and Hides for Tanning.* R. C. Want, London. From G. Mitchell and R. W. Rutherford, both of Dunedin, New Zealand. Eng. Pat. 13,542, August 27, 1889. 4d.

This invention is for the employment of an aqueous solution of sulphate of ammonia, or of sulphate of soda, or of sulphate of magnesium, or of sulphate of potassium for "bating" or "paring" hides preparatory to tanning. The hides are steeped in the solution in the ordinary manner, and are then "scudded" and washed. The strength of the solution is regulated according to the thickness of the hides to be treated, and the time to be allowed, and there is no difference in the treatment of hides and of skins. The solution of sulphate of ammonia is believed to be the best for the process.—B. II.

*An Improved Method of preparing Hides for Tanning.* A. Hull, New Winstead, Conn., U.S.A. Eng. Pat. 14,595, September 17, 1889. 4d.

This invention relates to the "bating" of hides, i.e., their treatment after unhairing and before tanning.

The "bate" is made by dissolving about 4 oz. of crystallised gallic acid in about 10,000 lb. of water. In this solution the hides, after washing to remove as much as possible of the lime, may be immersed for from 10 to 24 hours. The inventor states that after this treatment the hides are soft and flaccid, and by working on the beam the neutralised lime and any extraneous matter will be readily worked out, and the hides rendered in proper condition to be tanned in the usual manner.—B. II.

## XV.—AGRICULTURE, MANURES, Etc.

*Relation between the Colour of Plants and the Richness of the Soil in Fertilising Ingredients.* G. Ville. Compt. Rend. 109, 397–400.

The author's mode of analysing soils by growing the same crop in a number of measured experimental plots arranged as below, has been carried out by him year after year at Vincennes and elsewhere, the following table showing the results obtained at the station named:—

Plot.		Yield of Wheat per Hectare.	
		Total Harvest.	Grain.
1	Complete manure.....	Kilos. 9,579	Hectolitres. 39
2	Same manure without nitrogen ..	4,317	13
3	" " " phosphate.	7,533	24
4	" " " potash ....	7,524	28
5	" " " lime .....	8,290	37
6	No manure .....	3,542	11

These figures plainly indicate that the soil is most deficient in available nitrogen, well provided with lime, not so well with potash, and still less with phosphoric acid. For a number of years the author has observed that the colour of the crops on the different soils furnishes much the same indications as the weighing of the harvest, and, as an example, he gives specimens of the tints, with numerical values attached, afforded by a series of plots of hemp. The observations have been made in three ways: (1) By direct comparison, looking through blackened tubes, of the colour of the crops, with that of skeins of green worsted, selected according to the scale of colours in use at the Gobelin factories. (2) By drying equal weights of the leaves in vacuo, extracting with the same quantity of alcohol, and observing the tint in a colorimeter. (3) The last process is best carried out by first removing the yellow colouring matter (the crystallisable carotene of Arnaud) from the dried leaves by extraction with petroleum ether. The residue from this extraction yields a pure green tint to alcohol, but if the extract in petroleum ether be evaporated in vacuo and taken up with carbon disulphide, it furnishes another term of comparison, since the yellow colour is found to vary in tint, *pari passu*, with the green. The following are the figures attached to the coloured spaces illustrating the paper:—

Manure.	Colour of Plants viewed directly.*		Colorimeter Value of	
	Green.	Yellow.	Green Extract (in Alcohol).	Yellow Extract (in Carbon Disulphide).
Stimulating (100 kilos. N).....	9	15	100	100
Complete (75 kilos. N).....	9	14	74	90
Without nitrogen...	5	7	38	57
Without phosphate.	9	13	71	80
Without potash.....	5	10	66	72
Without lime .....	5	12	72	90
No manure .....	5	11	53	71

\* These numbers correspond with the scale of the Gobelin laboratory.

The author suggests the possibility of determining, by simple colour measurement of the crop and comparison with standard colours, the requirements of the soil as regards manure.—J. M. H. M.

*Fixation of Atmospheric Nitrogen.* Berthelot. *Compt. Rend.* **109**, 417—419.

THE author briefly reviews the controversy, and claims that Ville's early conclusions, as well as his own experiments of some years past, have been completely borne out by the results of Dehérain, Joulie, Hellriegel, Wilfarth, Breal, Frank, and Tacke. Admitting again that Schläsing's negative results as to the fixation of nitrogen by soil alone are due to such causes as the deleterious action of mercury vapour, overheating, or a too confined atmosphere on the microbes essential to the fixation, or else to experimenting on soils already surcharged with nitrogen by the growth of leguminous plants, the author announces a new research on the volatile nitrogenous bodies exhaled by plants and soil into the air.—J. M. H. M.

*Formation of Ammonia and Volatile Nitrogenous Compounds at the Expense of Plants and Vegetable Soil.* Berthelot. *Compt. Rend.* **109**, 419—423.

It has long been known that plants grow badly in a confined atmosphere saturated with aqueous vapour, and this fact was forced on the author's attention in those of his experiments on the fixation of nitrogen by soils and plants in which the pot of soil, or soil and plant, was placed on a large glass dish and covered with a bell jar. Better results were obtained by passing two or three times the volume of air contained in the bell jar through the apparatus once a day, but even then the growth was much inferior to that of plants grown in free air. Thinking it possible that the plants or soil exhaled small quantities of noxious compounds, the author collected with the greatest care the condensed water which ran down the sides of the jar and collected in the glass dish, and maintaining a slightly acid reaction by the cautious addition of sulphuric acid from time to time, submitted the whole quantity to examination at the close of the experiment. No particle of soil was suffered to come into contact with this condensed water, which remained perfectly limpid, and grew no moulds. The ammonia in it was determined by distillation with magnesia, and the organic nitrogen by evaporating down the neutralised and filtered residue and making a soda lime combustion. Two specimens of the results obtained are subjoined:—Pot No. 78: 2·139 kilos. of soil containing 1·702 grms. of nitrogen per kilo.; water 0·214 kilo. Duration of experiment, May 18 to June 13. Capacity of bell jar 50 litres. Nitrogen fixed, 2·8 per cent. of the initial nitrogen. The water of condensation contained ammonia 0·0012 grm., and organic nitrogen 0·0009 grm. Pot No. 76: 3·112 kilos. of the same soil with 0·311 kilo. of water. Sown with vetches. Duration of experiment, May 18 to June 10. Nitrogen fixed by plant and soil = 7·0 per cent of the initial nitrogen. 150 litres of air were passed through the bell jar each day, and one litre of CO<sub>2</sub> added. The water of condensation contained ammoniacal nitrogen 0·0000 grm., organic nitrogen 0·0007 grm. In another experiment, in which the plants grew badly, 0·0015 grm. ammoniacal nitrogen and 0·0012 grm. organic nitrogen were obtained, and in another, where the plants were still worse, and finally became mildewed, 0·0076 grm. ammoniacal nitrogen and 0·0051 of organic nitrogen were obtained. Small though these quantities are, they furnish some support to the idea that poisonous nitrogenous compounds, possibly analogous to the ptomaines exhaled from animals, are given off into the air by plants or soil.—J. M. H. M.

*Nitrification of Ammonia.* T. Schläsing. *Compt. Rend.* **109**, 423—428.

THE experiments described in this paper were made to ascertain whether during the nitrification of ammonia any of its nitrogen is given off in the free state. Soil mixed with a known weight of ammonium salt was introduced into an air-tight apparatus, together with an accurately measured volume of air, and as soon as nitrification set in oxygen

was supplied at intervals; the nitrogen at the end of the experiment was measured and compared with that at the commencement. The figures of one experiment will serve as a type of all. Two hundred grms. of moist earth containing 1·1 mgrms. of ammonia were mixed with 0·539 grm. of ammonium sulphate containing 138·8 mgrms. of ammonia. The ammonia lost by volatilisation from the mixture during the experiment was recovered and measured; it amounted to 1·8 mgrms. Therefore the nett ammonia to be nitrified was 138·1 mgrms. = 113·8 mgrms. of nitrogen. The experiment was started on March 20th, and from the 22nd to the 30th oxygen was supplied at intervals; the experiment ceased on April 9th. During the nitrification 552 mgrms. of oxygen disappeared and the nitrogen increased by 0·65 mgrm. 112·3 mgrms. of ammoniacal nitrogen disappeared and were replaced by 109·7 mgrms. of nitric nitrogen. Thus, of the 2·6 mgrms. of ammoniacal nitrogen which disappeared without being nitrified, only 0·65 mgrm. could have been evolved as free N, even supposing this, and not experimental error, to be the source of the extra 0·65 mgrm. of free nitrogen. Probably the balance went to support the nitrifying organism. Of the oxygen used, nine-tenths were recovered in the nitrate formed, so that in presence of ammonia to be nitrified the nitrifying organism seems to provoke but a very slight oxidation of the carbon and hydrogen of the soil. In this experiment the nitrogen nitrified per day averaged 9 mgrms.; this would give 168 kilos. per hectare of soil weighing 3,000 tonnes, showing how much more favourable the conditions were in the laboratory than in the field.—J. M. H. M.

*The Composition, Treatment and Use of Feces as Manure.* Imperial College of Agriculture and Dendrology, Komaba, Tokyo, Japan. *Bull.* **3**, 1888, 1—25.

LARGE samples of excreta of various origins—peasantry, own population, private houses of the middle class in Japan—were examined, and as the composition in each case is very similar, the mean results are shown in column I. of the following table; column II. shows the composition of excreta of soldiers and students of the nautical college; column III. for comparison, Wolff's numbers for the average composition of European excreta.

The numbers are per 1,000.

	I.	II.	III.	IV.	V.
Water .....	950	944·1	953	963·70	967·7
Organic matter .....	34	40·7	51	14·00	18·6
Ash .....	16	15·2	14	16·3	13·7
Nitrogen .....	5·7	7·96	7	4·20	5·7
Potash .....	2·7	2·07	2·1	2·84	1·37
Soda .....	4·6	3·61	3·9	5·57	5·23
Lime .....	0·2	0·20	0·9	0·03	0·04
Magnesia .....	0·5	0·51	0·6	0·02	Trace.
Iron and alumina .....	0·3	0·61	..	Trace.	0·04
Phosphoric acid .....	1·3	2·97	2·6	0·55	0·44
Sulphuric acid .....	0·5	0·72	0·5	0·77	0·90
Silica .....	0·5	0·37	0·2	0·12	0·07
Chlorine .....	6·2	5·08	4·0	7·88	6·93
Sodium chloride .....	10·2	8·37	6·6	12·93	11·42

THE vegetarian diet of the ordinary Japanese people accounts for the smaller proportion of nitrogen and phosphoric acid and the larger proportion of potash and sodium chloride these excreta contain as compared with European excreta or the excreta of the Japanese soldiers and nautical students, who have a moderate allowance of meat.

COLUMNS IV. and V. in the table show the composition of Japanese urine, IV. of the peasantry, V. of townspeople, which in both cases are more dilute than European urine.

For manure the night-soil is mixed with two or three times its volume of water and left for about ten days in cold weather and five or six days in warm weather to ferment before being applied to the soil. It undergoes decomposition with the evolution of carbonic anhydride and marsh gas, and the

production of large quantities of ammonium carbonate. The Japanese store it in large glazed earthenware vessels. Experiments were made to ascertain whether much nitrogen is lost by volatilisation of ammonium carbonate, with the following results:—

Date of Commencement of Experiment.	Original Percentage of N in the Fæces.	Percentage of original Nitrogen lost, after					
		One Week.	Two Weeks.	Three Weeks.	Five Weeks.	Seven Weeks.	Eleven Weeks.
February 20.....	7.90	2.58	..	5.98	..	..	..
April 24.....	7.97	..	4.63	6.32	10.57	13.02	20.17
July 6.....	7.22	..	5.09	..	12.70	..	..

The loss is so small as not to need precautions for preventing it, when the usual Japanese mode of storage is used. The night-soil is poor in organic matter in proportion to nitrogen, and therefore is made into a compost with vegetable refuse for application to porous soils poor in humus. On the other hand they are comparatively rich in soluble ammonium salts, and so are applied in moderate quantities, frequently, on the non-retentive Japanese soils. Various calculations are given as to the probable produce from a given quantity of night-soil, and it is shown that it is not a complete manure, for, compared with the proportion of nitrogen, both potash and phosphoric acid are deficient, part of the latter moreover being insoluble.—D. A. L.

**Fish Manure.** O. Kellner, Imperial College of Agriculture and Dendrology, Komaba, Tokyo, Japan. Bull. 4, 1889, 5—6; 29.

Waste fish are used as manure in Japan and are prepared either by simply drying in the air or the fish are first boiled in water to obtain fish oil and are then pressed and dried, the second method giving the best manure. The fishes most commonly employed are the herring (*Clupea harengus*) and sardine (*Clupea melanosticta*; *gracilis*) and the following table gives numbers taken from the tabulated results of seven analyses of sardine and three analyses of herring manure:—

—	Percentages in			
	Sardine Manure.		Herring Manure.	
	Highest.	Lowest.	Highest.	Lowest.
Moisture.....	16.36	5.73	17.91	9.43
Organic matter.....	82.94	64.85	74.94	61.45
Ash.....	29.42	10.94	20.64	15.63
Sand.....	11.70	8.04	8.60	6.55
Nitrogen.....	18.16	3.89	17.65	12.18
Potash.....	0.76	0.16	0.88	0.60
S. da.....	0.97	0.33	2.11	0.46
Lime.....	4.61	2.12	5.99	2.56
Magnesia.....	0.76	0.34	0.84	0.67
Iron oxide.....	1.53	0.02	1.99	0.24
Phosphoric acid.....	4.85	2.86	5.96	2.27
Sulphuric acid.....	0.70	0.02	0.34	0.00
Silica and sand.....	10.87	0.79	9.63	2.12
Chlorine.....	0.53	0.22	2.11	0.86

Next to these the following fish are frequently found in Japanese fishmanure:—*Scomber pneumatophorus japonicus*, *Chatoeius punctatus*, *Trachurus trachurus*, *Trichodon stelleri*, *Ammodytis*, *Hypoglossus vulgaris*. Recently the heads, bones, and tails of large fish (*Thynnus sibi*; *pelamys*) coarsely crushed have been employed as manure. Tunny fish manure, from head, bones, fins, and intestines has following percentage composition in two analyses:—Water, 7.23—9.47; ash, 2—28.7; fat and oil, 11.33—14.56; nitrogen, 8.16—5.34; phosphoric acid, 4.25—7.42; lime, 5.14—?; from flesh portions H<sub>2</sub>O, 8.23—12.63; ash, 2—6.16; fat and oil, 18.01—15.22; N, 13.01—10.50; P<sub>2</sub>O<sub>5</sub>, 0.94—2.38; lime, 0.65—?.

Kitchen waste from nine different kinds of Japanese food fish from the sea, dried and finely pulverised, forms a manure of the following composition per cent.:—Moisture, 7.85; organic matter, 63.86; ash, 28.29; N, 6.15; P<sub>2</sub>O<sub>5</sub>, 7.64; CaO, 10.45; MgO, 0.22. On the Japanese coast various sea fish are used for manure, such as small crabs, prawns, and star fish. The following numbers are from analyses of star fish and of a sample taken from a shipload of seaweed somewhat decomposed:—

—	Star Fish.	Seaweed.
Water.....	32.68	50.75
Organic matter.....	20.87	14.21
Ash.....	43.37	..
Sand.....	3.08	21.94
Nitrogen.....	1.41	0.35
Potash.....	0.98	1.63
Soda.....	1.70	3.11
Lime.....	21.90	0.73
Magnesia.....	0.45	0.04
Iron oxide.....	0.72	0.05
Phosphoric acid.....	0.61	0.12
Sulphuric acid.....	0.08	0.09
Silica.....	..	1.29
Chlorine.....	0.13	..
Carbonic acid.....	17.30	..
Total salt.....	..	8.41
Adhering salt.....	..	3.36
Other mineral matters.....	..	9.74

—D. A. L.

*Manurial Experiments with Felspar, Potassium Salts, and Thomas-Slag.* L. F. Nilson. *Tidskrift för landtman*, 1889, 273—281.

NUMEROUS experiments were tried to ascertain the manurial value of felspar as coarse powder, finely pulverised, and as mud; but no decided results were obtained from it with oats on a sandy or moorland soil, although in one instance a small increase is attributable to felspar mud. It is, however, certain that the potash in the felspar, although not immediately active, will become so as the mineral decomposes, which should proceed rapidly in the moorland. This soil contained 18·1 per cent. of mineral matter, 5·3 per cent. of which consisted of lime, 0·15 per cent. of phosphoric acid, 1·37 per cent. of sulphuric acid, 0·6 per cent. of potash; and 81·9 per cent. of organic matter, with 3·7 per cent. of nitrogen; dilute hydrochloric acid extracted from the natural soil, 0·3 per cent. of phosphoric acid, 0·11 per cent. of sulphuric acid, 5·5 per cent. of lime, and 0·03 per cent. of potash. But in spite of this abundance of plant food, owing to it being non-available, good results were obtained by manuring with Thomas-slag, potash (as sulphate), and Chili saltpetre, especially when all were employed together.—D. A. L.

*Investigation of Forest Soil.* E. Ramann. *Wollny's Forschungen a. d. Gebiete d. Agrikulturphysik*, 11, 299—337.

NUMEROUS researches have led to the following conclusions:—1. The specific gravity of the natural soil depends on the amount of humus, the crumbly character, and on the denseness of the layers. It is lowest at the surface on account of the humus, and then increases gradually with the depth. 2 and 3. The crumbling is due to chemical and physical causes, and not to earth-worms, their number being too few. 4. The amount of water in the natural soil is, in fine to medium grained sand, about 3—4 per cent. by weight, corresponding to 4—5 per cent. by volume in the upper layers, and 5—6 in the lower. 5, 6, 7. The distribution of water is the same as in any other soils; plentiful in the humus portions, less in the crumbled portions, and abundant in the denser layers; capillarity acting as usual more strongly in the dense than in the crumbled layers. 8. Impervious layers of clay or very fine-grained sand cause only temporary storage of water after a rainfall, when drainage is possible. 9. The quantity of water retained by the soil is very considerable; a layer of fine to medium grained sand from 7 to 8 metres thick retains as much water as the total annual rainfall yields. 10. The proportion of water falls rapidly in the spring, then slowly and probably reaches a minimum in the autumn. 11. The effect of altitude on the proportion of water is only observable where knolls and small ridges are met with; in a gentle slope the effect of position on the proportion of water is not noticed.

The soil investigated is fine to medium grained sand soil of the northern diluvium, situated on the road from Eberswald to Spechthausen.—D. A. L.

*Sea-Mud and its absorbing Capacity for Lime and Potash.* A. Müller. *Landw. Versuchsst.* 34, 257.

A MOOR in South Sweden consists partly of exposed sea-soil, which is covered all over by an extremely fine mud; this when wet is almost black, but when dry is dark grey, and is characterised by a large quantity of siliceous infusoria. The dry mud contains 0·737 per cent. of nitrogen, corresponding to 3·6 per cent. of organic matter, and 100 parts of ash contain:—20·51 of  $\text{Fe}_2\text{O}_3$  and  $\text{Al}_2\text{O}_3$ ; 0·48 of  $\text{P}_2\text{O}_5$ ; 0·38 of  $\text{CaO}$ ; 0·27 of  $\text{MgO}$ ; 0·25 of  $\text{K}_2\text{O}$  with trace of  $\text{Na}_2\text{O}$ ; and 78·11 parts insoluble in hydrochloric acid, consisting of 59·58 per cent. of soluble silica and 40·42 of quartz and silicate sand. When the air-dried mud is shaken and digested in the cold with lime water or potash, it absorbs 9·95 per cent. of lime or 1·47 per cent. of potash. It would also have great absorbing power for alkaline phosphates or superphosphates, a valuable property considered agriculturally.—D. A. L.

*The Composition and Preparation of Peat Manure.* P. Kulisch. *Chem. Zeit.* 13, 1429—1430.

For several years this material has been extensively used in the vineyards of the Royal School of Fruit and Vine Culture, at Geisenheim. From the results obtained, it seems that the growth of the wood, and, therefore, the fruitfulness, in old and weak vine-stocks is favourably influenced. Experiments were made, during a period of 20 days (in March), to determine the amount of manure obtainable from known quantities of peat. The peat used had the following composition:—

	Per Cent.
Dry matter.....	77·0
Moisture.....	23·0
Ash.....	2·11
Nitrogen.....	0·443
Phosphoric acid.....	0·043
Potash.....	0·017

For each cow (two milch cows) the average amount of peat used was 5·99 kilos. daily, and the average weight of manure obtained daily from each animal was 44·75 kilos. The manure gave the following results on analysis:—

	Per Cent.
Dry matter.....	18·84
Nitrogen.....	0·304
Phosphoric acid.....	0·124
Potash.....	0·565

The analyses of a slaughter-house manure and of a sample of woollen dust to be used for manurial purposes, are also given. The slaughter-house manure consisted chiefly of peat mixed with straw, solid animal excreta, blood, bone-splinters, hair, fragments of horn and flesh, &c. It contained—

	Per Cent.
Dry matter.....	20·59
Nitrogen.....	0·79
Potash.....	0·107
Phosphoric acid.....	0·185

The woollen dust contained—

	Per Cent.
Moisture.....	8·34
Ash.....	6·74
Nitrogen.....	2·95

—H. T. P.

## XVI.—SUGAR, STARCH, GUM, Etc.

*Crystallised Grape Sugar.* E. O. von Lippmann. *Chem. Zeit.* 13, 1474.

THE author has already described and shown (this Journal, 1888, 579) that the products of a dextrose factory working the Cords-Virneisel process are of greater purity than those usually obtained in German factories. Recently he has received from the same factory samples of dextrose low products, consisting of transparent hard reddish pieces formed of numerous more or less well built crystals closely grown together. Analysis gave—water, 4·75 per cent.; grape sugar, 91·84 per cent. (by polarisation) and 92·2 per cent. (by Fehling's solution); ash, 0·219 per cent.; etc. (non-sugar) 3·191 per cent.

The fact that Fehling's solution showed more grape sugar than the polariscope points to the absence of dextrine which is generally an impurity in commercial grape sugar. This absence of dextrine the author has already observed to be characteristic of sugars made by the Cords-Virneisel process.

The sugar consisted of glucose anhydride, and could be heated almost to 140° C. without melting or undergoing alteration.—W. M.

*Record of Experiments at Des Lignes Sugar Experiment Station, Baldwin, La., during the Season of 1888.* C. A. Crampton, U.S.A. Department of Agriculture. Bulletin, 22, 1889.

THE experiments conducted by the author commenced on November 5, 1888, and extended over four weeks or just one-half of the manufacturing season. The proprietors of the factory and of the plantation are Messrs. Shattuck and Hoffmann.

The extraction was performed by a six-roller mill, composed of two three-roller mills set tandem. Each mill was

driven by a separate engine. The second mill was provided with a hydraulic regulator. Maceration was practised all through the season, the water being added from a rose placed above the intermediate carrier. The juice was sulphured and clarified in the usual way and evaporated in a double-effect Rillieux pan. It had horizontal tubes of about 1,225 sq. ft. heating surface. Two 7½ ft. strike pans were used in reducing the syrup to massecuite.

The manufacturing data for the last four weeks of the season, while under chemical control, are given in full detail in the following table:—

	Cane Ground.	Sulphured Juice.	Weight per Gallon.	Weight of Juice.	Sugar in Sulphured Juice.	
	Tons.	Galls.	Lb.	Lb.	Per Cent.	Lb.
Fifth week.....	1,217	292,782	8.86	1,796,650	12.15	224,683
Sixth week .....	1,117	169,822	8.86	1,504,623	12.83	193,043
Seventh week.....	1,392	233,842	8.85	2,069,501	12.72	263,240
Eighth week.....	1,139	191,084	8.84	1,689,183	13.00	219,594
For four weeks .....	4,895	797,530	..	7,059,957	..	900,560

	Sugar per Ton of Cane, obtained in Sulphured Juice.	Sugar in per Cent. of Weight of Cane obtained in Sulphured Juice.	Merchantable Sugar Made.	Merchantable Sugar per Ton of Cane.	Molasses Obtained.	Molasses per Ton of Cane.
	Lb.		Lb.	Lb.	Lb.	Lb.
Fifth week.....	180.2	9.01	182,621	146.4	64,522	51.7
Sixth week .....	172.8	8.64	158,905	142.3	56,523	50.6
Seventh week.....	189.1	9.45	204,820	117.1	105,157	75.8
Eighth week.....	192.8	9.53	189,750	166.6	68,838	60.4
For four weeks.....	184.0	9.20	736,076	150.4	295,240	60.3

The total loss in manufacture during the four weeks was 72,361 lb. of cane sugar (of 100 per cent.) or 8.03 per cent. of the amount of sugar present in the sulphured juice which was taken as the starting point. Of this 8.03 per cent. of total loss, 3.37 per cent. is charged to inversion and the remaining 4.66 per cent. is grouped under the head of mechanical losses.

Experiments made on acid and neutral clarification showed that whilst with the former the colour is better, yet the loss from inversion is decidedly greater with acid than with neutral clarification. The practice of boiling and skimming in an open pan, with either neutral or acid syrup, is strongly condemned, and wonder is expressed that planters should adhere to this practice.

The amount of coal used during the season was 555 tons. The fuel consumption, exclusive of bagasse, is given as follows:—

Pounds of coal per 1,000 lb. sugar .... 946

Pounds of coal per ton of cane..... 128

The author made a few observations and experiments on the process of maceration by means of the addition of hot

water to the canes, or to the bagasse between the two mills. The amount of water thus added at Des Lignes was not subject to any careful regulation, the general idea being to give to the bagasse all the water it would hold. The author concludes "there is no doubt whatever that double milling with maceration could be made as superior to the old method of single milling in cane work as the continuous presses with maceration have been shown to be superior to the method of single pressure in a hydraulic press with beets," but he continues, "it seems hardly necessary to go into the refinements of the method of maceration in cane work, in view of the fact that improvements in this line have been abandoned for the better method of diffusion."

Some interesting experiments were made showing the improvement in cane by standing and ripening. The mill was set in operation too early in the season. The crop was over-estimated, and it was feared that if the campaign were not opened earlier the mill would not be able to handle it before freezing weather set in. The following tables give the analysis of juice from cane on four different cuts at two periods in each case:—

Date of Analysis.	Solids.	Cane Sugar.	Glucose.	Coefficient of Purity.	Available Sugar in Juice.	Pounds Per Ton Available Sugar at 70 Per Cent. Extraction.	Difference.
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#### STORE CUT.

	Per Cent.	Per Cent.	Per Cent.		Per Cent.	Lb.	Lb.
November 14.....	15.46	13.34	1.09	86.3	11.74	163.8	..
November 30.....	15.83	13.99	0.88	88.4	12.67	177.4	13.6

Average improvement per day in pounds per ton, 0.85.

Date of Analysis.	Solids.	Cane Sugar.	Glucose.	Coefficient of Purity.	Available Sugar in Juice.	Pounds Per Ton Available Sugar at 70 Per Cent. Extraction.	Difference.
BOYLE CUT.							
November 17 .....	15.87	13.82	0.94	87.1	12.41	173.7	..
November 30 .....	16.47	14.88	0.66	90.3	13.89	194.5	20.8

Average improvement in pounds per day per ton, 1.6.

CLARA CUT.							
November 19 .....	15.94	13.80	1.01	86.6	12.28	171.9	..
November 30 .....	16.23	14.75	0.53	90.9	13.95	195.3	23.4

Average improvement per day in pounds per ton, 2.13.

SILLAN CUT.							
November 21 .....	14.80	12.97	0.96	87.6	11.53	161.4	..
November 30 .....	15.77	13.80	0.81	87.9	12.63	176.8	15.4

Average improvement per day in pounds per ton, 1.71.

" " " " " " for all four cuts, 1.56.

From the above tables will be seen the improvement in each case. Taking the average increase of the four cuts at 1.56 lb. of available sugar per ton for each day the cane was allowed to stand, a crop of 10,000 tons would improve at the rate of 15,600 lb. of pure sucrose, equal to say 16,500 lb. per day of merchantable sugar, so that two weeks would add nearly one quarter for a million pounds to the amount of sugar actually obtainable from it. Next season Messrs. Shattuck and Hoffmann will adopt the diffusion process on the Des Lignes plantation.—A. J. K.

*Record of Experiments at the Sugar Experiment Station on Calumet Plantation, Pattersonville, La., 1888—1889.*  
H. Edson, U.S. Department of Agriculture. Bulletin, 23, 1889.

This report gives a very complete and detailed account of the season's work at the above sugar house. The machinery and appliances were all of the best, and the general arrangements admitted of very careful working. The unprecedented (for Louisiana) milling yield of 200 lb. of sugar per ton of cane was obtained. There were two mills employed, the raw juices from which passed to a vessel provided with a paddle, where they were saturated with sulphurous acid gas. The fumes produced by burning sulphur were first cooled and then passed over the surface of water to free them from any trace of sulphuric acid. The juice was then pumped to defecators and lime added. After settling the clear liquor went direct to the double-effect. The skimmings and settlings were re-heated and limed when necessary, and filter-pressed, hard cakes and clear liquors being always obtained. For convenience the season's work is divided into five runs, two on stubble and three on plant cane. In the first run the most noticeable thing was the boiling of molasses containing 33.2 per cent. sucrose and 35.74 per cent. glucose for sugar, and getting a massecuite which grained well and gave 12.06 lb. of commercial sugar per ton of cane.

In the second run the sulphur machine was unfortunately somewhat neglected, and some sulphuric acid was probably produced, as inversion took place to the extent of 1.32 per cent. of the sucrose in the raw juice. The final molasses contained 23.7 per cent. sucrose and 32.6 per cent. glucose. This run was also noticeable for the point to which crystallisation was carried, the glucose apparently not possessing the restraining power commonly attributed to it.

The third or first plant run was the largest, and the cane was the richest. "Maceration, or the addition of water to

the bagasse between the two mills," was commenced in this run and greatly increased the yield of sugar, a gain of 17 lb. of sugar per ton of cane being obtained by the addition of 12 per cent. of water. The water was sprinkled on the cane as it was released from the pressure of the first mill, it being argued that during the expansion following this pressure the bagasse would more thoroughly and uniformly absorb the water than at any subsequent period, a minimum of water thus producing the maximum effect. It is pointed out that although this is a great advantage over ordinary milling, diffusion is clearly the most economical method for the extraction of sugar from the cane. The fourth and fifth runs were not specially remarkable.

In regard to the use of sulphurous acid, the author is very favourably impressed with it as an aid in improving the quality of the output of a sugar house, and although in careless hands it can cause serious loss by inversion, he thinks that when carefully and intelligently applied its use can be unhesitatingly recommended. Experiments were also made leaving the juice slightly acid (with sulphurous acid), and also carefully neutralised. In both cases there was practically no inversion, but the colour of the sugar from the acid liquor was much better than that from the neutral.

Numerous tables are given showing in a most complete manner the working of the sugar house during the season.

—W. M.

#### *Separation of Raffinose from Cane Sugar.* J. fabr. Sucre, 30, 34.

ACCORDING to LOPÈS, ammoniacal acetate of lead precipitates cane sugar, but not raffinose. The possibility of the separation of these two sugars is thus indicated. The author is continuing his experiments on the subject.—H. T. P.

#### *Some Properties of Raffinose.* Beythien and Tollens. Zeits. f. Rübenzucker. Ind. 39, 911, 913, 917.

By the gentle inversion of raffinose (10 grms. of raffinose + 90 cc. of water + 6 cc. of hydrochloric acid of 1.19 sp. gr., heated for 10 minutes to 68° C.) and subsequent treatment of the inversion products with phenylhydrazine, an osazone is obtained whose melting point is identical with that of gluco- or levulosazone. The corresponding sugar is levo-rotatory and is therefore probably levulose. Galactose cannot be directly detected in the inversion products, being, as was pointed out by Scheibler (see this Journal, 1889, 718), still combined with the dextrose. The polarisation of inverted

raffinose is in mean  $+50.94^\circ$ , for  $+100^\circ$  original rotation. ee. of Fehling's solution is reduced by 7.7 mgrm. of inverted raffinose (= 6.53 mgrm. anhydrous raffinose). By the protracted boiling (three days) of raffinose with a solution of strontium hydrate, lactic acid is formed. Under similar circumstances cane sugar also yields lactic acid; but no trace of raffinose is formed, so that Pellet's former assertion on this subject is quite erroneous. Lactic acid occurs in molasses to the extent of about  $\frac{1}{2}$  per cent., and is probably formed by the decomposition of cane sugar during manufacture.—H. T. P.

*The Non-fermentable Dextro-Rotatory Constituent of Honey.* E. von Raumer. Zeits. f. angew. Chem. 1889, 607—609.

Pure honey contains a residual substance after complete fermentation by yeast, which has a decided dextro-rotatory action. This body can be isolated by evaporating the filtrate from the fermented honey, after treatment with aluminium hydrate and repeatedly precipitating by alcohol. It forms a white amorphous mass, having a specific rotatory power  $\alpha_D = 59.3^\circ - 68.06^\circ$  in two preparations (from different sources) examined and reduces Fehling's solution, the amount of reduction before and after inversion having a ratio 1 : 2.62 and 1 : 2.79. The ratio of the rotatory power of the body before and after inversion was found to be 1 : 0.514 and 1 : 0.45. Great care was taken to ensure the purity of the honey examined and to make quite sure that it was free from starch-sugar syrup. Since then this dextrin-like body is contained in pure honey, Sieben's method for detecting starch sugar syrup in honey by the fact that it leaves a dextro-rotatory residue after fermentation (this Journal, 1885, 411) is without value. The exact nature of the non-fermentable constituent of pure honey is still under investigation; it appears to be completely fermentable after inversion and its rotatory power decreases gradually as the inversion proceeds, but at no time does it show any levo-rotatory action, whence it is improbable that levulose results in the inversion.—C. A. K.

## XVII.—BREWING, WINES, SPIRITS, Etc.

*Mealy and Glassy Barley.* L. Just and H. Heine. Landw. Versuchst. 36, 269.

For the examination of barleys the authors employed Rimpau's method, in which the corns are examined by transmitted light; the mealy corns appear dark, and the glassy ones transparent. Seven barleys of German and Swedish origin were examined. The purely mealy and glassy corns of each barley were separated by the above method, and separately examined. The results obtained were as follows:—

1. Absolute weight:—The mean weight of the glassy corns was less than that of the mealy corns.
2. The volume of the glassy corns is smaller than that of the mealy ones, but their specific gravity is greater than that of the mealy corns from the same barley.
3. The moisture percentage is the same for mealy and glassy corns of the same barley.
4. The amount of ash is somewhat smaller in the glassy than in the mealy corns. Weight for weight, however, the glassy corns in most cases contain a higher amount of ash.
5. The glassy corns contain more nitrogenous compounds than the mealy corns of the same barley. Weight for weight, however, glassy barley may contain less nitrogen than mealy barley from the same sample.
6. The germinating energy is all through less in glassy than in mealy corns, but the total number of corns capable of germination is a trifle larger for glassy barley.

According to the authors, no direct connexion exists between the degrees of mealiness of various barleys and

their weights, and the percentages of ash, and especially of nitrogenous bodies, contained in them. The same may be said of the germinating energy and the percentage of corns capable of germination.—H. T. P.

*Studies on Diastase.* C. J. Lintner and F. Eckhardt. Zeits. f. d. ges. Brauw. 12, 389.

As the result of their experiments the authors find that the ferment of ungerminated grain (in this case, barley and wheat) differs considerably from the diastase of malt. At temperatures above  $35^\circ\text{C}$ , the former has a weaker fermentative action than the latter; at temperatures below  $35^\circ\text{C}$  the reverse holds good. The observation of Reyehler that a ferment is formed by the action of dilute acetic acid, &c., on wheat flour or gluten (unmedin) is confirmed by the authors. This formation of a ferment by the action of acids must not be confounded with the formation of diastase during germination. The action of Reyehler's (see this Journal, 1889, 407) ferment solutions is similar to that of wheat and barley extracts. To prevent mistakes, the authors propose that the name "diastase" be applied to malt diastase alone, and that the ferment of ungerminated grain be termed barley diastase, wheat diastase, &c.—H. T. P.

*The Colouring-Matter of Red Wines.* R. Heise. Chem. Zeit. Rep. 13, 283.

In the separation of the colouring matters of wine, two methods are chiefly employed: precipitation by means of basic acetate of lead, and direct extraction of the colouring matter. The author has employed the first of these methods. In Mulder's method the lead precipitate is decomposed by means of sulphuretted hydrogen, and the lead sulphide is extracted with acetic acid and alcohol. The author finds that the colouring matter thus obtained always contains lead. Glénard decomposes the lead precipitate with an ethereal solution of hydrochloric acid gas. Two different colouring matters are obtained in this manner, A and B. A is insoluble in absolute alcohol, B is soluble. The lead precipitate from wine is partially soluble, partially insoluble in glacial acetic acid. The insoluble portion corresponds to the colouring matter A above described, the soluble portion to B. A directly prepared extract of fresh ripe grape skins contains a colouring matter identical with B. Gautier's statement that grape skins contain colourless, readily oxidisable tannins, could not be confirmed. According to Nessler, not only the alcohol formed during fermentation, but especially the acid and the temperature have great influence on the solubility of the colouring matter. The results obtained by the author rather tend to show that the acid has not so much an effect on the solubility as on the intensity of the colouring matter. The tanning matter of wine, oenotannin, consists, according to the author, of three substances, gallotannic acid, quercetin, and an unknown body.

Grapes and red wine contain the same red colouring matter B. The lead precipitates contain in addition considerable quantities of A. The latter is chiefly contained in the lees of wine, and may be obtained from B by long boiling with hydrochloric acid. A can exist in the wine in solution only in very small quantity. It is probably formed during the treatment of the wine or grape extract with basic lead acetate. Possibly A is an oxidation product of B, its formation being favourably influenced by the alkalinity of the precipitant. The variations which occur in the reactions of wines are partly due to the colouring matter B, which is easily affected by external conditions, and partly to the amount of other substances which accompany the colouring matter. The ratio of these bodies to the colouring matters is continually changing as the precipitation of the colouring matter of the wine goes on.—H. T. P.



## XVIII.—CHEMISTRY OF FOODS, SANITARY CHEMISTRY, AND DISINFECTANTS.

### (A).—CHEMISTRY OF FOOD.

*The Chemistry of the Manufacture of Preserves.* W. Reuss. Chem Zeit. 13, 1428—1429.

The moiré-like film which is often found on the interior surface of preserve tins has been ascribed by Beckurts to the formation of sulphide of tin. He proved this in two ways:—

(1.) The film was dissolved in ammonium sulphide, and the tin precipitated as yellow tin sulphide by the addition of excess of sulphuric acid.

(2.) The film was removed by friction with cotton wool, which was subsequently treated with hydrochloric acid. The sulphuretted hydrogen evolved was detected by the yellow colour it imparted to a drop of silver nitrate solution spread on filter paper.

The author finds these reactions to be worthless for the purpose in view, since they are both yielded by perfectly clean, untarnished tin-plate. Even chemically pure tin yielded the second of the above reactions. This is no doubt due to the presence in the tin of traces of arsenic, antimony, or phosphorus, which yield hydrides capable of discolouring silver nitrate. The author proposes the following test:—Pure dry sodium carbonate is dusted on the surface of the tin-plate and well rubbed in with cotton wool. The sodium carbonate is afterwards fused with potassium nitrate, and the solution of the fused mass tested for sulphuric acid with barium chloride. The formation of moiré films is very common in the case of all vegetable preserves. This is no doubt due to the more or less acid nature of all plant juices. During sterilisation and subsequent storage, the organic acids act on the surface of the tin, forming the moiré film, in the same way that this appearance is produced for certain purposes, by means of mineral acids. In most cases, therefore, the dark film consists of minute tin crystals. In a few instances the author was able to detect sulphur in the films.—H. T. P.

*The Corrosion of Nickel-cooking Vessels by Organic Acids.* A. Rohde. Arch. f. Hygiene, 9, 331.

In all former researches on this subject the chemical corrodibility of nickel has been practically neglected. In his experiments the author used acid solutions of known strength. It appears to be not improbable that considerable quantities of nickel may pass into solution during the preparation of, especially, very acid foods in nickel utensils, and theoretically at least, the regular introduction of nickel into the human system might be attended by serious consequences. Taking into account his own observations, as well as the physiological experiments with nickel salts of Geerkens, Laborde, and Riche, the author is, however, of opinion that nickel cooking utensils may be employed with perfect safety, provided they are obtained from a reliable source.—H. T. P.

*Experiments on Blue Milk.* L. Heim. Arbeiten a. d. kaiserl. Gesundheitsamte, 5, 518.

MILK on standing shows small blue spots, which become gradually larger, and finally cover the whole surface, the milk turning at the same time sour. The phenomenon is due to bacteria, which have been isolated by Löffler. The present author has examined such bacteria more minutely. He finds them to consist of short rods with rounded ends. Formation of spores he was never able to observe, and subsequent experiments show spore formation to be improbable. By means of gelatin, &c. the bacteria lose their property of colour formation, and can then no longer render milk blue. At a temperature of 55° C. they die in ten minutes, at 75° C. in five minutes, and at 80° C. in one minute. This fact is opposed to the formation of spores. At 30° C. the growth was much weaker, and at 40° C. the bacteria died in a comparatively short time. In a 3 per

cent. solution of sodium carbonate they died in three hours, in a 10 per cent. solution only five minutes were necessary, in salicylic acid (1 to 300) 30 minutes, and in 5 per cent. caustic soda solution 45 seconds. In 4 per cent. boric acid solution they were in good condition, even after five days. The results of these experiments speak also against the formation of spores. Of course, if the experiments be made in the milk a much longer time is necessary to bring about the above results.—T. L. B.

### PATENTS.

*Improvements in Farinaceous Food for Animals.* A. Brunier, Marseilles, France. Eng. Pat. 19,003, December 29, 1888. 4d.

MEAL from the nigella or fennel flower and from maize husks is made into a mixture with meal obtained from other kinds of grain, and with any suitable oilcake. As an example, a mixture is made of 40 parts of nigella meal and 10 parts each of maize husk meal, bean meal, barley meal, oilcake, decorticated earthen meal, and earthen husks.—J. M. H. M.

*Improvements in and relating to the Treating and Preserving of Fish.* W. G. Pursell, Leith. Eng. Pat. 1033, January 19, 1889. 4d.

AFTER the fish have been cleaned, they are first smoked and then treated with boric acid or borax, or some compound containing one of these substances.—E. E. B.

*An Improved Process of and Means for the Preservation of Milk.* F. McIntyre, Glasgow. Eng. Pat. 1423, January 25, 1889. 6d.

THE cows are milked into a sterilised receiving vessel, precautions being taken to prevent contact with atmospheric germs. The teats between the milkings are covered with an elastic material or with an antiseptic solution, such as potassium permanganate or sodium silicofluoride, alone or with vaselin; and before milking, the teats are washed with the same solution. The milk is drawn from the teats by the insertion of sterilised metallic trocars, the orifices at their ends being capped with india-rubber. The trocars are inserted into the teats after removing the caps, and by means of india-rubber tubes on the other ends the milk, obtained by hand-milking in the ordinary way, is led into a receiver previously sterilised by steam or other heat, and having an exit tube for the air fitted with cotton wool or other filtering material, and coated internally with glycerin or other sticky liquid; or the entrance of germ-laden air may be prevented by interposing a sterilising liquid, or by other means.—J. M. H. M.

*Improvements in Apparatus for Assimilating Cow's Milk to Human Mother's Milk.* R. W. Seathing, Birmingham. Eng. Pat. 6318, April 13, 1889. 8d.

THE milk is boiled in a vessel constructed so that the milk is kept whilst boiling in a state of circulation. The vessel is heated by means of a spirit-lamp, and to prevent the milk from charring a plate of perforated iron is placed between the lamp and the bottom of the vessel.—E. E. B.

*Improvements in or relating to the Preparation of a Beverage adapted to be used as a Substitute for Coffee.* A. W. Rehnstrom, Malmhammar, Sweden. Eng. Pat. 14,970, September 23, 1889. 4d.

WHEY, milk, or a mixture of both, is boiled down in a vacuum apparatus or otherwise to a pasty consistency, then formed into cakes, dried at not above 100° C., and afterwards cut into pieces about the size of coffee beans, and roasted.—J. M. H. M.

## (B)—SANITARY CHEMISTRY.

*On the Examination of Water for Domestic Purposes.*  
F. Fischer. *Zeits. f. angew. Chem.* 1889, 502—505.

THE author shows by quotations from various authorities how diverse are the opinions held as to the kind and amount of examination needful for passing judgment on the fitness or otherwise of water for drinking. Since the discovery of the cholera bacillus by Koch, physicians especially have been prone to base their judgment on the results of bacteriological tests, a course which would be very praiseworthy if the conclusions drawn were not so widely at variance. Plagge and Proskauer discard a chemical examination, and insist on the water containing no more than 150 germs in 1 cc. by Koch's test, whilst Koch places the limit at 300, and Pfeiffer at 1,000. On the other hand, several investigators have shown that a water containing but few germs when freshly drawn from the spring developed large numbers on standing a short time at the ordinary temperature. Again, it has not yet been shown to be possible to distinguish between harmful and innocuous germs by any practicable test. The only condition generally agreed upon is that a water to be used for domestic purposes should not be contaminated by decomposing matters of human or animal origin. Animal excrementitious matters cannot be identified as such in water, but their ultimate products of decomposition can be determined by chemical means. In the case of spring or river water some of these substances, such as phosphates, ammonia, and potassium salts, may be absorbed by the soil over which the water flows, but the chlorides and nitrates will remain in the water to form a clue to previous contamination. Accepted that a determination of organic nitrogen, ammonia, nitrates and nitrites, and chlorine may be of great importance in forming an opinion on a water, still there can be no standard with which the figures may be compared. Even the results of chemical and bacteriological examination taken together can only be taken to afford an indication that the water may have been contaminated. In all important cases, especially when the water is to be supplied to a town, a reliable judgment can only be arrived at after a consideration of all the circumstances afforded by an inspection of the source and its surroundings as well as the results of examination of samples collected by the expert himself.—G. H. B.

*London Sewerage and Sewage.* Sir Robert Rawlinson, K.C.B. *Jour. Soc. Arts*, 38, 65—82 and 141—156.

IN introducing the reader of the paper the Chairman, Sir Henry Roscoe, M.P., said that in his first report to the Metropolitan Board of Works he had remarked that the use of deodorants ought to be regarded only as a "temporary" expedient, and that the sewage, whether clarified or not, must either be filtered through land or discharged into the estuary at a point not higher than Sea Reach. In a pamphlet laid before the Main Drainage Committee he had expressed his opinion that from a chemical point of view the present process of precipitation did not effect any improvement in the quality of the effluent, that the arrangements at Barking were based on false principles, and that the barging the sewage out to sea was a mistake.

He concluded by expressing his satisfaction that the Main Drainage Committee regarded as a probable alternative to the present scheme at Crossness and Barking the conveyance of the sewage to the sea upon the Essex shores by some such plan as that already sanctioned in the Act of 1865, and that in that event the present works might be available for storage purposes.

Sir Robert Rawlinson: The River Thames flows over beds of oolite, chalk, and London clay, covered mostly with water-washed gravel. These gravelly sub-areas along its course act as natural filter-beds, and justified the poetic title of "the Silvery Thames." The dry weather flow is practically spring water, and frequent floods carried down detritus from the clay and chalk, which acting as a disinfectant purified rather than polluted its waters. The River Lea was its most important northern tributary, having a watershed area of 500 square miles, and this, he thought,

ought to be comprised in the metropolitan main drainage area for purposes of purification.

For all sanitary purposes modern London ought to date from the year 1801, when water-closets were introduced, main sewers were flat-bottomed and arched, and house drains were built of brick from 1 to 3 ft. in diameter without ventilation and untrapped. Fever and disease followed of course, so Parliament passed an Act forbidding communication between these drains and the main sewers, and the remedy suggested at that date was that of cesspools within the basements of houses.

"Dry wells" or "dumb wells," as they were variously called, were universally adopted, and when full to overflowing were often emptied into the sewers with the connivance of the authorities. Under Windsor Castle he himself had found 53 such cesspools or "dumb wells" sunk in various parts of the basement, and under the Board of Health offices at Gwydyr House, Whitehall, he had found three such wells overflowing with putrid sewage.

Such was the condition of London and other large towns in 1848, when the first Public Health Act was passed.

Its local administration was carried on by 300 local bodies, under 250 different local Acts, until 1855, when the Metropolitan Board was established for the especial object of constructing a main sewerage system to prevent further pollution of the Thames, and purify its waters. Already millions had been expended for this purpose, but no provision had been made for conveying as much as possible of the water derived from rainfall on the surface, or from springs beneath it, directly into the river. This had necessitated extra sewers of relief. Moreover, the low-level intercepting sewer under the Thames Embankment would not do its work; its dimensions and capacity having been sacrificed to the Underground Railway, which had been allowed to usurp its site.

This made it necessary to provide storm overflows, which caused pollution of the Thames, contrary to the purpose and provisions of the Statutes under which the Board had been constituted. In the result, periodical floods and pollution were inevitable, and Londoners must bear it patiently. After many experiments, Barking and Crossness were chosen for the position of outlets to the river, in opposition both to public opinion outside and to the decision of the special referees appointed to investigate the subject under the powers of the Act. The total cost of the works, including 82 miles of intercepting sewers, equal to a flow of 400 millions of gallons of sewage daily, storm overflows, reservoirs, and other subsequent additions, was 6,000,000*l.*, and after all this expenditure it was discovered that the reservoirs at Barking and Crossness had not sufficient storage capacity to store sewage until the tide had turned, so that crude sewage forced its way into the river, within the limits of the Metropolis, on the flow, an effect which it was the especial purpose of the Acts to prevent.

London covers an area of 117 square miles, the population being about 5½ millions, residing in 750,000 houses. There are some 2,700 miles of streets sewered, and yet London is not, in its sanitary works, perfect, though on the whole it is the best drained city in the world.

It cannot be said that with all this expenditure and labour the Board performed the work which was the object of its existence, viz., to prevent the continual pollution of the Thames, and their final report proves that they have misinterpreted their Acts and misapprehended their instructions. As to the disposal of the sewage; the authors of those Acts evidently contemplated a profitable disposal. How, he asked, is this possible if it be turned bodily into the Thames? It is true that the old Board tried to find others to do this most important part of their work; indeed, a company was formed with this object in 1865, and obtained an Act under the title of the "*Metropolis Sewage and Essex Reclamation Act.*" It failed to obtain public support, expended 250,000*l.*, including a preliminary deposit of 25,000*l.*, and came to naught. Then came the great "mud contest" 10 years later, the Conservators complaining "that mud banks had been formed at the outfalls," and calling upon the Board to remove them by dredging, as provided by their Act. The Board replied that there were

no sewage sludge banks to be dredged, and the Conservators were defeated in their arbitration. Results have proved that there are fully 1,000 tons per day at Barking alone, and where did that pass, if not to form mud banks in the river channels?

Then came the formidable Royal Commission in 1882, which made inquiries and published reports, with conclusions and suggestions as to disposal of sewage. The Board accepted one part of the recommendations, neglecting the vital one, viz., that the sewage, as a permanent measure, should be purified by intermittent filtration through land. They adopted a separation process, let contracts for mud tanks and mud barges, using chemicals at great cost to do what chemicals cannot do, viz., produce an effluent which will not pollute the river in hot weather!

There have been many schemes propounded, commercial and scientific, but the Board stood to their plan, and left it as a legacy to their successors.

Barking had already protested and complained of the nuisance long before—it was his duty to inquire and report,—but the evidence of nuisance to Barking town failed, and he so reported, though he saw that the river was grossly polluted above and below both the outfalls.

With respect to sewage treatment, the word “disinfect” cannot properly be employed. To “disinfect” the sewage of the Metropolis would cost millions sterling per annum, and according to Dr. Parkes the most powerful “disinfectants” did not give safe results. “Clarification” may be accomplished “temporarily” by chemicals, but will not then produce permanent purity. It will cost 1,500*l.* to 3,000*l.* for each million of gallons per diem, and even 1,500*l.* per million of gallons will amount to 225,000*l.* per annum for the sewage of the Metropolis, whilst with mud barges and other attendant expenses we may calculate the cost at 450,000*l.* per annum, taking the amount of sewage sludge at 28,000 tons per week, which he believed was not an over-estimate.

Experience in this country and elsewhere serves to show that sewage irrigation is the cheapest and best plan, and it has been practised all over the world time out of mind.

Taking the solids in London sewage to amount to 10 tons of sludge per million gallons, we have 547,500 tons of sludge per annum to be removed. There will be in this volume 1,425 tons of water to every 75 tons of solids. Did it ever before enter into the heads of any body of men, engineers or vestrymen, to perpetuate such operations?

Moreover, in hot weather tanks and barges will become putrid, and if disinfectants be used the cost will be very high, whilst land would disinfect fresh sewage at once, and thus save the cost of chemicals, while that which is allowed to pass into the sea will be at once diluted and dispersed.

Meanwhile, the cost bequeathed to the County Council, including workmen's houses and the new sand barges, will exceed 1,000,000*l.* under the head of capital outlay, and that without preventing the continued pollution of the Thames. Such a legacy is equivalent to an entire herd of white elephants!

The expense of taking the crude sewage to the sea at Foulness Island by a new conduit 46 miles in length, with pumping engines and all other apparatus, will cost far less, without taking into account any profit from sewage farming or payments by farmers utilising it on the way. But we have sufficient experience to warrant the belief that it would be cheaper and better to bring the sewage from the south side under the river by a tunnel to Barking, and transport the whole, once and for all, to the North Sea, and then to let reclamation of land and sewage irrigation follow.

Instead of these proposed mud barges or tank steamers it will be possible to send sewage containing 90 per cent. of water to any distance in open troughs or pipes. Sand is so conveyed in Holland and on the banks of the Suez Canal, and this form of conveyance dispenses with the use of sludge vessels, as now proposed.

This or some similar work must be carried out, and the Crossness and Barking outfalls must be so altered and modified as not to pass any sewage at those points into the river.

The plan is feasible, and will supersede the use of costly chemicals altogether, whilst the grand old river, now a loathsome ditch, will become a highway of wholesome water.

Mr. J. G. Rhodes (late chairman of the Main Drainage Committee) contended that the Metropolitan Board had as far as possible carried out the recommendations of the Royal Commission, and in order that the works at Barking might have a fair trial they ought to be allowed to remove the sludge deposited. They had to remove daily about 3,000 tons of sludge, and the question was what the best method of doing so, at all events for the present. The two sludge barges already purchased were clearly inadequate. He admitted that the state of the river last summer was disgraceful! Its bed for a considerable distance above and below the outfalls was covered with sludge. This last summer all “hubbled up” and caused what was termed “black water,” and such was the condition of things that it was necessary to deodorise the river with one of the ships.

If the sludge were not removed next summer the same evil would be repeated and intensified. The public did not appear to fully apprehend the danger of pouring sewage in its crude state into the river. The committee's recommendation is the only practical course at present.

Sir Frederick Nicolson, Bart. (chairman of the Thames Conservancy Board), fully confirmed Mr. Rhodes' description of the state of the river. He was pleased to find that the County Council now admitted that which the old Board had persistently denied. Looking at the matter from a seaman's point of view he was firmly convinced that if these plans went on there must be a series of shallows formed in the part of the river near the outfalls.

Mr. Eneas Smith, as a member of the Main Drainage Committee, was dissatisfied with the present condition of things, and declined to order any more sludge ships. He had no faith in chemicals; and as to the expense, that did not rest on the shoulders of the County Council. Unfortunately there was no alternative but to finish the contract, so far as Barking was concerned. With regard to Crossness, that was a matter for further consideration. There ought to be full and complete inquiry before any further expenditure were incurred.

Dr. Alfred Carpenter commended the paper, and said that the great principle in dealing with sewage was to keep it in motion until it was delivered as fresh as possible on to the land. If this were done they could produce in abundance the meat and milk so much needed in London. The ratepayers must not look for profits, but thousands of acres of lands now worth 5*s.* might be made to yield an average value of 5*l.* per acre. There was plenty of land to be had at Canvey Island, or on some of the wastes round London.

Colonel Jones, V.C. (of Wrexham), said that sewage farming at Wrexham was a success, and his present farm had by means of irrigation during the past 18 years become so enhanced in value that the Corporation declined to pay the price now demanded for its acquisition. They preferred to take another farm two miles farther away from the town and spend money in preparing it for sewage irrigation. The tenant of the new farm was delighted to take the sewage, and if an experiment were made at Canvey Island on a small scale there would soon be a demand there for the privilege of utilising it. Mr. Bailey Denton and himself had already offered to relieve the County Council of London of the expense of building sludge ships, and to carry the semi-liquid sludge down to that island by 12-in. pipes in duplicate, four feet above the surface along the Essex shore; there was no difficulty in driving the whole of the sludge along the pipes, and if it were not liquid enough it might be made so.

Dr. C. Drysdale instanced what had been done by way of irrigation both at Paris and Berlin.

Sir Robert Rawlinson reiterated his conviction that “clarified sewage” was truly polluting sewage, and would cause mischief when it passed into the river.

At Leamington the A. B. C. people proposed to make a valuable manure from sewage, but they were driven out of the place because their effluent polluted the river, and they were restrained by injunction.

Mr. W. C. Sillar strenuously denied the truth of this statement. Their system was tried at Leamington for two years, while land was being prepared for irrigation. As a fact, they saved the town from an injunction, and were publicly thanked by the Corporation for the services rendered during that period.

The discussion on Sir Robert Rawlinson's paper, adjourned from December 18, was resumed, and an *Appendix* to the paper read.

Further tabulated statements had been prepared showing the increase of population since 1855 in the Metropolis, together with the current percentage of mortality in London and five other large cities from 1865 to 1888; another table set forth the population and death rate of London year by year. As the population increased the death rate diminished, the reduction being 6·5 per thousand during a period of 48 years, and the death rate in 1888, 18·5 per thousand. The lesson to be drawn from these figures was that great works of sanitary improvement conducted to the material advancement of the community, notwithstanding their cost and the unjustifiable grumbling over payments to professional men. Imperial and local governments were equally to blame in this respect. The new London County Council had a great work before it, and they could not cheapen it by underpaid professional assistance. The London sewerage system was not yet complete, since the population had nearly doubled since 1855, and had outgrown the works. Sewage tanks, pumping stations, and main sewers on both sides of the river were too small, and must be duplicated. Moreover, further relief sewers and storm overflows were required. Such things as these were the ordinary incidents of great engineering enterprises, and were inevitable at certain stages of their progress. The Metropolitan Board of Works had done great things, and the one great and prime work left undone was the freeing the Thames from pollution.

As to *chemical treatment* of sewage, all inquiry and experience lead to one result, that the richest sewage, unmixing with detritus, would cost the least (to get rid of it) when applied to land in broad irrigation. Excess of water was a disadvantage when land was not available. There was land enough between Barking and the sea to utilise the whole sewage of London either on the way or at the terminus of the Maplin Sands by washing the sludge and passing the clarified water into the sea. Chemicals would not "purify" sewage, nor in his opinion had sludge any commercial value, as shown by the history of Knostrop Works at Leeds, which had proved a costly failure.

A letter from Sir Edwin Chadwick, K.C.B., was then read. The writer said that Sir Robert had overlooked the fact that 20,000 acres in Essex, and 7,000 acres in Middlesex had been offered by practical agriculturists for the disposal of London sewage before 1858, and that sewage-farming was a profitable investment. He might have referred to Bedford, Malden, and Croydon as examples of fertilisation, and where food for five cows instead of one on the same land could be produced. The ratio of fertility might be stated as follows:—That whilst common culture was as 1, market-garden culture was as 3½, and liquefied manure culture as 5. On the subject of London drainage it was agreed that fully two-thirds of the existing sewage was pumped in to be pumped out again, and wasted. That wasted volume should be reduced in the sewers of half the Metropolis for the sake of economy and sanitation. Engineers long ago had been constrained to reduce the size of their drains, for houses as well as sewerage mains, in order to make them self-cleansing. This had been forgotten or "Sir Robert" would not have provided six-inch drains for a single house, which were enough for the drainage of 1,600 houses! He gave him credit for a partial adoption of the separate system by his recommendations at Berlin, Dantzic, and Paris. It was not just to speak of London "as the best-drained Metropolis" in Europe, it should rather be the "least badly-drained"! As to the sanitary condition of first-class houses recently built, not above one in three was free from defects, whilst in the dwellings of wage-earners the mortality was fully doubled by preventable causes. The author of the paper had done good service all over the world, especially on the Sanitary

Commission of the Indian army, by promoting a reduction of the death-rate from 67 to 14 per thousand; in Lancashire also, during the cotton famine, and for these great services the Treasury had awarded him only a third-rate pension of 600*l.* per annum! As to board schools, children were kept in health by constant washing, and changing the air every 10 minutes. This was his own (Sir Edwin's) work, but the reader of the paper did not seem to be aware of it.

Sir Robert Rawlinson explained that he had not mentioned these things—though he knew them well—because they were not germane to the subject of his paper from the point of view of an engineer.

A letter was then read from Mr. John Phillips, the oldest living sanitary engineer of the Chadwick school. He described the condition of London sewerage 50 years ago. Wide leaky sewers, flat-bottomed, full of deposit, opening into the natural watercourses, thence into the river, until it became a perpetually oscillating body of filthy water flowing between banks covered with sewage slush. This was the result of removing both sewage and rainfall together in the same channels, and by this means the ground, air, and water were alike polluted. The houses had leaky cesspools and leaky brick drains which reeked with filth. They emptied the cesspools and abolished them and laid down pipe drains for the first time in the houses and along the watercourses. The result was foul gas everywhere, whilst the river was poisoned ten times worse than before by the augmented volume. This led to a great outcry and to the main drainage of the Metropolis, which they were now discussing. He urged the Commission of 1849 to adopt the separate system when there was time and opportunity to do it extensively. The Commissioners rejected his advice for lack of knowledge of the subject. In his opinion the perpetuation of the old system of combined drainage in the same channels was a national calamity. The separate system suggested by himself is now adopted by the London County Council, but too late, and also by all sanitary authorities of understanding. With respect to the tunnel scheme referred to in the paper, he had proposed one in 1849. It was intended to be laid in an uniform curve from East Ham, threading the windings of the river up the valley as far as Windsor, and to be an intercepting channel for the sewage of the whole district instead of the Thames. From Twickenham to East Ham, 19½ miles, the fall would have been 49·25 ft., or 1 in 2,090, and its depth at the outfall would have been 65 ft. in the London clay . . . . . bored through the solid clay, and "not through water-bearing strata." This tunnel could not have caused the mischief imputed to it. Besides the tunnel, it was intended to lay from west to east on both sides of the river two other intercepting mains—with their outlets also at East Ham and Plumstead Marshes—where the sewage would have been lifted and conveyed by mains through Essex to the North Sea, and through Kent to the English Channel. Farmers on the way might have taken the sewage for irrigation. The result of this scheme would have been abundant meat, milk, and vegetables for the people, and the Thames unpolluted from Windsor to the Nore!

The rainfall would have gone into the river by the existing sewers, after diverting the sewage from them so far as practicable. If this project had been carried out there would have been no need of these proposed open gigantic sewage conduits, as the Thames would have been restored to its pristine purity. He was the inventor of the egg-shaped or parabolic sewer, and had determined the rate of the flow of sewage, so as to preclude the depositing of silt, at 150 feet per minute. The egg-shaped sewer was now universal, and his formula for non-deposit flow of sewage, together with the proposed separate system, had been adopted in all modern town drainage schemes. He agreed, with Sir Robert, that these standard improvements deserved recognition.

Mr. Clare Sewell Read said that cheapened nitrates at about 1*d.* per lb. had driven sewage fertilisers out of the field. A point in favour of sewage farming was the introduction of "silos." This enabled the farmer to store his grass in any quantity until it was wanted. Sewage

grass made capital ensilage, and produced the best possible milk. As to manure from sewage sludge, he had tried it, and it did no good to his crops. If fortified by other fertilisers it might be a good vehicle for distribution over the land. Of itself, it was not worth the carriage of five miles. He was in favour of broad irrigation when they could get the land. If not, a good deal was to be said in favour of precipitation processes. With respect to the alleged famine of milk in London, he could supply abundance of it at 6d. per gallon. In town they would have to pay 2d. per pint for it. Who got the profit? Certainly not the farmer. Sewage was a nuisance and they must get rid of it. As to the great conduit to the North Sea, if the farmer could tap it on the way and use it for his land only *when he wanted it*, it would do great good; when not required the surplus could be delivered into the ocean. The Craigentiny meadows near Edinburgh were let at a fabulous price. The land was porous, and when it did not take the sewage it was discharged bodily into the sea.

Mr. W. C. Sillar: Although the author of the paper spoke with authority, some of his conclusions were at variance with the facts. Sewage treatment consisted either of filtration, irrigation, or precipitation. In the old days precipitation meant nothing but treatment by lime, which was destructive of manurial value in the sludge and offensive withal. If that was what the author meant by precipitation he rightly preferred irrigation; but the world had gone round since that day, as might be seen at Kingston-upon-Thames—above Teddington Lock. The effluent, good enough above Teddington, was good enough also for Barking or the Clyde. The author did not believe in the efficiency of chemical systems, nor in the value of sludge, but Wellington College, Aylesbury, and Kingston-on-Thames had come to a different conclusion, and had expressed it in official reports. "Sir Robert" himself thought differently when he visited Aylesbury many years ago. He was pleased then, but his official sense of responsibility restrained him from expressing a preference for any one process over another. Against the unsupported statements of his paper he would place the opinions of the medical officer of health of Kingston and the public declarations of the town clerk of Aylesbury from time to time during the past ten years.

With regard to the value of the sludge, he could prove his case by volumes of testimonials from farmers and gardeners, who had repeated their orders from time to time.

Portable manure had been made from sewage for 14 years. It was made and sold at Kingston-on-Thames, and the mayor of that town was there to certify the fact. The author had said that "the Native Guano Company" had been driven out of Leamington by an injunction—or the fear of one. The facts were, that an injunction had been obtained long before against the Corporation of that town for a nuisance arising from the working by them of a different process, and the A. B. C. Company was then called in to supersede that process, and help the Corporation over their difficulties. They did so successfully until the works were completed, and were thanked for it. He could not believe in Lord Warwick's threat of an injunction, for the company never had reason to fear it. He objected to the "open conduit" proposed. There would be deposition in a sluggish stream of 46 miles in length, and the liquid would become stale and offensive before it reached the sea. When it did it would float, not sink, and the slime upon the beach would stink abominably in hot weather. This project might be useful if there were no alternative, but there is an alternative; and the County Council were justified in making the experiment of precipitation, and giving the works at Barking a fair trial. If it failed, a better scheme could be found and adopted in lieu of it hereafter.

Dr. Dupré, F.R.S., objected to feeding men with things cultivated on sewage farms. As to Berlin, they could not compare a sand waste to the heavy land of our sewage farms. The author was wrong in his estimate of the cost of the Board of Works present scheme. If it cost more than 118,000l. per annum it was not the scheme as recommended to and accepted by that Board. It had never been alleged that no sludge was sent into the river, nor did they deny that the so-called sewage banks were sewage-mud. At one

inquiry others admitted that 250 tons a day went in, at another 180 tons, and 207 tons. Precipitation processes "clarified" water, and removed sludge, and the minimum of material for such purpose should be ascertained and used, seeing that every grain of material added to a gallon of London sewage resulted in 100 tons of wet sludge per day. All the lime used would be effectually precipitated. None would pass into the river. By clarifying the sewage it reduced the polluting effect to about one-half; that is, the pollution of the river was reduced from the effect of 5,000,000 people to that of 2,000,000 people. In summer the effect was vastly greater, as every chemist who had investigated the subject knew; in cold weather the sludge matters remained and settled down unaffected, and when summer came they had to defecate the sludge of the previous eight months, besides the sewage of the day. In the hot weather the sewage of 5,000,000 people would be reduced to that of 1,000,000 people, and when London had reached a population of 10,000,000 inhabitants the polluting effect would be equal to that of 2,000,000 people. If the County Council were wise, they would accept the scheme handed down to them. By the year 1914 they might reduce the polluting effect to nothing. With respect to deodorisation, the production now of manganese was enormous, and the price had gone down from 40l. per ton, without a guarantee, to 8l. per ton with one.

Dr. Thudichum had noticed many inaccuracies in the paper. As to the cost of clarification he had charged almost double the actual expense.

The weight of sludge would average 3,500 tons per day, containing 90 per cent. of water. The London sewage amounted to 272,750,000 tons per annum, and if it were correct that land under favourable conditions could not take more than 5,000 tons per acre annually, then it would require 55,000 acres of land to take the whole sewage of the Metropolis! If Dr. Lethby were right, it would require 200,000 acres—not quite three times the present area of London; so that to lay London sewage in a farm was chimerical. The sludge steamers were fine vessels of 2,000 tons each, and well appointed. He had been on them, and it was untrue that they became putrid and required disinfection. The cost of wages would be 40,000l., not 50,000l., per annum, and chemicals for precipitation would cost 20,440l. per annum. He put the total cost of treating the sewage and taking the sludge to sea at 130,000l. per annum. Give the scheme a trial, otherwise the 1,000,000l. sterling spent would be sunk altogether.

Professor Corfield approved the conclusions of the paper. London was the healthiest city of the world, because the excreta were removed continuously. Although removed from London it became a nuisance in the Thames, and it now remained to get rid of the nuisance there by carrying it practically to the sea, and by this measure the County Council would justify its existence.

Mr. Arthur Angell denied that the sewage would be got rid of when it came to the end of the proposed conduit. It would not mix with the sea, and could not be disposed of in that manner. It was simply shifted from one situation to another. Sewage farms were a nuisance in many places, and at Croydon, the best managed of all, they lost 12,000l. per annum. In a conduit with a sluggish flow of two miles per hour, there would be silt and deposit of sewage matter, just as in the Mersey and the Dee there were banks of mud owing to the sewage poured into these rivers on both sides. As to the Paris farm near Clichy, it was a chemical swindle, for the sewage was allowed to pass crude into the Seine hard by.

Mr. Isaac Shone was of opinion that the data and calculations of the paper were accurate. Taking the population at 5,500,000, the solid faeces amounted to 384 tons daily, and the fluid to 6,140 tons, or a total of 6,254 daily. The sewage, at 30 gallons per head per diem, amounted to 736,607 tons. Thus the sewage proper was only .82 per cent. of the volume of sewage, which would be represented by one glass of brandy in 99 glasses of water.

Mr. James Lemon said that there was much diversity of opinion among eminent authorities as to the effect of taking the sewage lower down the river. Some thought that it

would not remedy the nuisance, but only alter its locality; others thought that it ought to be subjected to a purifying process before discharge at any point of the river. To carry the sewage of 7,000,000 inhabitants to the sea—for that was the least number to be provided for—was a gigantic operation, and would cost, according to Mr. Baldwin Latham, at least 6,000,000*l.* sterling.

They might purify sewage well on high land, according to his experience; on low land it was difficult to do it. It might at some seasons be effectual, at other times the sewage would all go into the river in its crude state.

Sir Robert Rawlinson, in reply, said that he had not intended to treat the subject from a chemical standpoint, but apologised to Mr. Sillar for his mistake concerning Leamington. It was a mistake to say that "the company were driven" from Leamington. The fact was that the injunction was obtained against the local board of that town, but was suspended for some time to allow the construction of pumping works to remove the sewage to the land. In the year 1869 the A. B. C. Company undertook to deal with the sewage, and did so for a while. The result was set forth in an official report of that date. He also referred to another old report of the Metropolitan Board of Works on experiments made at Crossness in the year 1872 as to the cost of the process. All this, he said, went to show that commercial profit out of sludge was impossible. It ruined two companies at Coventry, although they received 2,000*l.* per annum by way of subsidy.

The Chairman, Sir Henry Roseoe, M.P., F.R.S., in moving a vote of thanks to the author of the paper, said that the subject was of the utmost importance to the nation, and some rational and thorough-going scheme ought to be adopted. Whatever scheme were selected, the work should be done effectually. The question of cost was of much less consequence. If the County Council achieved the work to the satisfaction of the coming generation, they would have earned the thanks of the nation.

Mr. Frank Candy wrote, that Sir R. Rawlinson's estimates were not dated on present prices, but upon data given in 1857! At modern estimates, the annual charge would amount to 300,000*l.*!

He pictured a "new river" of London sewage flowing sluggishly through Essex for 46 miles. Such a river would be in a putrescible condition, and in hot weather would become offensive to the districts through which it passed. With respect to discharging this filth into the estuary of the Thames, what would the towns near the coast have to say? He did not believe in taking the sewage through the marshes for purposes of agriculture. It had failed before—and ignominiously at Liverpool—although the company offered to give the sewage for nothing if the farmers would utilise it.

In this case, if the farmers did not refuse, there was no guarantee that it would be effectively purified, some of it might go crude into streams and watercourses.

Mr. Alfred Roechling wrote, that where London and Paris had failed, Berlin had succeeded. That city was divided into 12 separate drainage systems, called "radial systems," seven being north and five south of the River Spree. Each radial system was entirely independent, and all the sewers radiated from the circumference of the city towards the centre, where the pumping station was situated. Each area averaged about 1,150 acres, with a maximum population of 200,000 in the largest of them. There were storm-overflows connected with the river. The systems lying north of the river pumped their sewage to farms on that side of the city, those on the south to farms on the south side. A total area of 7,900 acres had been specially prepared for irrigation, and more was dealt with every year as the radial systems were completely connected.

The total average of all the farms had now reached 16,477 acres, and the total dry weather flow amounted to 27,000,000 gallons, the water supply only amounting to 14 gallons per head. The population in 1885 was 1,293,920. The farms on the northern side were six miles distant from the heart of the town, those in the south about 12 miles. The health of the labourers on the farms was good, the death rate being

11 per thousand during the last year. The effluent from the northern farms joined the River Spree above Berlin. Sewerage and sewage disposal works had cost 3,943,402*l.* The income from the farms last year was 95,000*l.*, the expenses 86,570*l.*, leaving a surplus of 8,457*l.* in aid of capital and interest. The cost of sewage disposal on the farms was about 10*l.* per head of the population.—C. N. C.

*The Composition, Treatment and Use of Feces as Manure.* Imperial College of Agriculture and Dendrology, Komaba, Tokyo, Japan. Bull. 3, 1888, 1—25.

See under XV., page 86.

## PATENTS.

*Improved Apparatus for Preparing Lime and other Substances for the Treatment of Sewage, and for Automatically Supplying them in Measured Quantities thereto.* E. Knowles, Accrington. Eng. Pat. 15,810, November 2, 1888. 8*d.*

THE object of this invention is to reduce, as far as possible, the amount of manual labour in the preparation and application of lime or other substances used in the purification of sewage, and their more equal distribution to the sewage.

Lime is first slaked to a dry "fallen" condition, and then fed into a "pulveriser," a revolving box of such suitable form and having such interior arrangements as to induce a further disintegration of its charge.

The box is provided with a grating or gratings, through which the lime is sifted and delivered in a finely-divided state. It is then lifted, by means of an elevator, into a receiver, from which it is drawn out through an opening at its lower end, by means of a helical rotating "creeper," and is introduced, together with a stream of water, to the "mixer," a vessel fitted with revolving stirrers, and having separate driving gear.

The supply of lime to the mixer is automatically proportioned to the varying quantity of sewage to be dealt with, the quantity of material delivered to the mixer being due to the greater or less velocity of the above-mentioned creeper, or its equivalent. The differential action of the creeper is made to depend on the vertical movements of a float situated in the sewage flow. The float is connected by rods and levers to actuate the creeper, so as to deliver the lime to the mixer in varying proportions due to the quantity of sewage passing.—F. S.

*Improved Method of and Apparatus for the Formation of Organic Oxides, and the Oxidation of Matter Suspended or Dissolved in Liquids.* J. G. Lorrain, London. Eng. Pat. 16,093, November 7, 1888. 6*d.*

OXYGEN, or common air, is blown against the surface of porous carbon immersed in the liquid to be treated, or oxygen is liberated at the surface of the carbon by electrolysis, if such be possible. The carbon absorbing the oxygen facilitates chemical action with the liquid. The invention may be employed in the apparatus described in Eng. Pat. 3826 of 1886 (this Journal, 1887, 377). Freshly prepared "kerosene coke" or ordinary cinders are preferred for the immersed carbon.—E. T.

*Improvements in and connected with the Purification of Sewage and other Foul Liquids.* C. H. Beloe, Westminster. Eng. Pat. 18,568, December 19, 1888. 11*d.*

THE object of the invention is to provide means whereby the deodorising material is automatically mixed in the proper proportions with the liquid under treatment. For this purpose the inventor suspends in the sewer or other channel through which the liquid passes a lever or arm, free to swing therein, and having at the lower end a paddle shaped to correspond with the bottom of the sewer. The upper end of this arm is connected with a cut-off valve, which controls the mouth of a hopper containing the material to be supplied to the liquid. Above this cut off valve is a second valve which regulates the maximum



amount of material which is to be added to the liquid, and below it is a spout to deliver the material into the sewer. When no liquid is flowing the arm hangs vertically, and the mouth of the hopper is closed, but on the flowing of the liquid the arm is deflected and the valve opened. The greater the flow the greater the deflection of the arm, and consequently the greater the amount of material which can pass down from the hopper. Immediately adjoining the spout is a chamber containing a number of baffle-plates or weirs to cause the liquid and the disinfectant to become thoroughly mixed. In another modification the valve is worked by an under-shot water-wheel.—F. S.

*Improved Apparatus for Separating, Filtering, and Disinfecting Fæcal Matters.* J. Chabanel, Marseilles, France. Eng. Pat. 18,791, September 18, 1889. 8d.

THE object of this invention is to secure efficient working of apparatus for separating fæcal matter on the portable tank system, and to enable it to be emptied without stoppage of its working.

The separator is cylindrical in the body, tapering at the bottom to a narrower mouth which fits on to the mouth of a removable tank placed below. The separator contains a perforated cylinder of the same diameter as, and connected with, the mouth of the vessel itself, and the soil pipe opens directly into this cylinder. The mouth can be closed—to enable the tank to be emptied—by a piston which can be raised or lowered, from above, by a screw, the piston being in connexion with a door which closes the mouth of the soil pipe. The tank below is mounted on a table which can be raised or lowered by a screw, and is provided with a cock for drawing off a part of the contents should it be found too full for removal.

A pipe from the separator conveys away the liquid matter, so that if need be the mouth of the soil pipe can be got at for cleaning.

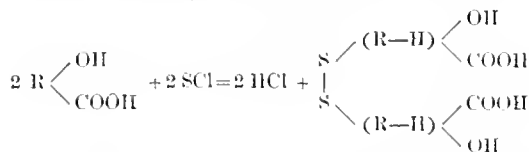
The inside of the cylinder is cleaned by the ascent and descent of the piston.—F. S.

## (C)—DISINFECTANTS.

### PATENTS.

*Process for the Production of Aromatic Dithio-organic Acids.* H. Baum, Frankfort, Germany. Eng. Pat. 14,443, October 8, 1888. 4d.

A SERIES of interesting and more or less important dithio-acids may be prepared by the action of sulphur chloride or bromide upon "oxycarbon acids" of the aromatic series, their formation being thus represented:—



To produce dithio-salicylic acid, for example, 138 kilos. of salicylic acid are heated with 68 kilos. of sulphur chloride ( $\text{S}_2\text{Cl}_2$ ) between  $100^\circ$  and  $150^\circ$  C. until the evolution of  $\text{HCl}$  ceases. The resulting acid is a honey-yellow substance readily soluble in soda without leaving any residue of sulphur. The acid is easily soluble in "benzole" (benzene) and glacial acetic acid, forming when dried and cooled a straw-yellow brittle mass meltable like resin.—O. J. S.

*New or Improved Compositions for Disinfecting, Deodorising or Antiseptic Purposes.* G. R. Tweedie, London. Eng. Pat. 17,042, November 23, 1888. 4d.

THE improved composition consists of a mixture of phenols and terpenes with or without a dry mineral or vegetable powder as vehicles. For example, 1–2 parts of carbolic acid, 4–5 parts oil of turpentine, 1–2 parts of camphor.

These may be simply mixed, or distilled together. As a dry vehicle, powdered alum, borax, kieselsäure, peat, sawdust, moss, &c., may be used.—J. M. H. M.

*Improved Means to be used in the Preservation of Perishable Substances by Fumigation.* G. D. Wheaton, Chicago, U.S.A. Eng. Pat. 18,079, December 11, 1888. 8d.

THE apparatus consists of a chamber divided into two parts. The lower receives the substances to be preserved whilst the upper consists of refrigerating chambers containing flues through which the fumes of the antiseptic compound pass before they can gain access to the substances in the lower part of the chamber. Any suitable antiseptic may be employed, but preference is given to the compounds patented by W. D. Daniels (Eng. Pats. 17,813 of 1888 and 2858 of 1889; this Journal, 1889, 411). Drawings of the apparatus accompany the specification.—E. E. B.

*Improved Means of Effecting the Preservation of Materials of a Perishable Nature, and the Application thereof for Curative Purposes.* G. D. Wheaton, Chicago, U.S.A. Eng. Pat. 771, January 15, 1889. 6d.

THE articles to be preserved are treated with water which has been exposed to the fumes of a mixture of sulphur, sugar, sassafras, cinnamon and potassium nitrate.

If a liquid has to be preserved a portion of it may be subjected to the action of the fumes of the above mixture and then mixed with the remainder of the liquid. The same methods may also be adopted with solid substances, such as butter.—E. E. B.

*An Improved Manufacture or Preparation of Antiseptics.* F. G. A. Roberts, A. Shearer, and W. B. Giles, London. Eng. Pat. 11,974, July 27, 1889. 4d.

DRY salicylic acid is mixed with an anhydrous alkaline sulphite, preferably in about equal parts by weight.

—E. E. B.

*An Improved Disinfecting, Deodorising, and Preserving Compound.* J. G. Fowler and D. Macdonald, Boston, Mass., U.S.A. Eng. Pat. 14,032, September 5, 1889. 4d.

A MIXTURE of boracic acid, 10 parts; salicylic acid, 10 parts; permanganate of potash, 40 parts; and silicate of potash or soda, 40 parts. Ten parts of fresh water sand may be substituted for 10 parts of the alkaline silicate. The mixture forms a hard mass, lumps of which may be placed in water used to supply closets, &c.; it may also be used for washing, preserving meat, &c.—J. M. H. M.

## XIX.—PAPER, PASTEBOARD, Etc.

*Microscopic Examination of Paper.* W. Herzberg. Mitt. Königl. tech. Versuchs. 1889, 3, 113–115.

See under XXIII., page 112.

*Contributions to the Knowledge of the Sizing of Paper.* W. Herzberg. Mitt. Königl. tech. Versuchs. 1889, 3, 107–113.

(A.) *Action of Light on Rosin Sizing.*—It was observed that the label of a laboratory book which had been begun to be used in 1884, and was then quite free from any tendency to make the ink "run," had completely lost this property when the date on which it was filled was inserted in 1887, proving that some alteration had taken place in its sizing. Five kinds of paper were therefore exposed between double windows to direct sunshine and air for two periods of two



months each, and tested at the end of each period as to the permanence of their sizing. They had been proved normal in this respect at the beginning of the experiment, both by their ability to bear writing upon without running, and by Leonardi's test (see below). At the end of the second period of two months, four out of the five kinds (which comprised paper from linen and cotton rags with and without wood and straw cellulose) were no longer fit for writing upon, while the fifth, made of a mixture of wood pulp and wood cellulose and containing 13.5 per cent. of ash, was almost unaffected, although its colour was most altered, having changed to a well-marked brown tint. That this effect of exposure to air and sunshine on the rosin used in sizing the papers was due to the action of light, and not to that of oxygen or carbon dioxide, was shown by experiments in which the papers were enclosed in vessels filled with these gases and kept in diffused daylight without suffering change.

The exact nature of the alteration in rosin brought about by the action of light is difficult to determine. A piece of rosin exposed to sunshine loses its vitreous appearance and becomes covered with a yellow powder; a similar change in the rosin on the paper would cause it to lose its impermeability. The reason for this change in the rosin may be the loss of certain volatile constituents followed by crystallisation of the residue, in a manner analogous to the alteration of barley sugar from the vitreous to the crystalline state consequent on the volatilisation of the small quality of water it originally contained.

(B.) *Characteristic Difference of Animal and Rosin Sizing.*—A paper sized with rosin, when extracted with absolute alcohol gives a solution which, poured into excess of water, yields a milky turbidity due to precipitated rosin. Another test is based on the Raspail reaction, rosin giving with sugar solution and sulphuric acid, a violet-red colour similar to that produced by albuminoids. The sugar may be omitted as enough is formed for the reaction by the action of the sulphuric acid on the cellulose of the paper.

The presence of animal size is detected by treating the aqueous extract of the paper with tannin. The following fundamental distinction between papers sized with rosin and gelatin is found to exist. In the former the rosin is distributed uniformly throughout the substance of the paper, while in the latter, whether the sizing has been performed in the pulp or sheet, it is always found exclusively on the surface of the finished product. This peculiar property of gelatin can be shown by saturating a plaster of Paris slab with gelatin solution coloured suitably, and breaking it when dry, on which it will be found to be coloured to a trifling depth, the inner part being white. On these facts this test is based:—A half-sheet of paper is repeatedly crumpled and unfolded, and when the surface has been thoroughly chafed is smoothed out and written upon; if it is sized with rosin the inscribed characters are but little blurred, while if animal size has been used they run freely and are visible from the opposite side of the sheet. Leonardi has modified this test, removing the doubtful element introduced by the manual use of pen and ink. A pipette of which the exit is 10 cm. above the paper, and which delivers drops weighing 0.03 grm. each, is filled with a solution of ferric chloride containing 1.531 per cent. of iron. A single drop is allowed to fall and to remain on the paper for the same number of seconds that 1 sq. m. of the paper weighs in grammes, when it is removed by blotting paper, and the under side of the paper dabbed with a plug of wadding wet with a weak solution of tannin; the production of a black colour proves the iron solution to have penetrated, and therefore shows the sizing to be of animal origin, while in the contrary case the presence of rosin is demonstrated.—B. B.

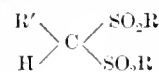
## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

*Physiological Action of Sulphones.* E. Baumann and A. Kast. *Zeits. f. physiol. Chem.* 1889, **15**, 52.

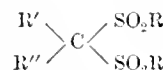
EXPERIMENTS on the physiological action of sulphones and disulphones showed a distinct connexion between constitution, on the one hand, and action on the system, with corresponding decomposition of the substance, on the other hand. It was thought desirable to ascertain in how far the action of sulphonol corresponded with the action of similarly constituted bodies; whether any influence was exerted by the sulphonic group, or by the ethyl, or methyl groups; and, finally, what differences manifest themselves in disulphones by substituting ethyl groups for methyl in the sulphonic groups. Disulphones, in which the sulphonic groups are attached to different carbon atoms, were not considered; the action of those only was considered in which the sulphonic groups are both attached to one and the same carbon atom. Such disulphones are divisible into three distinct classes—

- (1)  $\text{CH}_2(\text{SO}_2\text{R})_2$  ..... methylene disulphones.
- (2)  $\text{CH}.\text{R}'(\text{SO}_2\text{R})_2$  ..... methenyl disulphones.
- (3)  $\text{CR}'\text{R}''(\text{SO}_2\text{R})_2$  ..... ketone disulphones.

The experiments show that diethyl sulphone, the methylene disulphones, and ethylenediethyl sulphone pass through the system undecomposed, and consequently these bodies exert no action on the system. The methenyl disulphones—



are for the most part decomposed in the system, but the ketone disulphones—



are so completely transformed that traces of the same may only be detected in the urine after administration of considerable doses. It would thus seem that in the system the ketone disulphones are the most easily decomposed of all. At the same time they resist the action of alkalis, strong acids, and oxidising agents, as sulphonol itself does, more than the other disulphones. And in physiological action, just as in chemical character, the methenyl disulphones rank themselves between the other two classes. The experiments show, moreover, that only such disulphones are active as contain ethyl groups, and the intensity of such action is dependent on the number of ethyl groups. The group  $\text{SO}_2$ , as such, exerts no action; the group  $\text{SO}_2\text{C}_2\text{H}_5$  is equivalent to an ethyl group combined with the same carbon atom. The authors are thus led to consider that the sulphur has only an indirect action on the system in so far as a stable compound containing two ethyl residues is thereby obtained. Owing to this is doubtless due the limited action of sulphonol on the system; and this may serve to explain why fatalities by excessive doses of sulphonol have not occurred.—T. L. B.

*Synthesis of Pyridine and Piperidine Bases containing Oxygen.* A. Ladenburg. *Ber.* **22**, 2583—2590.

REFERRING to previous investigations on the subject, the author states that he obtained remarkable results by means of the three following reactions:—(1) Substituting alkyl radicles in the pyridine nucleus, by heating pyridine bases with alkyl-iodides. (2) Converting pyridine bases into the corresponding piperidine bases by means of sodimi and absolute alcohol. (3) Forming condensation products by acting with aldehydes upon substituted pyridine bases, and thereby forming unsaturated compounds.

For the last two years the author tried to find a method of preparing pyridine bases containing oxygen, and has at

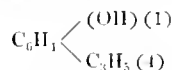
last succeeded, by forming direct addition products from a substituted pyridine bases and aldehydes, without elimination of water. He describes the bodies thus obtained as "alkines," and has prepared the following compounds: *α-picolylalkine*,  $C_5H_7NCH_2CH_2OH$ , boiling at  $179^\circ C$ , under 25 mm. pressure, and forming a colourless syrup. On treating this base with potash or concentrated sulphuric acid, vinyl-pyridine,  $C_5H_4NC_2H_3$ , is obtained. *α-pipecolylalkine*,  $C_5H_9(CH_2 \cdot CH_2OH)NH$  (the expression "pipe" being used as an abbreviation for the radical of piperidine). By methylating this body, methylpipecolinalkine is obtained, which is of some interest, as it only contains two hydrogen atoms more than tropine. He also prepared *α-pipecolylmethylalkine*,  $C_5H_9(CH_2 \cdot CH_2OHCH_3)NH$ , which body stands in close relation with Wertheim's *conydrin*.—O. J. S.

*Cinnamylcocaine*. C. Liebermann. Ber. 22, 2661—2662.

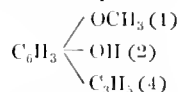
The cinnamylcocaine found by Giesel (this Journal, 1889, 1004), as predicted by the author, in the amorphous bases accompanying cocaine, has been further examined by the latter. As Giesel has proved the identity of the natural base with that synthesised by the author as far as regards the following properties, melting point, appearance, solubility, and behaviour on decomposition, there only remained the careful examination of the crystalline form to definitely establish the identity. The crystalline form of cinnamylcocaine obtained from coca leaves is now found to be precisely the same as that of the artificial product.—E. B.

*On the Etheral Oil of Betel Leaves*. J. F. Eykman. Ber. 22, 2736—2754.

The author prepared, in 1885, betel oil from freshly gathered leaves of *Charicia Belle Miq.* = *Piper Belle L.* of the botanical gardens in Buitenzorg (Java). This oil is a slightly greenish-yellow coloured liquid of an aromatic taste and smell, which turns brown on being exposed to the action of air, and at the same time acquires a burning taste. The oil is slightly levo-rotatory ( $-3.5^\circ$  pro 2 dm. layer in sodium light), and has a specific gravity of 0.959 at  $27^\circ$ . The author has succeeded in isolating three chief components from this oil, citreneterpene, a sesqui-terpene, and para-allylphenol (*Charicol*).—



whereas, according to a report of Schimmel and Co., in Leipzig, betel oil contains chiefly eugenol and a hydrocarbon boiling at  $260^\circ$ . In a later publication, however, on this subject by the above firm, betel oil was found not to contain eugenol, but an isomeric body—



(Bertram and Gildemeister, this Journal, 1889, 816), which they named "betelphenol." The phenolic body charicol, which was isolated from the oil by means of KOH and precipitation with dilute acid, boils at  $237^\circ$ , and forms a colourless clear liquid of characteristic taste and smell, and has a specific gravity of 1.041 at  $13^\circ$ . According to the author, charicol possesses a far greater antiseptic power than either phenol or eugenol, being about five times stronger than phenol and twice as strong as eugenol. The hydrocarbons isolated from the oil boiled between  $173^\circ$ — $190^\circ$  and  $190^\circ$ — $220^\circ$ , did not yield a pure terpene of a constant boiling point, but probably contained cymol and cineol, &c., but no pinene. The lower boiling portion of citrene, with a decided smell, had a specific gravity of 0.848 at  $16^\circ$  and a rotatory power of  $-5.20^\circ$ . The other part had a greater rotatory power, and probably contains menthol. The author concludes, after carefully comparing this oil with the oil from Schimmel and Co., that they are distinctly different, and as the latter does not contain the lower boiling bodies, they can hardly claim the name betel oil for their product.—O. J. S.

*On the Chemical Constitution of Safrol*. T. Poleck. Ber. 22, 2861—2863.

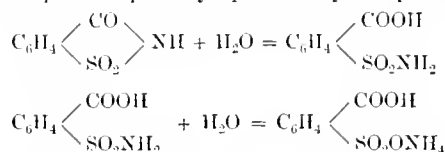
In order to substantiate the formula of safrol, which was supposed to be allyldioxybenzene,  $C_6H_6(O_2CH_2)_2C_3H_5$ , this body was oxidised with a 4 per cent. solution of potassium permanganate. The products obtained, however, were piperonal, piperonic acid, carbonic acid, formic, acetic and oxalic acid, but no propionic acid. Under these circumstances the present formula of safrol can be maintained no longer.—O. J. S.

*On Hydrocarbon in the Etheral Oil of Carlina Acaulis L.* F. W. Semmler. Chem. Zeit. 13, 1158.

The etheral oil of the earline thistle, which is dark reddish brown with the characteristic narcotic odour of wurzel, sp. gr. 1.026 at  $21.9^\circ$ , boiling point  $265^\circ$ — $300^\circ$ , gave on ultimate analysis C, 85.89; H, 6.75; O, 7.36. By distilling in vacuo a clear yellow fraction distilling at  $155^\circ$  was obtained, of which the ultimate analysis was C, 87.42; H, 10.84. This fraction redistilled in vacuo over metallic sodium gave a colourless distillate of elementary composition C, 88.21; H, 11.74. Thus undoubtedly a hydrocarbon is obtained: the formula  $(C_5H_8)_n$  corresponds to the terpene series. The boiling point at the ordinary pressure is  $250^\circ$ — $253^\circ$ . From the crude oil 12 per cent. of this hydrocarbon can be obtained. The author is engaged on research of further bodies in this oil.—D. A. S.

*Action of Dilute Acids on Benzoylsulphonic-imide and the Analysis of Commercial Saccharine*. I. Rensen and W. M. Burton. Amer. Chem. Jour. 11, 403—408.

When benzoylsulphonic-imide is boiled with hydrochloric acid it is first converted into orthosulphamiebenzoic acid, but on continued boiling it is completely converted into hydrogen ammonium orthosulphobenzoate; the changes which take place are probably represented by the equations—



A number of experiments showed that this change is brought about most quickly (in 8—10 minutes) by an acid prepared by diluting pure hydrochloric acid of sp. gr. 1.17 with 8—10 parts of water. The concentrated acid has only a slow action at  $100^\circ$ , but at  $150^\circ$  the change takes place rapidly. The end of the reaction can be readily ascertained by tasting the solution, the quantity of the orthosulphamiebenzoic acid present being insignificant.

The easy conversion of benzoylsulphonic-imide into a substance which is readily soluble in water suggests a method for determining this constituent of saccharine.

The commercial article contains at least three substances, namely, benzoylsulphonic-imide, parasulphamiebenzoic acid, and potassium hydrogen orthosulphobenzoate, possibly also some unoxidised sulphoniamides. When commercial saccharine is boiled with excess of dilute (1:8) hydrochloric acid the sulphimide is converted into ammonium hydrogen orthosulphobenzoate, whilst the other compounds remain unchanged.

If the solution is concentrated and allowed to cool, the parasulphamiebenzoic acid, which is very sparingly soluble in cold water, crystallises from the solution (quite free from any ortho-acid) and, on filtering, the potassium and ammonium salts of the orthosulphobenzoic acid will be present in the filtrate. The combined weight of these salts can be determined by evaporating to dryness, and the quantity of potassium salt in the residue can be ascertained by estimating the quantity of potassium.

The difference between the combined weight and that of the potassium hydrogen orthosulphobenzoate alone gives the weight of the ammonium hydrogen orthosulphobenzoate, and consequently that of the original benzoylsulphonic-imide. No potassium salt, other than that of orthosulphobenzoic acid, was detected in experiments with commercial saccharine.

The most convenient method for the analysis of saccharine was found to be the following:—About 2 grms. of saccharine are boiled for an hour with 100 cc. of hydrochloric acid (1:8) in a flask of 250 cc. capacity provided with a reflux condenser. The clear solution is then concentrated to 15 cc. in a dish, allowed to stand for several hours, the crystals of the parasulphonic acid collected on a weighed filter, washed with cold water, dried at 80° and weighed. The filtrate and washings are evaporated to dryness, weighed, and the potassium in the residue determined by treating a weighed portion with sulphuric acid.

The results of five parallel analyses of two samples of saccharine are given below (in percentages):—

SAMPLE I.

—	I.	II.	III.	IV.	V.
Parasulphamiebenzoic acid..	49.20	50.36	48.70	51.01	50.75
Benzoylsulphonic-imide .....	32.12	32.71	33.19	33.80	32.18
Potassium hydrogen sulpho- benzoate.....	7.20	6.94	7.21	7.22	7.12

SAMPLE II.

—	I.	II.	III.	IV.	V.
Parasulphamiebenzoic acid..	43.79	45.13	45.26	43.48	44.78
Benzoylsulphonic-imide .....	48.65	48.29	47.95	48.22	48.53
Potassium hydrogen sulpho- benzoate.....	8.10	7.98	7.90	7.99	7.99

These analyses show that the quantity of benzoylsulphonic-imide in commercial saccharine is somewhat less than 50 per cent., so that the commercial article has a sweetening power about 125 times as great as that of cane sugar.—E. S. K.

#### A New Iodoform Industry. S. Casthélaz, Bruère, and Co. Rép. de Pharm. 45, 481.

Iodoform is prepared from Varce soda by Suilliot and Reynaud's process (see this Journal 1889, 210) at the manufactory of S. Casthélaz, Bruère, and Co., at la Poterie-de-Belbeuf, near Rouen. The alkaline iodides are extracted from the Varce soda by systematic washing with cold water, or by means of steam extraction, aided by a mechanical stirring apparatus. The solution obtained is desulphurised by known methods and filtered. The clear filtrate is mixed in a large vat, provided with stirring apparatus, with sodium hydrate, acetone, and sodium hypochlorite. The iodoform which separates is purified in the usual way. The mother-liquor from these operations is worked up, first for potassium sulphate and chloride, and then for bromine.—H. T. P.

#### Action of Hydrocyanic Acid on Calomel. Fouquet. J. Pharm. Chim. 1889, 397.

THE gray colour caused by the action of hydrocyanic acid on calomel has long been known. *Scheel* gives the reaction:  $2 \text{HgCl} + 2 \text{HCN} = \text{Hg} + \text{Hg}(\text{CN})_2 + 2 \text{HCl}$ . *Bussy* and *Buquet* treated 1 gm. of calomel repeatedly with hydrocyanic acid (1:10) and obtained 0.42 gm. of mercury and 0.56 gm. of mercuric chloride, and concluded that the hydrocyanic acid simply decomposed the calomel into mercury and mercuric chloride. As, however, this ran counter to thermo-chemical considerations, the author repeated their experiments and obtained the same quantity of mercury left, but on examining the filtrate found that it was not a solution of mercuric chloride mixed with hydrocyanic acid, but one of mercuric cyanide in hydrochloric acid, for it was strongly acid in reaction, gave no precipitate with potash, and if first neutralised none with

potassium iodide. On evaporating, mercuric chloride was certainly left, due to the expulsion of hydrocyanic acid by hydrochloric acid as concentration proceeded. It, therefore, appears that the reaction occurs in the manner indicated by *Scheel*, but it is never complete by a single treatment with hydrocyanic acid, for it ceases directly the proportion of hydrochloric acid present rises to a certain limit. It can be aided by neutralising the acid as it forms, and hindered by the addition of an initial dose of acid. Pharmaceutical preparations containing hydrocyanic acid and calomel, though apt to become discoloured from this reaction, are not more dangerous on account of the mercuric cyanide formed than they are from the corresponding quantity of hydrocyanic acid originally present.

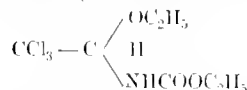
—B. B.

#### Ouabaïo—The Poison of the Somalis. Cathelineau. J. Pharm. Chim. 1889, 436.

ALONG the whole of the west coast of Africa, especially in the country of the Somalis, occurs a tree used by the natives for the preparation of their arrow-poison, and called by them Ouabaïo. Its wood is yellowish white, very hard and dense, odourless, but possessing a strong bitter taste. *Arnaud* has isolated from it (this Journal, 1889, 211; 1888, 586) Ouabaïo,  $\text{C}_{30}\text{H}_{40}\text{O}_{12}$ , which has a physiological action similar to that of strophanthin,  $\text{C}_{31}\text{H}_{45}\text{O}_{12}$ , but which is twice as toxic, the poisonous dose being about 0.2 mgrm. per kilo. of body-weight (this Journal, 1888, 765).—B. B.

#### Sommal. E. Ritsert. Pharm. Zeit. 1889, 674.

RADLAUER has patented and brought upon the market a soporific to which he assigns the formula—



and the name *Sommal*. He prepares it by heating a mixture of equal parts of chloral hydrate, urethane and alcohol to 100° C. The author disputes the possibility of such a mode of preparation; there is no evidence of any reaction occurring when these bodies are mixed cold, no chloral ethylate is formed at 100° C., and chloral urethane is decomposed at 100° C. Although chloral hydrate will give no reaction under these conditions, chloral will, and one obtains chloral alcoholate or chloral urethane according to the order of addition of the reacting substances, the product being in one case unattacked by the subsequent addition of alcohol, and in the other of urethane. "*Sommal*" is therefore nothing but a mixture of chloral hydrate, urethane and alcohol.

—B. B.

#### PATENTS.

##### Improvements in the Preservation of Peroxide of Hydrogen. C. T. Kingzett, London. Eng. Pat. 15,993, November 5, 1888. 4d.

A SMALL quantity, say 1 per cent. of either of the following substances, is added to the solution of hydrogen peroxide: alcohol, acetic acid, chloral, chloroform, or glycerin, alcohol being preferred.—J. M. H. M.

##### Apparatus for the Production of Ozone. E. Fahrig and H. E. Billing, London. Eng. Pat. 16,139, November 7, 1888. 6d.

THIS invention relates to the production of ozone by subjecting air to electrical action in the following manner:—By means of a pump, blower, or fan a current of dry and purified air is passed in thin layers between a number of corrugated plates. "Each plate is made of non-conducting material coated on one surface with metal foil, and is connected to one of the terminals of the secondary coil of an inductor, the next plate in order being connected to the other terminals of a dynamo-electric machine producing alternating currents."—O. J. S.

*Improvements in Dental Anodynes or Obtundents.* R. I. Hunter, Norfolk, U.S.A. Eng. Pat. 17,627, December 3, 1888. 4d.

THE compound consists of an aqueous solution containing chloral, cocaine, arsenic, creosote, and carbolic acid.  
—E. E. B.

*The Manufacture of a Substitute for Musk.* E. Schnauffer and H. Hupfeld, Frankfort, Germany. Eng. Pat. 18,521, December 18, 1888. 4d.

THIS invention relates to the manufacture of a substitute for musk. Aromatic hydrocarbons such as benzene, toluene, or xylene, are converted into isopropyl, isobutyl, or isoamyl derivatives and then nitrated, and the products of this reaction are poured into water, whereupon a reddish-brown oil separates out, which is washed several times in alkaline water. For instance, if metaxylene be heated with isabenzylalcohol and chloride of zinc under pressure, and the resulting dimethylisopropylbenzene subsequently treated with nitric acid, an oil,  $C_{12}H_{17}NO_2$ , is obtained, which, in a concentrated condition possesses a sweet smell, whilst in a dilute alcoholic solution it gives off a penetrating and enduring musk-like odour.—O. J. S.

*Improved Apparatus for Percolating and Expressing Tinctures, Fluid Extracts, or Concentrated Infusions.* F. Tebbutt, London. Eng. Pat. 486, January 10, 1889. 6d.

THE apparatus consists of a cylindrical vessel, having a perforated false bottom and provided with a "screw or press" by means of which the ingredients to be treated may be compressed between this false bottom and a perforated plate.—O. H.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

*Development with Quinol.* E. Himly. Phot. Correspondenz, 26, 152—159.

POTASSIUM metasilphite preserves the quinol solution better than sodium sulphite alone, but it is not advisable to reduce the quantity of the latter since it exerts a beneficial effect on the colour of the image. Four parts of quinol in 1,000 is the smallest quantity which can be used with good results. The addition of 2 to 3 per cent. by volume of a solution of sodium hydroxide (1:8) to the ordinary solution of quinol and an alkaline carbonate reduces the time required for development, and gives a considerable increase in the amount of detail in the image, and a more brilliant negative generally. It is also an advantage when developing positive images on glass or paper. Potassium metasilphite acts as a restrainer of considerable power when used with either quinol or pyrogallol, and in the latter case the addition of a small quantity of the dilute sodium hydroxide solution to the ordinary developer containing sodium or potassium carbonate is a distinct improvement. In all cases the time required for the first appearance of the image and for the completion of development was considerably less with pyrogallol than with quinol.

Solutions recommended are as follows:—

A. Quinol, 10 parts; potassium metasilphite, 4 parts; water, 250 parts.

B. Sodium or potassium carbonate, 10 parts; sodium sulphite, 5 parts; water, 100 parts.

Mix 10 parts of A with 50 to 75 parts of B and 25 to 50 parts of water.

The quinol solution may contain 25 to 50 parts of sodium sulphite, with or without the potassium metasilphite. Instead of the alkaline carbonate solution, 10 parts of the quinol solution may be mixed with 100 parts of water and 10 parts of a solution (C) sodium hydroxide, 5 parts; sodium sulphite, 5 parts, and water 40 parts.—C. H. B.

*Development with Quinol containing Potassium Ferrocyanide.* E. Himly. Phot. Correspondenz, 26, 160—162.

THE addition of potassium ferrocyanide to the quinol developer gives a greater amount of detail with increased contrast in the image, and at the same time reduces the time required for development. The use of potassium bromide is not necessary unless the film has a tendency to fog. The ferrocyanide is best kept in the form of a 30 per cent. solution, which is added in drops as occasion requires.

—C. H. B.

*Gelatinobromide Emulsion prepared with Ammonio-Silver Nitrate.* J. M. Eder. Phot. Correspondenz, 26, 169—171.

TWENTY-FOUR grms. potassium bromide and 25 grms. of Winterthur's gelatin are dissolved in 230 cc. of water in the usual manner, with or without the addition of 8 cc. of a 10 per cent. solution of potassium iodide. Three grms. of citric acid are dissolved in 230 cc. of water, mixed with 30 grms. of silver nitrate, and then ammonia is added until the liquid just becomes clear. Both solutions are heated to 50°, carefully mixed with vigorous agitation, and then heated for 45 minutes in a water-bath at 40° to 45°, or, if greater sensitiveness is required, at 50°. Fifteen grms. of gelatin previously swelled in cold water and melted is added, and the other operations are conducted in the usual manner. The filtered emulsion, before coating the plates, should be mixed with 1 per cent. by volume of a 2 per cent. solution of chromic alum.

The plates prepared in this manner are of moderate sensitiveness, but are free from tendency to fog, and give images of good quality. They are especially suitable for landscape work, and for micro-photography. Greater sensitiveness can be obtained by digesting the emulsion at 60° to 70°, but the quality of the plate is by no means so good.—C. H. B.

*The Acid Fixing Bath.* A. Lainer. Phot. Correspondenz, 26, 273—274.

THE acid fixing bath (this Journal, 1889, 637) is not only advantageous with all alkaline developers on account of its power of removing stains, but also because it exerts a distinct toughening and contractile influence on the gelatin film and thereby reduces the risk of frilling.—C. H. B.

*Development with Catechol and with Paraphenylenediamine.* J. M. Eder. Phot. Correspondenz, 26, 309—311.

*Catechol.*—One part of catechol and 4 parts of sodium sulphite were dissolved in 40 parts of water, and mixed with twice its volume of a solution of 2 parts potassium carbonate in 40 parts of water. Sodium sulphite keeps the solution clear, although it somewhat retards development. Development proceeds much more quickly than with a quinol developer of corresponding composition. Even when dilute to one-third of the strength given, the developing power remained considerable.

*Paraphenylenediamine* gives about the same degree of sensitiveness as pyrogallol or quinol. Development is regular, and the image is grey and soft. A 2 per cent. solution of *p*-phenylenediamine hydrochloride was mixed with 1 to 2 vols. of a 10 per cent. solution of potassium carbonate. Sodium sulphite acts as a very powerful restrainer of development.—C. H. B.

*Development with "Eikonogen."* J. M. Eder. Phot. Correspondenz, 26, 519—521.

"EIKONOGEN" is the sodium salt of amido- $\beta$ -naphthol- $\beta$ -monosulphonic acid. When mixed with alkalis it develops gelatinobromide plates successfully, giving soft and well-graduated images, free from fog. It is best to keep the eikonogen and the alkali in separate solutions. For ordinary purposes the following solutions may be used:—(A.) 50 grms. of Eikonogen dissolved in a solution of 200 grms. of sodium sulphite in 3,000 cc. of water. (B.) 150 grms. of crystallised sodium carbonate in 1,000 cc. of water.

Three parts of A are mixed with 1 part of B, potassium bromide being added if a restrainer be needed. For brief exposures 5 grms. of potassium carbonate and 10 grms. of sodium sulphite are dissolved in 150 cc. of water, to which are added 5 grms. of Eikonogen.—C. H. B.

*Deterioration of Gelatinobromide Plates.* E. Vogel. Phot. Mittheilungen, **26**, 9—11 and 126—128.

THE black rims which appear on the development of dry plates which have been kept for some time are due to the use of impure packing paper. In order to test the latter it may be boiled with water, and the solution mixed with a few drops of silver nitrate solution and again boiled, when a brown or black precipitate of silver sulphide will be formed. The original solution may be mixed with starch paste and iodine solution, when it will be found that several drops of the iodine solution are required to produce a blue colouration. Neither of these tests always give satisfactory results, and it is better to enclose a piece of the paper between a sensitive film and a sheet of perfectly clean glass in an ordinary printing frame in the dark room for about a week, and then develop the sensitive film as if it had been exposed to light. The following results were obtained:—*strongly-marked action*, common black paper, glazed common black paper, smooth black paper, ordinary note-paper; *weak action*, ordinary photographic paper, smooth yellow packing paper, filter paper; *no action*, Swedish filter paper, tissue paper, and tissue paper soaked in paraffin. The effect seems to be due to the evolution of some reducing gases from the paper, for it is not prevented by the insertion of porous pure paper between the impure paper and the sensitive film.—C. H. B.

*Gelatinobromide Plates with fine Grain.* O. Lohse. Phot. Mittheilungen, **26**, 25—26.

SILVER bromide is appreciably soluble in gelatin solution, forming an opalescent solution which is reddish by transmitted light and forms an almost perfectly transparent layer if allowed to dry upon glass. In contact with ammonia vapour the silver bromide separates in a granular form, and in both this and the ordinary process of making the emulsion, the fineness of the grain depends on the duration of the time of action of the ammonia. An emulsion with a very fine grain, of comparatively low sensitiveness, but especially suitable for micro-photography and similar purposes, can be obtained in the following manner:—Two solutions are prepared (A) by dissolving 2 grms. of gelatin in a small quantity of water at the lowest possible temperature, and then adding 1 gm. of silver nitrate dissolved in a small quantity of water, the total bulk of liquid being 50 cc.; and (B) by dissolving 2 grms. of gelatin and 0.6 gm. of ammonium bromide in 50 cc. of water. The two solutions are mixed whilst warm, and, after vigorous agitation, poured into 200 cc. of alcohol. The precipitate is washed with cold water until all soluble salts are removed, melted at a low temperature, and diluted to 100 cc. Plates are then coated with it in the ordinary manner, and, after being dried, are immersed in a mixture of equal parts of absolute alcohol and water, exposed to the action of ammonia vapour for not more than a minute, and again dried.—C. H. B.

*Sodium Bichromate in Photo-mechanical Printing Processes.* J. Husnik. Phot. Mittheilungen, **26**, 238.

SODIUM bichromate can be used in photo-mechanical processes for all purposes for which the potassium salt has hitherto been employed. It is much cheaper, can be used in much more concentrated solutions, and has not the same tendency to crystallise in the films. It has a lower molecular weight, but since it always contains water, there is no need to make any alteration in the weight of bichromate given in the usual formulae.—C. H. B.

*Orthochromatic Photography with Gelatin Plates.* C. H. Bothamley. Phot. News, **33**, 563—565.

IT is well established that a higher sensitiveness to the less refrangible rays is obtained by treating ordinary gelatinobromide plates with an ammoniacal solution of an Eosin dye or Cyanin than by adding these substances to the melted emulsion or to the materials before emulsifying, although in the first case the plates are more liable to deteriorate if kept for some time before being used. In order to ascertain the best method of working the bath process the author has examined the various modifications which have been proposed, the special points investigated being the necessity or advantage of a preliminary bath of dilute ammonia, of adding alcohol to the sensitising bath, and of washing the plate after treatment with the dye solution. The sensitiveness of the plates to the less refrangible rays was determined by the method previously described (this Journal, **7**, 767). All experiments were made in duplicate with plates prepared by different methods, and the results are given in the form of tables. The selective sensitiser (*loc. cit.*) employed was Erythrosin, and the conclusions arrived at are:—(1) Alcohol up to 10 per cent. has no influence whatever and may be dispensed with in all cases where the dye is soluble in water; alcohol in larger proportion produces a distinct decrease in sensitiveness. (2) With a concentration of the dye solution not exceeding 1 in 5,000 the washing of the plate after immersion in the bath is unnecessary. (3) A preliminary bath of dilute ammonia is also unnecessary. With the particular plates used no increase of sensitiveness was gained by increasing the concentration of the dye from 1 in 10,000 to 1 in 5,000, but if the plates have been prepared with hard gelatin or have been treated with chrome alum it is advisable to use the stronger bath or to increase the time of immersion. Ives floods ordinary dry plates with an alcoholic solution of the dye (1 in 1,750), allows the alcohol to evaporate, and then washes with water; but it was not easy to see why this method should give results different from those obtained by simple immersion in a dilute aqueous solution of the dye, and photometric measurements confirm this supposition. The author was unable to obtain satisfactory results by using Cyanin in this manner.

The best results, as regards sensitiveness to the less refrangible rays, are obtained in the following simple manner: Dust the plate, and immerse it for two or three minutes in a solution containing—

	Parts.
Dye solution (1:1,000).....	1 to 2
Ammonia (1:10) .....	1
Water.....	up to 10

Allow the plate to drain for some time, place the lower edge on filter paper, to remove the ridge of liquid which collects there, and dry in a pure atmosphere in the dark.

Some experiments on the sensitising action of rhodamin were made in a similar manner. The sensitising effect for the less refrangible rays varies considerably, according to the manner in which the original plates were prepared, but is always less than that of Erythrosin.

The results of a series of experiments on the use of orthochromatic plates for landscape work are also described, and the considerations involved in the use of a transparent yellow screen to reduce the intensity of the blue and violet rays are discussed.—C. H. B.

*The Substitution of Gelatin for Collodion in Photography.* J. Franklin Inst. **98**, 233, 234.

THE Committee on Science and the Arts of the Franklin Institute has awarded the "John Scott Legney Premium and Medal" to Dr. Maddox for his invention of the gelatinobromide process as published by him in the *British Journal of Photography*, September 8, 1871, in the following terms: "Originally thirty grains of gelatin were swelled in cold water, then dissolved by heat, four drachms of pure water and two drops of aqua regia being added. To this solution eight grains of cadmium bromide and fifteen grains of silver nitrate were added, forming a fine milky emulsion of silver bromide. Without further treatment this was spread upon glass plates and dried. The plates were tested by

exposing them beneath negatives and gave a faint but clear image when developed with a plain solution of pyrogallie acid: intensification with pyro and nitrate of silver followed.

\* \* \* So far as can be judged, the process seems worth more carefully conducted experiments, and if found advantageous, adds another handle to the photographer's wheel."—B. B.

*Formic Aldehyde and its Compounds applied to Photography.* J. Schnauss. Chem. Zeit. 13, 1633.

THE well-known property of the aldehydes to reduce silver to the metallic state from an ammoniacal solution of silver nitrate, especially when warmed, suggests their application to photography. But their use has heretofore been impractical owing to their strong odour, their insolubility or difficult solubility in water, and the facility with which they oxidise on exposure to the air. The aldehydes, however, and in particular formic aldehyde, form easily soluble compounds with the alkaline bisulphites, and these salts may be used with advantage, in many ways, in photography. A process in which they are employed has been patented by Schwartz and Mercklin.

Sodium oxymethyl sulphonate (formic aldehyde sodium bisulphite) is valuable (particularly in conjunction with an alkaline carbonate) both as an addition to ripening gelatin-emulsion—the ripening of which it accelerates, imparting vigour and clearness to the negative—and as an addition to the developer, be it ferrous oxalate, pyrogallol, or quinol (hydroquinone), it which case it acts not only as a preservative, owing to its affinity for oxygen, but also as an accelerator, and preventer of "veiling" or "fog."

These compounds have the same kind of effect although in less degree, when they are used in very dilute solution, together with a carbonate of the alkali or of ammonium, as a bath for exposed plates before developing.

If finished dry plates be plunged in a bath of the alkaline solution of the aldehyde salt, and then dried and exposed, results are obtained similar to those produced by adding the aldehyde salt to the emulsion before ripening.

The action of formic aldehyde (and its compounds) depends no doubt upon the energy with which it absorbs or acts as a transferer of bromine and iodine. This property can be demonstrated as follows:—When a solution of iodine is partially precipitated with silver nitrate, the precipitate is of a brown or grey colour owing to the retention of free iodine; but if formic aldehyde be previously added the precipitate will be of a pure yellow-white colour. Again, when caustic magnesia is treated with iodine solution, no decolouration takes place unless upon long standing, but on adding formic aldehyde the colour of the iodine is at once discharged and magnesium iodide goes into solution.

The oxymethyl sulphonates, e.g., the sodium or the silver salt, show an even greater activity as absorbents of bromine and iodine. The sodium salt decomposes silver bromide forming silver oxymethyl sulphonate, silver sulphite, sodium bromide, and formic aldehyde. When finished dry plates are treated with a solution of the sodium salt, the small quantity of formic aldehyde that is formed does not all volatilise on drying, but a portion of it remains in the film as fixed paraformaldehyde, possessing the same photochemical properties as the volatile aldehyde. Possibly also when formic aldehyde salts act on silver bromide in an alkaline solution bromoform is formed, the free formic and hydrobromic acids being taken up by the alkali of the bath, tending to the production of clearness in the pictures.

—H. S. P.

#### PATENTS.

*Improvements in Colour Photography.* A. H. Cros, Paris, France. Eng. Pat. 9012, November 8, 1888. 8d.

AN apparatus for obtaining simultaneously three negatives of an object by means of the rays which have passed respectively through orange, green, and violet glass. These negatives are intended for use in the Cros method of producing coloured pictures by photography. The apparatus, of which drawings are given, consists of three cameras placed side by side and enclosed in a larger camera. Each

of the internal cameras is provided with a plate holder and a coloured glass screen. One lens only is used, and inside the larger camera is a rotating disc with transparent sectors and opaque reflecting sectors, so arranged in conjunction with a plane mirror that when the disc is rotated the rays from the lens are admitted to the three cameras in rapid alternation.—C. H. B.

*Improvements in the Production of Films Sensitive to Light and of Materials for Assisting or Continuing the Effect of Light on Photographic Films.* A. F. Y. Schwarz and F. H. Mercklin, Hanover, Germany. Eng. Pat. 741, January 15, 1889. 6d.

FORMALDEHYDE, paraformaldehyde, or the compounds of formaldehyde with the acid sulphites of the alkalis, silver or iron are added to the gelatinobromide emulsion either just before coating or before ripening, in the proportion of 1 to 2 per cent. of the amount of haloid silver salt present. The prepared plates may be bathed for two or three minutes in a solution of 1 to 2 parts of these compounds in 1,000 parts of water and then dried. A few drops of the dilute solution of any of the alkaline salts may be added to the developing solution. It is claimed that in this manner a much higher degree of sensitiveness is obtained.—C. H. B.

*Improvements in or relating to Mixtures or Solutions for Developing or Fixing the Latent Images on Photographic Plates or Sheets.* W. P. Thompson, Liverpool. From C. Spiro, New York, U.S.A. Eng. Pat. 13,523, August 27, 1889. 6d.

THE developing solution is mixed with some yellow or orange aniline dye, such as Aniline orange, or some other colouring matter which absorbs all the rays of high refrangibility. When the plate has been placed in the coloured developer in the dark room, it may be brought out into daylight and the process of development watched, but of course the plate must not be lifted out of the liquid. When development is complete the plate is quickly transferred to the dish containing the fixing solution, which is then covered over. The fixing solution may be coloured with the same orange dye.—C. H. B.

## XXII.—EXPLOSIVES, MATCHES, Etc.

*Perforated Cake-Powder for Ordnance.* G. Quick. Proc. Inst. Civ. Eng. 1888—89, 107.

THIS paper describes a new modification of powder, by means of which it is proposed to obviate some of the disadvantages of the powders now in use for military purposes. Among such disadvantages are mentioned the large amount of heated and sometimes fused residue left in the gun, the irregularity of the velocity and pressure produced, and the erosive action upon the bore of the gun. Although the brown or cocoa powder gives better results in general, in regard to moderate pressure, than black powder, yet the amount of residue which it leaves is greater. The author claims that his method of forming the "propelling material" is equally applicable either to the new chemical propelling materials, or to the old varieties of powder.

After considering the relation between the pressure and expansion of the gases produced by the combustion of powder in the bore of the gun, the conclusion is arrived at that the best results are to be looked for by using a slow burning powder rather than by increasing the air-spacing of the charge. The rate of burning of a powder is dependent upon various modifications during manufacture, and upon the pressure of the atmosphere of fire to which it is exposed. With most powders the area of combustion surface decreases

as the time increases, while the object to be aimed at is that the pressure shall very rapidly reach its maximum, which shall be maintained constant till the completion of combustion.

By making the surface increase as combustion proceeds the pressure can be made approximately constant during the time combustion is proceeding.

When a cylindrical block of powder, pierced by a hole, is burnt, the surface will decrease as time increases; but if, in addition to the centre hole, a number of smaller ones are ranged round it, then it will be possible to prevent the surface from diminishing as combustion proceeds, and it may even be increased.

The form of powder proposed by the author, and of which illustrations accompany the paper, is that of circular discs with one central circular perforation, also circular in section. Alternate quadrants of the cake are in relief, so that when two cakes are placed together the raised quadrants of the one fit into the sunk ones of the other and the two cakes are, in a manner, locked, so that all the perforations are in line parallel with the axis through the whole length of the cartridge. The quadrants are dished or somewhat hollowed out towards the centre, to permit of the flame readily reaching the whole surface of the cakes. In forming a cartridge a number of these cakes are united by means of a waterproof cement, which is highly combustible and leaves only a very small amount of residuum.

The author believes that the pressure generated in the bore of a gun charged according to his system may be so regulated that it would be possible to discharge shells containing high explosives, such as dynamite.

Although the calculations are made on the assumption that the perforated powder cakes will not break up during the period of combustion, yet it is not expected that this will be found mathematically true in practice. The experiments which are referred to show, however, that the assumption is true enough for all practical purpose.

The author summarises the conclusions which he has arrived at as follows:—1. The pressure should, if possible, be maintained at its maximum till the projectile has travelled some considerable distance up the bore. 2. This can be most satisfactorily obtained by using a powder whose surface of ignition increases as combustion proceeds, or remains practically uniform. 3. That a comparatively low maximum pressure should be adopted if the best results, in the fullest sense of the term, are to be obtained. 4. That the density of the powder should be very high, so as to secure uniformity of combustion, and that the initial ignition and terminal combustion surfaces shall be large enough to secure a sufficiently rapid combustion of this very dense and slow-burning powder, so that none may be blown out of the gun in an unconsumed state. 5. That the air-spacing should be reduced to the lowest possible amount compatible with the due ignition of the charge, and with a sufficient channel space to allow of a fair flow of the powder gas direct to the base of the projectile as soon as the charge is ignited. 6. That the powder charge should be so arranged that the whole of it should be simultaneously ignited, in order that each and every element (cake) should be surrounded by an atmosphere of fire equal in pressure in all directions.

The results of exhaustive experiments are given in tabular form, which confirm the advantages claimed for the "Quick" powder over ordinary service powder.—C. N. H.

## PATENTS.

*Improvements in the Manufacture or Treatment of Explosives to better Control Combustion, or Prevent Detonation.* W. R. Hodgkinson, Blackheath. Eng. Pat. 17,456, November 30, 1888. 6d.

In order to render gun-cotton, nitro-glycerin, and other similar bodies less rapidly combustible, and to lessen the tendency to detonate, the inventor mixes certain compounds of aniline, toluidine, xyldine, cumidine, or of other similar amido compounds with the explosive in such proportions as have been found by experience will reduce the rate of

explosion or combustion to the extent required. The compounds of these bases preferably used are their salts with formic, acetic, and oxalic acids, and their derivatives known as formyl, acetyl, and oxetyl compounds. These compounds may be dissolved in nitro-glycerin, but to impregnate gun-cotton with these substances they must first be dissolved in some solvent, such as alcohol, and the dry gun-cotton caused to absorb the solution; the solvent is afterwards allowed to evaporate spontaneously.—W. M.

*An Improved Manufacture of Nitro-Glycerin and Dynamite.* H. Ritter von Dahmen, Vienna, Austria. Eng. Pat. 17,631, December 3, 1888. 4d.

This invention consists in first adding to pure glycerin 5 to 10 per cent. of a mono or higher nitrate of a hydrocarbon of the benzene or toluene group, and then treating this mixture with the usual acids, nitrogen or air being continuously blown through during the reaction. It is asserted that nitro-glycerin produced in this way will not freeze at  $-25^{\circ}\text{C}$ , and is also less liable to explosion by concussion than ordinary nitro-glycerin.—W. M.

*Improved Apparatus and Process for the Manufacture of Nitro-Glycerin.* H. H. Leigh, London. From La Compagnie Forcite, Paris, France. Eng. Pat. 13,907, September 3, 1889. 8d.

This apparatus consists of a horizontal wooden tank, the cover of which is provided with a sight hole and a chimney for the escape of fumes.

In this tank is placed a smaller leaden tank provided with an inclined gutter-shaped bottom, and which can be closed by the cover of the outer tank. Serpentine leaden pipes are placed at different levels within the leaden tank, and are separately supplied with water, so that all or any particular layer of the liquid can be cooled. Perforated pipes are provided for the admission of compressed air for the purpose of rousing and cooling the contents of the tank.

The main feature of this invention consists in a small pot with perforated sides which stands on the bottom of the inner tank, and into which two pipes deliver, the one glycerin in the form of spray, the other mixed acids. The process of manufacture is conducted by first filling the leaden tank about three-quarters full with the mixed acids. The glycerin is then introduced along with further acid, and the nitric acid is thus effected in the pot, and in the presence of strong acid. The nitro-glycerin and excess of acid then disperse into the surrounding tank.—W. M.

*Improved Machinery for the Manufacture of Explosive Cartridges.* H. H. Leigh, London. From La Compagnie Forcite, Paris, France. Eng. Pat. 13,908, September 3, 1889. 8d.

This invention consists in a novel combination of screws whereby the cartridge material is driven through the machine and delivered in a condition of uniform consistency. The main feature is a screw acting under the feeding piston, and two archimedean screws adapted to deliver two rods of cartridges simultaneously.—W. M.

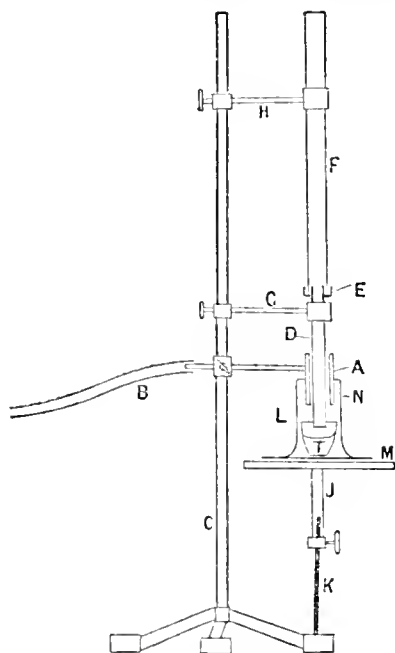
## XXIII.—ANALYTICAL CHEMISTRY.

*Apparatus for Evaporating Liquids in Small Vessels.* W. Hempel. Ber. 22, 2479—2481.

The apparatus is shown in the figure. A is a large inverted Argand lamp fed through the tube B, which may be clamped to the support at any height. The lamp is of porcelain or soapstone. It is fixed to the metal arm by a paste composed



of manganese dioxide and silicate of soda. A porcelain tube passes through the centre of the Argand lamp, on the top of which a groove, E, is formed. The tube is supported by the arm G. F is a glass chimney which rests in the groove E. M is a platform, which is adjustable to any height. L is an inverted beaker with the bottom removed, which



regulates the supply of air by being raised or lowered on the stand M.

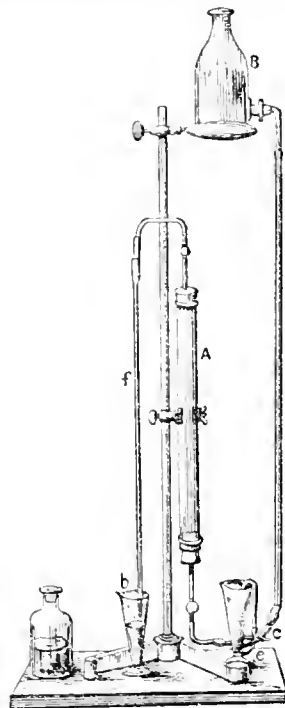
To evaporate a liquid in the crucible T, the porcelain tube D, and glass tube F, are so placed, that the lower end of the former rests in the centre of the Argand lamp. The gas is then lighted, and the gas regulated to give a small flame. The crucible is placed on the support M, with a sheet of asbestos between the two, and upon this the open beaker. The support is then raised so as to rest about 7 mm. below the flame of the Argand. The porcelain cylinder is now lowered almost to the surface of the liquid in the crucible. The decomposition of clays and fluorides may be performed with the greatest readiness, as no portion of the apparatus which comes in contact with acid fumes is attacked.—J. B. C.

*Lecture Experiment in Agricultural Chemistry.* M. Müller.  
Zeits. f. angew. Chem. 1889, 501—502.

THE sketch shows an arrangement of apparatus for demonstrating the absorbing power of earth for soluble salts in dilute solution.

The method is one of upward filtration, which allows of control over the rate of flow of the solution, and ensures that the earthy material is thoroughly permeated. The glass tube A is about 75 cm. long and 4 to 5 cm. in diameter, closed at each end with a perforated rubber cork. A small layer of glass wool at either end serves to keep the earth in its place. The solution is placed in B, and its flow is regulated by a screw clamp at c. A glass T-piece at e serves to deliver a sample of the unfiltered solution, whilst the filtered liquid runs out at b. The best kind of material for the experiment is a sandy earth, poor in humus substances, and unmanured. It should be allowed to dry in the air, and sifted from coarse particles. The solutions used must be dilute: for example, a litre may contain 1.5 grm. of potassium carbonate or crystallised sodium phosphate, or 0.1 grm. of ammonium sulphate. Litmus, ammonium molybdate solution, or Nessler's test may be

used in each case respectively to render the change in the solution visible to an audience. The solution should pass slowly through the soil. A striking experiment may be



made by filling the tube with turf and passing through it some foul refuse water, when a disappearance of the smell will show the distinctive absorptive powers of the soil.

—G. H. B.

*Technical Gas Analysis.* W. Thörner. Zeits. f. angew. Chem. 1889, 641—650.

THE author recommends the use of only such burettes as are surrounded with a cooling jacket, such jacket being connected with a reservoir containing water of the temperature of the room; it is much more satisfactory to have the jacket fused on to the burette and not attached merely by corks or rubber stoppers, which in time are very apt to leak.

In the examination of *generator gas*, *water gas*, *coal gas*, &c., after removal of carbon dioxide and hydrogen in the usual manner, the heavy hydrocarbons of the ethylene and benzene series are absorbed by means of fuming sulphuric acid. The pipette necessary may be fitted with ground glass capsules, and the upper bulb should contain pieces of glass. Absorption of carbon monoxide is easily carried out by means of a fresh hydrochloric acid solution of cuprous chloride. Hydrogen can then be removed by means of palladium asbestos. If the gas still contain methane, as is the case in generator gas, and especially coal gas, the dilution caused by addition of atmospheric air becomes inconvenient; the accompanying Figure (1) shows a very handy way of introducing gases necessary for combustion. The apparatus is of exactly the same size as the usual absorption apparatus and may be easily connected with the measuring burette; one limb of the U-tube is divided into 0.5 cc. and contains two platinum electrodes, the other limb contains one, so that at will oxygen, hydrogen, or electrolytic gas may be obtained and measured and then drawn into the gas burette. Fig. 2 shows a very convenient form of apparatus for burning methane with oxygen; the wires *a a* are of silver, and are thick, so that fracture of the bulb by heating is avoided; these wires are connected inside the bulb by a thin platinum wire, which, when heated by the electric current, brings about the combustion of the

methane. Combustion with electrolytic gas may be carried out in a similar apparatus, an elongated thick walled cylinder taking the place of the combustion bulb, the usual pair of

Fig. 1.

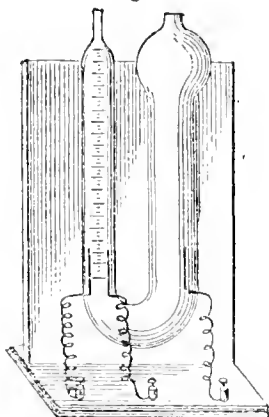
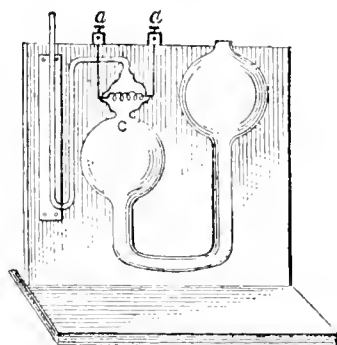


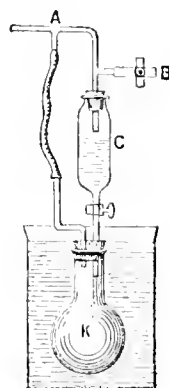
Fig. 2.



platinum wires taking the place of the silver and platinum wires, and a rubber stopper closing the further bulb.

For the *valuation of soda*, or determination of carbon dioxide in minerals, where this may be liberated in the cold by acids, the following apparatus (Fig. 3) serves well.

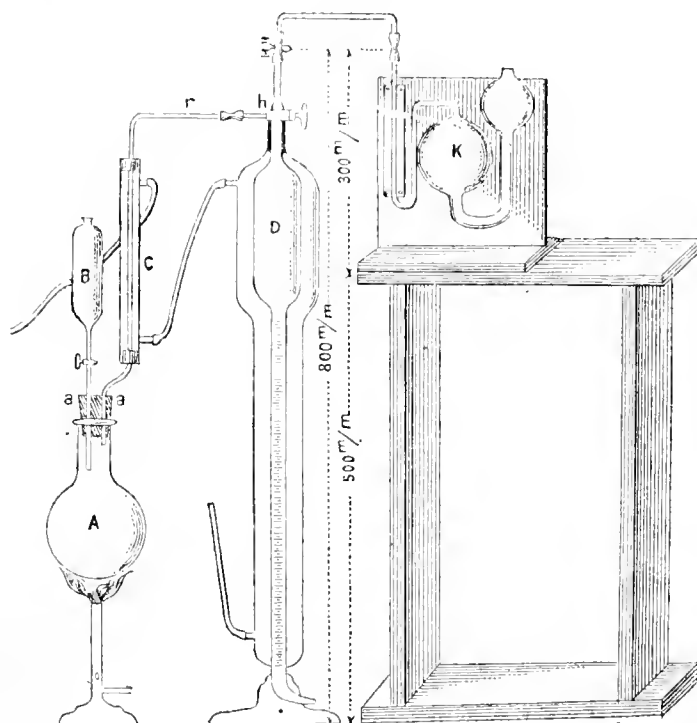
Fig. 3.



The flask K has a capacity of 100 cc.; into it is weighed a quantity of the dry substance, about 0.25—0.5 gm. The small 50 cc. funnel C contains hydrochloric or sulphuric acid, saturated with carbon dioxide by addition of a small quantity of sodium bicarbonate. During the experiment the flask is kept cool as shown. A is connected with the gas burette and before the experiment levelling is performed by aid of the clip B. Now, by allowing the acid to flow from C into K, the carbon dioxide is evolved, and its quantity ascertained by the volume of gas collected in the burette.

To determine the quantity of *carbon dioxide dissolved or in combination in beer, wine, &c.*, the sample is boiled with hydrochloric or sulphuric acid in a long-necked flask, the neck of the flask being surrounded by a condenser; the gases evolved are received in a collecting vessel, being

Fig. 4.



finally completely displaced from the boiling-flask by running in water; from the collecting vessel they are transferred to a burette and the  $\text{CO}_2$  is determined in the usual way.

To determine volumetrically the amount of carbon in iron and steel the apparatus shown in Fig. 4 may be conveniently used. 0.5 gram of pig iron, spiegeleisen, &c., or 2.5 grms. of steel are introduced into a flask of 150 cc. capacity, along with 10 cc. of a saturated solution of copper sulphate which has been previously filtered through asbestos; the whole is shaken several times, and, after about five minutes, are added for every gramme of iron 5 cc. of a 100 per cent. chromic acid solution. The ground-glass stopper *a*, carrying the condenser *C* and tap-funnel *B*, is inserted, and from *B* about 120–130 cc. of sulphuric acid (sp. gr. 1.594) is slowly run in until the flask is nearly full. The condenser is then immediately connected, by means of the tube *r* and three-way cock *h*, with the burette *D*, which is surrounded, as shown, with condenser. The contents of the flask are next brought gradually to the boil, and are kept boiling for about half an hour. At the end of this time after withdrawal of the flame the whole of the gas may be driven from *A* by running in water from *B*, and finally so much air is admitted that the volume reaches nearly 100 cc. The  $\text{CO}_2$  is then determined by absorption in *K*. From the quantity of  $\text{CO}_2$  of course the amount of carbon may be calculated, correction being made for temperature and pressure. Comprehensive tables giving the carbon corresponding to different quantities of  $\text{CO}_2$  at various temperatures and pressures are given in the original article. As the gas volumes are often small and require to be very accurately measured, the vessel *D* has been narrowed below, the narrow portion showing readings from 75 to 100 cc. This method of determining *C* in iron gives results very closely agreeing with those obtained gravimetrically. The tables above named are of course of the greatest use in all cases where  $\text{CO}_2$  is volumetrically determined.—T. L. B.

*Continuous Recording Apparatus for the Detection of Sulphuretted Hydrogen in Coal Gas.* Ledig. *J. T. Gasbeleucht.* **32**, 925–928.

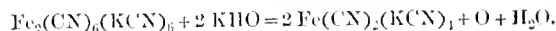
THE apparatus consists of a strip of paper, dipped in lead solution, driven by the counting mechanism of a small gas-meter, so that a new portion is continually exposed to the action of the gas to be tested, which is kept streaming through the vessel containing the paper strip, at the rate of 50 litres an hour. The strip enters and leaves the atmosphere of gas by mercury traps, and only exposes a length of 2 cm. at any given instant, and travels that distance in one hour. Thus a continuous record of the purity of the gas in respect of sulphuretted hydrogen is obtained.—B. B.

*A New Application of Potassium Ferrieyanide.* G. Kassner. *Chem. Zeit.* **13**, 1302, 1338.

POTASSIUM ferrieyanide in alkaline solution is well known as a useful oxidising agent. It will doubtless also in course of time be used for bleaching of yarn and textile fabric when the price is more moderate.

When peroxide of hydrogen is mixed with an alkaline solution of ferrieyanide a lively, often violent, evolution of oxygen takes place, which may be represented by the following equation in two phases:—

I.



II.



This reaction can be turned to good account as a means of obtaining quickly a regular stream of pure oxygen gas. An undoubtedly pure gas is obtained, which is not the case with potassium chlorate.

The author also considers it preferable to the method of Gohring (*Chem. Zeit.* **13**, 660; also this Journal, 1889, 84), by using hydrogen peroxide and manganese dioxide, or

that of Lindner (this Journal, 1885, 508), by means of sulphuric acid, potassium permanganate, and hydrogen peroxide.

The author recommends 58 grms. of potassium ferrieyanide with 100 cc. of 3 per cent. hydrogen peroxide, which would yield two litres of pure oxygen.—D. A. S.

*Double Sulphocyanides of Iron.* G. Krüss and H. Morah. *Ber.* **22**, 2061–2064.

THE authors have prepared the following double sulphocyanides:—

$\text{Fe}(\text{CNS})_3 \cdot 9 \text{ KCNS} + 1 \text{ H}_2\text{O}$ .—The salt is obtained by adding the calculated quantity of potassium sulphocyanide to a neutral solution of ferric sulphocyanide. The salt is insoluble in dry ether. In moist ether it is decomposed into its constituents, potassium sulphocyanide, which is insoluble, and the ferric salt which dissolves with a red colour.

$\text{Fe}(\text{CNS})_3 \cdot 3 \text{ KCNS}$ .—The salt is prepared in the same manner as the above, using 3 molecules of the potassium salt to one of the ferric salt. It is very hygroscopic. The colour is more orange than the above double salt, and has no green metallic appearance. The dark red colouration, which ferric salts give with potassium sulphocyanide, is due to the salt containing 9 molecules of  $\text{KCNS}$ , which must therefore be added in excess.—J. B. C.

*Qualitative Analysis of the "Iron Group."* F. Mayer. *Ber.* **22**, 2627–2630.

THE author uses sodium acetate for the separation of zinc and manganese from aluminium and iron, the latter being precipitated completely on boiling as basic acetates. If chromium is present it is necessary to have about 5 atoms of iron as ferric salt to 1 of chromium to ensure complete precipitation of this metal, otherwise some chromium is liable to remain in solution. The separation is performed as follows:—The ammonium sulphide precipitate containing chromium is dissolved in dilute hydrochloric acid, oxidised with nitric acid. This solution is tested for iron, and then ferric chloride and sodium acetate added and a sample boiled and filtered. The filtrate, on adding hydrogen peroxide and caustic potash, should give no yellow colouration, otherwise more ferric chloride must be added to the original solution. This solution should not be boiled until an excess of iron is present. The precipitate and filtrate are then treated in the usual way.—J. B. C.

*On the Electrolytic Estimation of Antimony.* A. Leerenier. *Chem. Zeit.* **13**, 1219.

IN Classen's process for above, hydrogen gas at rate of .5 to 2 cc. per minute is passed. The author finds that 5 cc. per minute may be passed without impairing the accuracy of the estimation, and of course saving much time. (Compare this Journal, 1889, 258.)—D. A. S.

*Electrolytic Separation of Cadmium from Copper.* Smith and Frankel. *J. Franklin Inst.* August 1889.

CYANIDE solutions (see this Journal, 1889, 639) were found very suitable for separating cadmium from copper. The conditions most favourable are essentially the same as those just recorded. The separation is most complete, copper not being found in any of the deposited cadmium, and the filtrate being quite free from cadmium (see page 80).

—W. W. H. G.

*Copper Sulphate as an Indicator in Titration of Zinc and Manganese.* F. Moldenhauer. *Chem. Zeit.* **13**, 1220.

STRIPS of an almost smooth filter paper are drawn through a 4 per cent. solution of copper sulphate and dried quickly.

Decinormal potassium ferrieyanide solution is added to an ammoniacal solution of the zinc to be estimated, until the end reaction is shown by a drop of the solution colouring the prepared paper slightly red. This occurs when 0.1 to

0.2 cc. of ferrocyanide is in excess in 100 cc. of ammoniacal solution. The author prefers to titrate with a warm solution of zinc.

Manganese ore is dissolved in sulphuric acid, filtered to separate lead (which must not be present), the filtrate precipitated with ammonia, filtered, washed, redissolved in sulphuric acid, reprecipitated, and refiltered. The filtrates are made up to a known volume and run from a burette into a measured amount of decinormal ferrocyanide until the red colour is no longer shown by placing a drop of solution on the copper sulphate paper. The author notices the fact that while potassium manganese ferrocyanide (*sic*) contains only one hydrogen equivalent, the precipitated zinc ferrocyanide corresponds to two equivalents.—D. A. S.

*A New Application of Peroxide of Hydrogen in Analysis.*  
C. Hiepe. Chem. Zeit. **13**, 1303.

In analysis of alloys the author recommends adding caustic soda to the solution of the fused mass of sulphides, heating to boiling, and adding hydrogen peroxide till the yellow colour is gone. The polysulphides are oxidised directly to sodium sulphate. No more peroxide than just suffices should be added.

The peroxide must of course be pure, and should be tested. The author mentions he has found in the residue of commercial hydrogen peroxide an inexplicable impurity, namely, magnesium chloride.—D. A. S.

*Müller's Method for Estimating Oxygen in Water.*  
R. Hofelman and K. Barth. Chem. Zeit. **13**, 1337.

MAX MÜLLER described (this Journal, 1889, 993) an ingenious and rapid iodometric method of estimating free oxygen in water. The authors contend that in presence of organic matter false results are obtained.—D. A. S.

*Estimation of Cadmium in Cadmium Dust from Zinc Manufacture.* W. Minor. Chem. Zeit. **13**, 1376—1377.

THE estimation of cadmium in the by-product cadmium dust presents some difficulties. The author suggests the following process:—

Twenty grms. of the cadmium dust are dissolved in hydrochloric acid (avoiding excess of acid), filtered, filtrate diluted to 1 litre; 50 cc. (= 1 gm. Cd dust) are precipitated in the usual way with sulphuretted hydrogen, in a beaker of 500 cc. capacity. The precipitate is filtered, dried at 110°, and weighed. The precipitate is redissolved in hydrochloric acid (having calculated from the weight of the precipitate the exact amount required for solution) and reprecipitated with sulphuretted hydrogen. This treatment is repeated twice. In order to obtain cadmium sulphide of constant weight, it is necessary thus to precipitate it four times. Excess of acid decomposes the sulphide. Washing the sulphide precipitate with alcohol and carbon disulphide is quite superfluous.—D. A. S.

*Technical Analysis of Wolframite.* B. Setlik. Chem. Zeit. **13**, 1474.

THE author recommends that the mineral be first finely ground, and then dried at 110° C. Three to five grms. are taken for analysis, and fused for two hours in a platinum crucible with 3—4 times the amount of sodium carbonate. The fused mass is extracted with water, and the solution is filtered, and the residue washed until the washings are no longer rendered turbid by hydrochloric acid. The solution is now heated and poured into excess of hydrochloric acid, and boiled for half an hour. The precipitate, which consists chiefly of tungstic oxide, but may also contain stannic oxide, silica, and molybdic acid (the last, however, is rarely found), is washed, dried, and burned, after removal from the filter, and weighed. It is then treated with hydrofluoric acid in order to remove the silica.

If tin be present the precipitate, after removal of the silica, is fused for half an hour with cyanide of potassium, extracted with water and filtered from the metallic tin.

The tin is dissolved in ferric sulphate and its amount found from titration with permanganate. To estimate the iron and manganese the residue insoluble in water is dissolved in hydrochloric acid, and the solution is divided into two equal portions, one of which is reduced with zinc and titrated with permanganate, and the other precipitated with sodium carbonate and oxidised with bleaching powder. The precipitate, after being filtered and washed, is dissolved in a solution of ferrous sulphate of known strength, and the excess of ferrous sulphate titrated back. Wolframite generally contains from 60—80 per cent. of tungstic oxide and from 10—20 per cent. of manganese. This process may also be used for estimating the tungstic oxide in schellite.—W. M.

*Ferric Hydrate as an Indicator in the Determination of Zinc.* W. Minor. Chem. Zeit. **13**, 1566.

IF the ferric hydrate is freshly precipitated an excess of two drops of sodium sulphide solution can be clearly recognised, but if kept for some days, however carefully, it becomes granular and colours slowly with the reagent, so that the end reaction is undecided, and 0.5 to 1 cc. excess of sulphide is required to finish. Ferric hydrate prepared from the commercial chloride or nitrate behaves in the same way. The author recommends the preparation of ferric chloride from ferrous ammonium sulphate, and states that ferric hydrate obtained by precipitating the chloride so prepared with ammonia is a more reliable indicator than lead paper or even sodium nitroprusside.—S. B. A. A.

*Determination of Phosphorus in Pig Iron, Steel, &c.*  
G. Weissmann. Chem. Zeit. **13**, 1604—1605.

THE author has compared various quick methods, and recommends the following modification of Reis's process as the most rapid and accurate:—5 grms. of metal are heated with 30—40 cc. of dilute  $\text{HNO}_3$  (1:5) in an Erlenmeyer flask till the violent evolution of gas diminishes; 30—40 cc. of concentrated  $\text{HNO}_3$  (sp. gr. = 1.41) are then added, and when solution is complete, additions of 5 cc. of a solution of potassium permanganate (containing 15 grms. per litre) are repeatedly made till a brown precipitate of manganese peroxide makes its appearance. The solution is cooled, reduced with hydrogen peroxide, made up to 250 cc., filtered, and 100 cc. taken out. According to the richness of the sample 50—100 cc. of a standard solution of ammonium molybdate prepared as below (*a*) are added, the solution heated on a water-bath to 80—90° C. for one hour, cooled and filtered.

The yellow precipitate is washed three or four times with a dilute solution of ammonium molybdate (1:1), dissolved off the filter paper with dilute ammonia (1:6); the solution should occupy about 50—60 cc. 10—15 cc. of magnesia mixture (*b*) are added drop by drop with constant stirring, taking care not to touch the sides of the beaker. The precipitate is allowed to subside for two hours, filtered, washed five or six times with dilute ammonia (1:6), dried and ignited. The weight in grammes  $\times 13.975$  = percentage of phosphorus. The process can be completed in 4—5 hours, and gives very accurate results. An approximate result is more speedily obtained by Meineke's method (this Journal, 1887, 387) of washing the yellow precipitate first with a 10 per cent. solution of ammonium nitrate, and then with water, drying, igniting at a low heat, and weighing as  $\text{Mo}_2\text{O}_3 \cdot \text{P}_2\text{O}_5$ , of which 1 gm. = 1 per cent. of phosphorus.

(*a*.) 630 grms. of ammonium molybdate are finely powdered and gradually dissolved in 750 cc. of ammonia (sp. gr. = 0.91), and 1,200 cc. of water, filtered and stirred into a mixture of 2,500 cc. of nitric acid (1.41), and 4 litres of water. After standing for two or three days the clear solution is drawn off; 10 cc. = .001 gm. of phosphorus.

Otherwise 500 grms. molybdic acid are dissolved in 1 litre of ammonia (0.91) and 1,100 cc. of water; the solution being added to a mixture of 2,500 cc. of nitric acid (1.41), and 4 litres of water as before.

(*b*.) 160 grms. of magnesium chloride ( $\text{MgCl}_2 + 6\text{OH}_2$ ), and 200 grms. of ammonium chloride are dissolved in  $2\frac{1}{2}$  litres

of water; 500 cc. of ammonia (0.91) are added, the mixture stirred, allowed to stand for a few hours, and filtered; 10 cc. = .04 grm. of phosphorus.—S. B. A. A.

*A New Method of Pyrites Analysis.* P. Jannasch. Jour. prakt. Chem. [2], 40, 233—235.

THE sulphur in pyrites can be accurately estimated in a comparatively short time by the following method, which is more especially advantageous when the sample contains substances which render the application of solvents more difficult but allow of the expulsion of the sulphur in the dry way.

The finely divided pyrites is placed in a small boat and introduced into a combustion tube which can be heated in a small furnace. A current of air (about 150—200 bubbles per minute), dried by means of sulphuric acid and saturated with nitric acid vapour by passing through a Drechsel's flask containing about 50 cc. of fuming nitric acid, is passed through the combustion tube into a tubulated receiver containing 100 cc. of bromine water, to which is attached a Peligot's tube containing 40 cc. of bromine water, and finally through a cylinder of distilled water.

The pyrites is gradually heated, commencing at the hinder portions of the tube, the front parts being also warm to prevent the condensation of the sulphuric acid. In about three-quarters of an hour, the process being at an end, the Drechsel's flask containing nitric acid is removed and the boat is allowed to cool slowly in a stream of air. The sublimation of the sulphur by too rapid heating is to be avoided.

The contents of the receivers are washed into a beaker, the excess of bromine evaporated, the solution mixed with 1 cc. of concentrated hydrochloric acid, heated to boiling and the sulphuric acid precipitated with barium chloride; the precipitate must be carefully washed with boiling dilute hydrochloric acid, to free it from barium nitrate, but any admixture with this salt can be avoided by evaporating completely in a porcelain basin before adding the barium chloride.

The boat containing the ferric oxide, which is quite free from unchanged pyrites, is warmed with concentrated hydrochloric acid, the solution evaporated almost to dryness and the insoluble residue estimated in the usual manner; the iron solution is completely free from sulphuric acid.

Two analyses by this method gave the following results:—

	Per Cent.	Per Cent.
Sulphur .....	52.59.	52.67
Iron .....	45.67	45.52
SiO <sub>2</sub> , &c. ....	1.08	1.12
	<u>99.34</u>	<u>99.31</u>

—F. S. K.

*Estimation of Sulphuric Acid in Presence of Iron.* P. Jannasch. J. prakt. Chem. [2], 40, 236.

THE author and Richards have stated (J. prakt. Chem. [2], 39, 321; this Journal, 1889, 819), that Lunge's method of pyrites analysis is not accurate; but this statement was made with reference to the older method only, the new method having been overlooked. Lunge himself has shown (Handbuch der Soda-Industrie) that his method gives accurate results if the iron is precipitated with a slight excess of ammonia at a moderate temperature before determining the sulphuric acid. The author now confirms Lunge's statement, and gives analyses of a sample of pyrites carried out (I.) by Lunge's improved method, (II.) by the nitric method described in the preceding abstract:—

I.		II. (Average of two analyses.)	
	Per Cent.		Per Cent.
Sulphur .....	52.56		52.63
Iron .....	45.88		45.59
SiO <sub>2</sub> , &c. ....	1.08		1.10
	<u>99.47</u>		<u>99.32</u>

—F. S. K.

*The Determination of Chlorine in Water.* A. Hazen. Amer. Chem. Jour. 11, 409—414.

IN estimating chlorine in water or sewage by titration with silver nitrate solution the results commonly obtained are too high, owing to the fact that the red colour of silver chromate does not become perceptible until a certain excess of silver has been added. Under certain conditions, however, the required excess is constant, and by making the proper corrections very accurate results can be obtained.

The author has made a large number of experiments with a solution of silver nitrate, 1 cc. of which is equal to 0.5 mgrm. of chlorine, prepared by dissolving the calculated quantity of pure silver nitrate in distilled water. The following is an account of his experiences:—

The amount of potassium chromate used has a considerable influence on the results, especially in a large volume. In general, the greater the quantity of chromate used the smaller will be the required excess of silver, but when the quantity has reached a certain point the advantage gained by any further increase is more than overcome by the increase in colour which obscures the exact end-point. 0.001 grm. of potassium chromate per cubic centimetre of liquid titrated was found to be a suitable quantity, but in practice the author employs 0.06 grm.

The required excess of silver solution increases regularly with the volume of the liquid titrated; when 0.06 grm. of potassium chromate is used and the volume is not more than 60 cc., this excess may be represented by the formula  $x = .003v + 0.02$  where  $v$  is the volume of the liquid at the end of titration. Thus with a volume of 3 cc. the correction is 0.03 cc., whilst with a volume of 50 cc. the correction is 0.17 cc.

The required excess of silver solution, in addition to the correction for volume, increases regularly with the amount of the precipitated silver chloride, and is nearly proportional to the amount of the silver solution used. The correction is nearly 1 per cent. of the volume of the silver solution, and may be most conveniently applied by employing a solution containing 1 per cent. more than the theoretical quantity of silver. If the silver solution is standardised against sodium chloride it will be of this strength, and no correction will be required for the amount of the precipitate. If the amount of chlorine is very low the results are somewhat irregular, so that if less than 1 cc. of silver solution is required for 50 cc. of water it is preferable to run in 1 cc. of salt solution and deduct the equivalent of silver solution from the result.

Instead of preparing the silver solution by dissolving the theoretical quantity of silver nitrate it is practically more convenient and better to depend on titrations of salt solutions for its strength, always making the correction for volume.

It is possible by direct titration in this way to get results within 0.05 part of chlorine in 100,000 in clear waters when the chlorine is not higher than 10 parts. When the chlorine is low and greater accuracy is required the water must be concentrated before titration. In such cases excess of sodium carbonate must be added before evaporating, otherwise there may be a serious loss of hydrochloric acid; commonly 0.02 grm. of sodium carbonate, or less is sufficient, and this quantity does not interfere with titration. If too much has been used calcium nitrate may be added before titration, as an excess of nitrate does not influence the result. The water can be concentrated (1) by boiling down 500 cc. or more to 50 cc. with sodium carbonate in a flask; if much organic matter is present the solution becomes brown and must be decolourised with aluminium hydrate, and filtered before titrating. (2) By evaporating on the water-bath with sodium carbonate. (3) By evaporating to dryness and igniting in a radiator: the chlorides are extracted from the residue with hot water and titrated as usual. The last method seems to be most accurate when much organic matter is present.—F. S. K.

*Precipitation of Aluminium and Iron by Means of Ammonia.* G. Lunge. *Zeits. f. angew. Chem.* 1889, 634—635.

THE usual method is to precipitate with slight excess of ammonia, and then to expel excess of ammonia by boiling. Fresenius remarks that this operation should be conducted in a platinum or good porcelain dish, and not in a glass vessel, owing to the glass being attacked. The whole is boiled because the precipitate is, to a slight extent, soluble in free ammonia; if, however, the boiling be continued too long, some of the ammonium chloride which may be present is decomposed, and the hydrochloric acid thereby formed dissolves a portion of the alumina and oxide of iron. A further precaution usually taken is to ignite the precipitate with the blow-pipe before weighing—this because the precipitate persistently retains sulphuric acid (should this be present) in the form of basic sulphates. The author has pointed out, in connexion with the analysis of pyrites (this *Journal*, 1889, 966), that by using only very slight excess of ammonia, and then warming gently for 10 minutes before filtering, a precipitate is obtained which settles well, filters easily, and is perfectly free from sulphuric acid. Still, however, the excess of ammonia dissolves some of the alumina and oxide of iron. It has been pointed out by Fresenius (*Quant. Anal.* II., 807, 6th German edition), and it is a fact too often overlooked, that in presence of ammonium chloride not a trace of alumina goes into solution. This is perfectly in agreement with the author's observations. By adding ammonium chloride and excess of ammonia, and then warming gently, three advantages are thus gained:—(a) there is no danger of decomposing ammonium chloride, since there is no boiling, and hence no danger of thereby getting a portion of the precipitate into solution; (b) the precipitation, filtration, and washing occupy a much shorter time; and (c) igniting with the blow-pipe is rendered unnecessary, as the precipitate contains no sulphuric acid.—T. L. B.

*Determination of Iron and Aluminium in Phosphates.* E. Glaser. *Zeits. f. angew. Chem.* 1889, 636—638.

THE method of determining iron and aluminium in phosphates by precipitating with ammonia and adding acetic acid is not applicable in every case; as, for instance, when calcium and magnesium phosphates are present in addition; again, the results obtained vary with the amount of acetic acid used, aluminium phosphate being soluble in acetic acid. The author proposes the following method, which has been tested with various phosphate mixtures of known composition, and shown to give reliable results. The calcium is separated from the phosphoric acid by sulphuric acid in presence of alcohol, and the iron and aluminium phosphates may then be obtained free from calcium and magnesium without the use of acetic acid:—

Five grms. of phosphate are dissolved in 25 cc. of nitric acid (sp. gr. 1.2) and in 12.5 cc. of hydrochloric acid (sp. gr. 1.12), and the solution is made up to half a litre. 100 cc. of filtrate (= 1 gm. of substance) are transferred to a 250 cc. flask, and 25 cc. of sulphuric acid (sp. gr. 1.84) are added. After the lapse of about five minutes, during which time the flask with its contents has been occasionally shaken, 100 cc. of 95 per cent. alcohol are added, the whole is cooled, and the flask filled to the containing mark with alcohol, and well shaken. Contraction takes place and the flask is again filled to the containing mark with alcohol, and again shaken. The whole is allowed to stand half an hour and is then filtered. 100 cc. of the filtrate (= 0.4 gm. of substance) are transferred to a platinum dish and the alcohol is evaporated away, after which the solution free from alcohol is transferred with 50 cc. of water to a beaker and boiled; ammonia is then added, the whole again well boiled, allowed to cool, and filtered. The residue is ignited and weighed as phosphates of iron and aluminium. A determination may be conveniently carried out in  $1\frac{1}{2}$  to 2 hours.—T. L. B.

*The Götz Method of Determining Phosphorus.* C. Bornmann. *Zeits. f. angew. Chem.* 1889, 638—639.

IT has been stated that this method gives good results only with samples of steel poor in carbon. According to the author, however, the percentage of carbon does not influence the result, if the method be carried out as follows: In a new beaker of about 100 cc. capacity 1.2 grms. of steel is dissolved in 25 cc. of nitric acid (900 cc. of nitric acid of sp. gr. 1.4 to 100 cc. of water); the solution is then concentrated, and 8 to 16 drops of potassium permanganate are added (cast iron requires 8 drops, rail steel 16); the peroxide of manganese is dissolved by addition of 5 to 10 drops of hydrochloric acid (sp. gr. 1.19). Excess of hydrochloric acid is then driven off and the solution concentrated as far as possible. To the hot solution are next added 10 cc. of a 25 per cent. solution of ammonium nitrate, and 25 cc. of Finkener's ammonium nitromolybdate solution. The contents of the beaker are then washed with the aid of a 25 per cent. solution of ammonium nitrate into a small pear-shaped vessel with graduated stem, such vessel having a capacity of about 60—70 cc., and the graduated stem (40 mm. in length and divided into 40 parts) having a capacity of 0.2 cc. The whole is well shaken for about a minute, and the vessel is thereupon filled with 25 per cent. ammonium nitrate solution and fixed into a centrifugal machine prepared for it, and rotated for two minutes at the rate of 1,200 revolutions per minute, after which the machine is stopped as suddenly as possible. The reading on the stem divided by two now gives the amount of phosphorus in hundredths per cent. The results are affected by silicon, if this reaches over 0.25 per cent., in which case silica separates.

The potassium permanganate solution used for oxidation contains 12 grms. per litre. Finkener's molybdate solution is prepared by dissolving 160 grms. of ammonium molybdate in 795 cc. of cold water, adding 320 cc. of ammonia (sp. gr. 0.925), and pouring the whole into a mixture of 1,710 cc. of nitric acid (sp. gr. 1.2) and 1,205 cc. of water, keeping well cooled the while.

The author gives a series of analyses performed by this method, and also gravimetrically; the greatest difference obtained was 0.005 per cent. of phosphorus.—T. L. B.

*The Estimation of Phosphorus in Phosphor-Tin.* W. Hempel. *Ber.* 22, 2478.

THE alloy is heated in a current of chlorine and the chloride led into 10 cc. of concentrated nitric acid. The phosphoric acid is precipitated in the solution with molybdic acid in the usual way.—J. B. C.

*Estimation of Ferrocyanogen Compounds in the By-Products of Gas-Manufacture.* R. Gasch. *Journ. f. Gasbeleucht.* 1889, 966—971. (Compare this *Journal*, 1889, 732—733.)

THE author has modified the method proposed by Zulkowski by using a 1 per cent. solution of uranium acetate instead of ferric chloride as indicator, and by employing a more dilute solution of zinc sulphate, 20.62 grms. of the crystalline salt per litre, which is standardised against pure potassium ferrocyanide. The process applied to old gas-purifying material is as follows: 20 grms. of the finely pulverised dry substance are triturated in a warm mortar with warm (not above 50°) 15 to 20 per cent. caustic soda, washed into a 200 cc. flask, made up to mark (the quantity of soda and water used being noted), and filtered direct into a burette. The solution is run into 10 cc. of the zinc solution which has been strongly acidified with sulphuric acid and heated to 70°—86° C. until a drop in excess is indicated by the browning of the drop of uranium acetate on the porcelain slab. The method may be applied to determine cyanogen compounds by previously converting them into ferrocyanides by boiling with caustic soda and ferrous sulphate.—D. A. L.

*A Reaction for Cocaine.* O. Lerch and C. Schürges.  
Schweiz. Wochenschr. Pharm. **27**, 293.

THE cocaine to be tested is dissolved in water and a drop of ferric chloride solution is added. On boiling the solution an intense red colour is developed owing to the formation of benzoic acid. Benzoyl-ecgonine also gives this reaction, but this substance differs considerably from cocaine in melting point and solubility. Cocaine is readily soluble in ether, benzoyl-ecgonine is not soluble.—H. T. P.

*Microscopic Examination of Paper.* W. Herzberg. Mitt. Königl. tech. Versuchs. **1889**, **3**, 113—115.

THE method adopted for preparing the paper fibre for microscopical examination consists in treating the sample with 500 cc. of water to which has been added 5 cc. of a concentrated solution of caustic soda, boiling and shaking until the paper is disintegrated, draining and washing from the alkaline liquor on a fine sieve, and thoroughly separating any clots of fibre by shaking in a 100 cc. flask with distilled water and a large number of small garnets, by which means an emulsion of single fibres with water, well suited for the purpose in hand, is obtained.

The discrimination of fibres of different origins cannot with certainty be effected by any of the colour reactions that have been from time to time proposed.

A reagent composed of 20 cc. of water, 2 cc. of glycerol, 1·15 grms. of iodine, and 2 grms. of potassium iodide is useful, not so much on account of the fairly characteristic colourations it gives with paper fibre from various sources, as because of the increase of clearness and differentiation of structure under the microscope which it effects.—B. B.

*Solidifying Points of Viscous Oils.* Holde. Mitt. Königl. tech. Versuchs. **1889**, **3**, 119—126.

SEEING that both mineral and fatty oils may be regarded as solutions of bodies of higher melting point in others of lower melting point, it is not surprising that it is difficult to assign to such mixtures definite solidifying points; the temperature at which any deposit appears may be regarded as that of the commencement of solidification. Between this point and the completion of the process a certain liberation of heat occurs, which is usually more considerable for mineral than fatty oils; in the latter, however, the change of state is generally sharper. Besides these inevitable difficulties, avoidable causes of error are the failure to expose the oil to the cooling-bath for a sufficient period, and the irregular agitation of the sample, *e.g.*, *Salkowsky* found that a cod-liver oil which remained perfectly clear when cooled for a short time to  $-15^{\circ}\text{C}$ ., became quite solid after several hours' exposure to  $-4^{\circ}\text{C}$ .

The author proceeded, therefore, to investigate these two factors, at present somewhat neglected. The tests were carried out in test-tubes 15 mm. in diameter, filled to a height of 3 cm., and *Hoffmeister's* method of cooling (Mitt. Königl. tech. Versuchs. **1889**, **1**, 24; this Journal, **1889**, 423) was employed. The results are recorded in voluminous tables unfitted for useful abstraction.

The conclusions arrived at were:—

Provided agitation be avoided, vegetable oils require very lengthy exposure to cold before beginning to solidify. For example, rape oil, which remained clear for two hours at  $-3^{\circ}\text{C}$ ., gave a white deposit after three hours, and was in great part of the consistency of tallow after 6½ hours; a mustard-seed oil took 5½ hours' and one day's cooling to reach the same conditions respectively; this peculiar behaviour accounts for the low solidifying point usually quoted, *viz.*,  $-18^{\circ}\text{C}$ ., and may be viewed as analogous to that of a supersaturated solution, a view strengthened by the hastening of solidification caused by the introduction of a particle already solid. Mineral oils deposit solid matter sooner than vegetable oils, and reach their final condition for any given temperature more quickly than the latter. Stirring, instead of increasing their consistency as in the case of vegetable oils, diminishes it, by breaking up the network of paraffin or vaseline-like particles which has

formed. It follows that a mineral oil should be tested by cooling to the desired temperature without agitation, and inclining the tube, or otherwise judging of its consistency only after the lapse of one hour.—B. B.

*The Detection of Sesame Oil.* M. W. Bishop. J. Pharm. Chim. **1889**, **20**, 244.

SESAME oil, when treated with a mixture of equal parts of nitric and sulphuric acids, quickly assumes a fine green colour. With a freshly prepared solution of cane sugar in hydrochloric acid of 22° B. it yields, on shaking, a cherry-red tint. Fresh sesame oil suffers no change when agitated with pure hydrochloric acid of 21—22° B. (2 volumes of oil to 3 volumes of acid). If, however, the oil be previously exposed to air and light for a few days, it gives with hydrochloric acid a green mixture. On standing, the acid layer which separates alone is coloured. In the case of old oil the acid deposits bluish violet flakes. Very old oil, which has been exposed for years to air and light, imparts to the acid an almost blue colour. On standing a copious flocculent blue precipitate separates from the acid, the latter becoming of a clear green colour. Sesame oil, which has been deprived of its colouring matter by hydrochloric acid, still gives a cherry-red tint with a solution of cane sugar in hydrochloric acid. When testing for sesame oil in olive oil it is best to expose the sample to bright light for a few days. Then mix about 6—8 cc. of the oil with 12—14 cc. of hydrochloric acid of 21—22° B., and shake violently in a stoppered cylinder of about 35 cc. capacity.—H. T. P.

*The Detection of Resin Oil in Castor Oil.* H. Gilbert. Chem. Zeit. **13**, 1428.

THE ordinary tests for castor oil prescribed by the German Pharmacopœia do very well for the detection of fatty oils, but fail in the presence of resin oil. A sample of castor oil recently examined by the author answered all the ordinary tests satisfactorily, but its taste and smell were suspicious. On saponification it was found to contain 19 per cent. of resin oil, and its saponification equivalent was 126, whilst the saponification equivalent of pure castor oil is, according to Valenta, 180—181·5. Pure castor oil, when agitated with an equal bulk of nitric acid of 1·31 specific gravity, is turned slightly brown, the acid remaining colourless. Under the same conditions resin oil is turned almost black, and the acid assumes a yellowish-brown tint. The adulterated sample of castor oil yielded, on treatment with nitric acid, the colour reaction of resin oil, but of course in a degree corresponding to the dilution.—H. T. P.

*Comparison of Different Methods of Determining Free Acid in Oils.* Holde. Mitt. Königl. tech. Versuchs. **1889**, **3**, 116—119.

GEISSLER'S method, consisting in dissolving the oil in ether-alcohol and titrating it with decinormal alcoholic (50 per cent.) alkali, is accurate and can be used in all cases, save where the oil is so dark coloured as to hide the change of the indicator (phenolphthalein) used. By employing a cylinder about 4 cm. in diameter instead of a flask, this difficulty is much lessened. The process is especially suited for clear mineral oils. *Burstyn's* plan of shaking the oil with an equal volume of alcohol, drawing off and titrating an aliquot portion, is fallacious, as the whole of the free fat acid is only extracted by repeated treatment with alcohol, and thus the results are low when compared with those given by Geissler's method, *e.g.*, a refined rape oil containing 0·730 per cent. of acid, calculated as  $\text{SO}_3$ , gave only 0·374 per cent. when thus treated.

*Merz* shakes the oil with alcohol in a stoppered flask and titrates the mixture with repeated agitation. This gives accurate results, and is well adapted for oils containing a considerable quantity of free fat acid, as if treated by Geissler's method they require an inconveniently large amount of alcohol to prevent the oil being precipitated from its ethereal solution by the standard caustic alkali in weak alcohol.

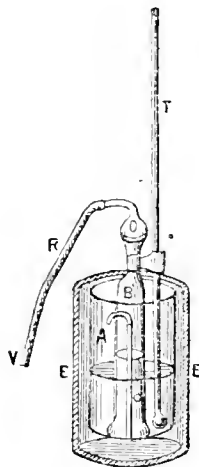


Burstyn's physical method, which consists in shaking the oil with alcohol of known specific gravity and deducing the quantity of fat acid it extracts by the increase in density it suffers, is quite untrustworthy; e.g., an oil containing 0.091 per cent. of acid, calculated as  $\text{SO}_3$ , gave 0.280 per cent. Many causes conduce to this result; among them may be mentioned the different specific gravities of the acids extracted, the error caused by the presence of a mineral acid, such as sulphuric acid, and the varying quantity of neutral oil dissolved. The following method is adapted for dark opaque mineral oils: 25 cc. of the oil is shaken with 50 cc. of absolute alcohol in a stoppered cylinder about 2 cm. in diameter, and the mixture allowed to settle; 25 cc. of the alcohol is then drawn off, 10 cc. of ether added if turbid, and titrated with decinormal caustic soda. If more than 2 cc. be used, the rest of the alcohol is poured off, another 50 cc. added, and the process repeated.

The free acid has been calculated as  $\text{SO}_3$  throughout the paper for the sake of uniformity.—B. B.

*The Examination of Oils.* F. Jean. J. Pharm. Chim. 1889, 20, 337.

THE author recommends a method of examining oils which depends on the determination of the specific gravity, the melting point of the fatty acids, the heat evolved on mixing the oil with sulphuric acid, and lastly of the refractive index. The specific gravity is determined by means of Westphal's areothermic balance. The melting point is determined in an apparatus consisting of a thin-walled U-tube, the limbs of which are of unequal length. The shorter limb ends in a small funnel through which the melted substance is introduced, in quantity sufficient to fill the bend of the U-tube. A platinum wire is passed so far into each limb that the two ends nearly meet in the bend. The tube is supported in a beaker of water by means of a clamp which also carries a thermometer. The two platinum wires are connected to an electric bell and battery, and a little mercury is poured into one of the limbs of the U-tube. On warming the water in the beaker, the fat melts and allows the mercury to pass to the lower portion of the bend, where it closes the circuit between the two platinum wires, and as a consequence the bell rings. At this moment the temperature is noted. The heat evolved by mixing the oil with sulphuric acid is determined by means of the following apparatus (see diagram) named by the author "Thermoeleometer."



A is a small vessel, 4 cm. in diameter and 6 cm. high. It is graduated at 15 cc., and serves for the reception of the oil. B is the acid holder, and is fitted with a hollow glass stopper, to which is attached the india-rubber tube R. The neck of the acid-holder carries a clamp, in which a thermometer is fixed. 5 cc. of sulphuric acid of 65° B. are placed in the acid-holder, and 15 cc. of the oil to be tested

in the vessel A. The oil is heated up to about 40°–50° C., when the acid-holder B is placed in it, and the apparatus is allowed to cool, with occasional stirring, to 30° C. To hinder further cooling it is then placed in the felt-lined brass case E. The acid is now forced out of B, through the small tube, by blowing through R, the oil and acid are well mixed, using B as a stirrer, and the maximum temperature is noted. If the oils are much oxidised they must be first treated with alcohol, or, better still, saponified, and the fatty acids separated and employed for the determination of the degree of heating. The author obtained the following numbers with the thermoeleometer:—

Fatty Acids.

Olive oil.....	41.5	45
Linseed oil.....	61	109
Colza oil, Pas de Calais.....	37	44
„ India.....	37	46

For the determination of the refractive index the author and Amagat employ a modification of Tramm's oleo-refractometer. Each oil possesses a characteristic refractive power which varies but slightly, according to the source of the oil. The author gives the following deviations for various oils:—Olive oil,  $1\frac{1}{2}^{\circ}$ – $2^{\circ}$ ; earth-nut oil,  $4^{\circ}$ – $5^{\circ}$ ; colza oil,  $16\frac{1}{2}^{\circ}$ – $17\frac{1}{2}^{\circ}$ ; sesame oil,  $17^{\circ}$ ; cotton-seed oil,  $20^{\circ}$ ; castor oil,  $40^{\circ}$ ; linseed oil,  $53^{\circ}$ ; hemp oil,  $33^{\circ}$ ; poppy oil,  $30^{\circ}$ ; maize oil,  $27^{\circ}$ ; sweet almond oil,  $6^{\circ}$ ; cod-liver oil,  $42^{\circ}$ ; whale oil,  $30\frac{1}{2}^{\circ}$ . All the above deviations are to the right; the following oils give a levo-reading:—Sheep-trotter oil,  $0^{\circ}$ ; horse-hoof oil,  $-12^{\circ}$ ; ox-foot oil,  $-3^{\circ}$ ; sperm oil,  $-12^{\circ}$ . Olive oil from Nizza, giving a deviation of  $1\frac{1}{2}^{\circ}$ , when mixed with 10 per cent. poppy oil, gave  $6\frac{1}{2}^{\circ}$ , and on addition of 20 per cent. poppy oil the reading was  $10^{\circ}$ . Mixed with 10 per cent. cotton-seed oil the deviation was  $3^{\circ}$ , and with 20 per cent.  $5^{\circ}$ . Pure poppy oil, giving a reading of  $34^{\circ}$ , when mixed with 20 per cent. of earth-nut oil, only yielded  $25^{\circ}$ ; 20 per cent. of cotton-seed oil added to castor oil diminishes its reading by  $7^{\circ}$ . The resin and heavy mineral oils are readily detected, in admixture with animal and vegetable oils, by their high refractive power. According to the author, an oil may positively be regarded as pure when its specific gravity, refractive power, and degree of heating with sulphuric acid correspond with the values obtained for a typical sample of known purity.—H. T. P.

*The Technical Examination of Glues.* W. Kalmann, Leipziger Monatsch. für Textil-Ind.

THE author gives the results of his experience in examining glues used for sizing warps.

He finds that Kissling's method (this Journal, 1887, 565–566) for ascertaining whether glue is made from leather or from bones, and based on the different behaviour of the ashes on heating, and on their qualitative analysis, is a reliable one. For the purpose mentioned above, bone glues are almost exclusively used.

Since in woollen manufacturing both dyed and undyed wool is sized, glues used for the purpose should be free from acid. The author found that glue solutions which did not alter litmus, might give an acid reaction with phenolphthalein, probably owing to the presence of an organic acid which does not effect litmus. He estimated the amount of this acid by titrating directly with decinormal alkali and using phenolphthalein as indicator.

Glue to be used in woollen manufacturing, besides being neutral to litmus, and therefore free from mineral acids, should contain no sulphurous acid, whether free or in the form of normal sulphites. It was found that glue containing these substances is capable of producing light patches on dyed wool, which defect is sufficient to exclude any such glue from use in the woollen manufacture. Sulphurous acid may be estimated by titrating the solution to which starch paste has been added with about  $\frac{1}{10}$  normal iodine solution. For this titration the same solution can be used as that in which the acid has been estimated with alkali and phenolphthalein, thereby shortening the examination.

In order to determine whether organic matter contained in glue affected the iodine and thus made the amount of sulphurous acid too high, the author has often estimated after the iodine titration the hydriodic acid which is produced,  $M_2SO_3 + H_2O + I_2 = M_2SO_4 + 2HI$ , with decinormal alkali, and has always found the two estimations to coincide, proving that the iodine is not affected. This additional titration requires very little time, and should be made as a check.

The following is the method:—1 gram. of the coarsely powdered sample of glue is dissolved in water on the water bath, the solution first tested with blue litmus paper and then titrated with decinormal caustic alkali, using phenolphthalein as indicator, until it turns red. The liquid is then

cooled, starch paste added and then iodine solution until it turns blue. The blue solution is next decolourised with a drop of sodium bisulphite solution and again titrated with decinormal caustic alkali, until it becomes red. The amount of decinormal alkali used in the last titration is equivalent to the amount of sulphurous acid contained in the glue, and also to the iodine solution.

In determining the comparative value of samples of glue, the water and ash estimations are of chief importance, but with glues to be used in the woollen manufacture, care should also be taken that they contain no ingredient which affects colouring matters.

The following table contains the results of some of the author's analyses:—

No.	Variety of Glue.	Amount taken.	Reaction with Litmus.	Na <sub>2</sub> O equivalent to Amount of Free Acid present.	Amount of Iodine required.	SO <sub>2</sub> .	Amount of $\frac{1}{10}$ N Alkali Corresponding to the Iodine, cc.	Amount of $\frac{1}{10}$ N Alkali taken, cc.	Ash.
		Grams.		Per Cent.		Per Cent.			Per Cent.
1	Gelat'n.	3.363	Faintly acid.	0.46	0.60336	0.03	..	..	..
2	Bone-glue.	3.0478	" "	1.32	0.055410	0.16	..	..	3.55
3	" "	3.1535	" "	0.81	0.023611	0.20	..	..	3.69
4	" "	3.3524	Acid.	1.56	0.127882	0.16	10.1	10.3	..
5	" "	3.6412	Neutral.	0.14	0.016262	0.11	1.28	1.3	..
6	" "	3.3770	Acid.	2.02	0.185522	1.41	11.16	11.6	1.63
7	" "	3.1330	Neutral.	0.67	0.011827	0.09	..	..	2.21
8	" "	2.9808	Faintly acid.	0.95	0.042427	0.26	..	..	2.37
9	" "	3.1263	Acid.	1.49	0.110418	1.03	11.06	11.2	1.92

Samples 2, 4, 6 and 9 on material dyed with logwood produced distinctly lighter spots.

According to the foregoing the requirements for glue to be used in the woollen manufacture are as follows:—

1. The solution of the glue should be neutral to litmus paper, or at the most only slightly acid.

2. The amount of free acid which reacts with phenolphthalein should not correspond to more than 1 per cent. of Na<sub>2</sub>O.

3. The amount of sulphurous acid contained should not exceed 0.5 per cent.

4. According to Kissling's estimations, with which those given above coincide, the amount of ash should not be much above 3 per cent., and the amount of water not above 15 per cent.

#### *The Estimation of Cane Sugar by Inversion.* Böhm. Zeits. f. Zuckerind. 13, 559.

Working by Herzfeld's method, Herls found the rotatory power of a cane-sugar solution reading + 100 before inversion, to be - 33.3° after inversion. Croydt's method always gives a lower value, owing to the destructive action of the hydrochloric acid on the sugar. The addition of various salts (5.2-10 parts of potassium nitrate, sodium acetate, and a mixture of these with potassium sulphate and chloride to every 100 parts of sugar) generally increases the levo-reading (up to - 33.69°), but acetates diminish it (- 32.86°). According to Clerget, the levo-reading diminishes by 0.5° for every 1° C. increase of temperature. This does not hold good for all concentrations. Thus, for a solution reading + 50° it is 0.48°, and for one reading + 100 it is 0.51°. Practically these variations are of no consequence. For clarifying purposes, basic lead nitrate is recommended, which is formed in the sugar solution itself by the addition of lead nitrate, followed by potassium hydrate. These additions raise the levo-reading to - 33.5 at 20° C. When working in other respects by Herzfeld's

method, the cane sugar may be calculated by the following formula:—

$$Z = \frac{100S}{1.035 - 0.5t};$$

If raffinose be present the formulae are—

$$Z = \frac{0.5182 P + s}{0.8532}; \quad R = \frac{P - Z}{1.85}; \quad \text{at } 20^\circ \text{ C.}$$

Z = percentage of sucrose. P = polarisation before inversion.  
R = percentage of raffinose. s = polarisation after inversion.  
S = change of reading produced by inversion. t = temperature.

If the temperature of observation be not 20° C. the following formula must be employed:—

$$Z = \frac{r + 0.0015 t}{(r + i) - 0.0035 t}$$

r = reading of raffinose after inversion. i = For each degree  
t = reading of cane sugar after inversion) original polarisation.

$$\text{At } 6^\circ \text{ C. :— } r = 0.1882 \\ i = 0.435.$$

—H. T. P.

#### *The Estimation of Cane Sugar in Presence of considerable Quantities of Raffinose and Invert Sugar.* Wortman. Zeits. f. Zuckerind. 29, 767.

Compared at 20° C. with cane sugar, the rotatory power of raffinose is 1.85; of inverted raffinose, 0.9598; of inverted cane sugar, 0.3266; and of invert sugar, 0.3103. Calling P the direct polarisation, P<sup>i</sup> the polarisation after inversion, and N =  $\frac{C \times 47}{q}$  the approximate percentage of invert sugar (q = wt. of substance taken for analysis; 47 = the average factor of Meissl's tables), then—

$$P = S + 1.85 R - 0.3103 N \quad (1)$$

$$P^i = -0.3266 S + 0.9598 R - 0.3103 N \quad (2)$$

From these equations it follows that—

$$S = \frac{0.9598 P - 1.85 P^i - 0.277 N}{1.5048};$$

$$R = \frac{P - S + 0.3103 N}{1.85};$$

P = 1.85 R will then represent the sum of the readings due to invert sugar and raffinose. From this number Meissl's factor, and consequently the correct percentage of invert sugar may be derived. Substituting this value for N in the above equations, the real percentages of saccharose and raffinose are obtained. A mixture was prepared containing 46 per cent. of cane sugar, 3 per cent. of raffinose, and 4 per cent. of invert sugar. On analysis, the following numbers were obtained:—Cane sugar, 46.004 per cent.; raffinose, 2.993 per cent.; invert sugar, 4.02 per cent.—H. T. P.

*On the Estimation of Sugar and on Polarimetric Observations in Sweet Wines.* A. Bornträger. Zeits. f. angew. Chem. 1889, 477—486, 505—508, and 538—545.

The saccharine matter in sweet wines is composed of varying quantities of dextrose and levulose, and a means of accurately determining the relative quantities of these two sugars cannot be said to be established. The present extensive article forms an inquiry into the modifications recommended for adoption by various investigators, the basis of all the methods being titration with Fehling's solution and observation of the rotation shown by the polariscope. The author recommends the following treatment:—The wine is exactly neutralised with caustic alkali, and heated on the water-bath to drive off alcohol, allowed to cool in a flask, treated with a moderate quantity of lead acetate, and after dilution to the original volume with water, is filtered. The filtrate must be neutral or, if alkaline, must be carefully neutralised, and after standing for one day it is fit for observation in the polariscope and for titration by the Fehling-Soxhlet method.

The article is chiefly devoted to a description of experiments which show the necessity for adherence to the instructions given by the author as above. The influence of temperature and concentration of solution in the polarimetric observations receive special attention. If alkalinity of the wine during evaporation be avoided, the sugars present undergo no change, whilst an evaporation sufficient to drive off the alcohol is indispensable. For sweet wines a dilution to the original volume is sufficient, and as the action of lead acetate is intensified if the free acid be first neutralised, a moderate quantity of that reagent will be found to produce a sufficiently clarified solution for observation in a 200 mm. tube with all wines, except a few deeply coloured Malaga wines, in which case a shorter tube must be used.—G. H. B.

*Comparative Estimations of the Fat in Milk by Different Methods and in Different Conditions of the Milk.* J. Klein. Chem. Zeit. Rep. 13, 284—285.

In continuation of his previous experiments (Chem. Zeit. Rep. 12, 290 and 309), in which he found that Adams' method for the determination of milk fat yielded higher results than the sand or gypsum method, the author describes some comparative results obtained by Adams' and Rose's methods. The milk was examined when fresh, sour, and treated with borax. The results obtained show a close agreement between the two methods in the case of fresh and sour milk. Considerable differences occurred in two out of six analyses, when borax had been added. Estimations of fat were also made by the gypsum method, the results being compared with those got by Adams' process. It appears that even after 10 hours' extraction with ether, the results obtained by the gypsum method are distinctly lower than those got by the second method. Half an hour's extraction with ether serves to remove all the milk fat from the strip of paper employed in Adams' process. The paper strips are, previously to being used, freed from all fatty and resinous bodies by extraction with boiling alcohol. One hour's extraction is more than sufficient for this purpose. When ignited abestos was substituted for the paper strips, the results obtained were somewhat irregular. Assuming the results obtained by Adams' method to be correct, the author finds that the average error of the numbers obtained by Soxhlet's and the gypsum methods is 0.11 per cent. The author therefore holds to his former assertion that "all gravimetric determinations of milk fat made by the methods hitherto in use are low by 0.1 to 0.15 per cent."—H. T. P.

*International Chemical Congress at Paris.* July 30—August 3, 1889. Chem. Zeit. 13, 1391—1393.

### Agricultural Section.

#### I. Analysis of soils:

(a.) Separation of fine sand from clay:—Schloesing's method is recommended without modification. 10 grms. of soil and 2 litres of water are taken and allowed to stand 24 hours. Analyses performed under the same conditions are not exact, but they are sufficiently comparable for all practical purposes.

(b.) Determination of nitrogen:—The soda-lime method gives sufficiently good results; the amount due to nitrates may be left out of account by reason of the exceeding smallness of the quantities present. Kjeldahl's method gives results very slightly higher than the above.

(c.) Determination of nitrates:—The method of Schloesing is recommended, the nitric oxide being collected over mercury and not over water.

(d.) Determination of ammoniacal nitrogen. In this connexion two questions arise, viz.:—Should the determination be made with the soil directly, or with a solution? Should an acid be used? The Congress considers that the soil should be treated with a very slight excess of a weak acid, a sufficient quantity being used to decompose the whole of the calcium carbonate. The determination is then made with the solution. If determination directly from the soil be attempted, it is found that the quantity of ammonia cannot really be ascertained on account of the action of the alkalis on the organic substances contained in the soil.

(e.) Is there any means of ascertaining what percentage of the total phosphoric acid can be assimilated? It is considered in the first place that such a question has little practical interest, and, furthermore, that such a determination is not possible. Phosphate of iron is dissolved to a very considerable extent by treatment of the soil with acetic acid, owing to its solubility in organic iron salts. At the same time, the phosphoric acid of iron phosphate is of service to vegetation, but its assimilation is not easy.

(f.) Determination of potassium:—According to experiments of Schloesing, weak acids (e.g. 0.05—0.1 per cent. nitric acid) extract from soils a quantity of potash, gradually increasing for 3—4 hours; concentrated acids now extract a further quantity, and after this a third lot may be extracted by hydrofluoric acid. The potash seems then to exist in three forms: (1) as silicates difficultly acted on and of little interest to the agriculturist; (2) as more basic silicates, which are decomposed by concentrated acids and which furnish to vegetation a certain quantity of potash; and (3) in a form easily soluble in weak acids, and which can be easily assimilated by plants. It is therefore proposed to examine soils for potash by treating with boiling *aqua regia* until the remaining sand is perfectly white.

(g.) Determination of calcium:—The calcium, useful to vegetation, exists as carbonate, but calcium in other forms exists, and is useful as neutralising the acids of the soil. It is therefore important to distinguish these two forms in which calcium may exist. Total calcium may be determined by the usual methods in the solution obtained for potash determination; carbonate (in powdered form) may be determined in Mondesir's apparatus by using tartaric acid, or total carbonate (powdered and conglomerate) by using hydrochloric acid.

For the potash determination soils should be passed through a sieve having 60 meshes to the inch; decomposition is then complete in two hours; whereas, if a sieve of only 10 meshes to the inch be used (as Gasparin suggests), 12 hours are necessary for decomposition. For the nitrogen determination the soil should be as fine as possible. In every case drying at 100° should be carried out.

#### II. Analysis of manures:

(a.) Determination of nitrogen contained as ammonia, nitrates, and organic:—Total nitrogen may be determined by Dumas' method with copper oxide, and the nitrate nitrogen by Schloesing's method, collecting the nitric oxide over mercury. This latter may also be determined by treating with lime and 50 per cent. alcohol, whereby sulphuric acid, phosphoric acid, silica, &c. are obtained in

the residue, and the nitrate in alcoholic solution; by then evaporating this solution and igniting with ammonium chloride the nitrates are converted into chlorides; the chlorine is then determined, and from this (diminished by the original chlorine in the sample), the nitrates may very simply be calculated. For determination of ammoniacal nitrogen the nitric acid is decomposed by means of ferrous chloride and hydrochloric acid; the ammonia may then be displaced by magnesia. Organic nitrogen may be ascertained in the residue from the two preceding operations by means of soda-lime. After freeing from nitrate nitrogen the Kjeldahl process may be used.

(b.) Distinction of manures according to their source: natural phosphates, slags, dried blood, horn, leather, &c.—The complete solution of this problem is considered of little practical worth, and in some cases it is extremely difficult.

### III. Analysis of fodder:

(a.) Separation and determination of so-called fatty substances and resins:—This may be done by extraction with ether. In special cases such as ensilage, where the fermentation has produced a considerable quantity of lactic acid, which is also soluble in ether, the extract should, before weighing, be washed with water.

(b.) Non-nitrogenous extractive substances are ascertained by difference, being considered as the total of substances not directly determinable.

(c.) Determination of nutritious nitrogenous compounds:—It is not sufficient to determine, as is often done, total nitrogen and calculate this to albuminoids. By doing this, ammonia, nitrates, &c., are all taken as albuminoids. Only such nitrogenous compounds as are insoluble in dilute acetic acid are to be looked upon as nutritious.

(d.) As to tabulation of results, it is considered that these should be given in two forms: (1) on undried; (2) on dried stuff. If two different kinds of fodder are to be compared, they should be compared after being dried; if, however, the value of a fodder is simply required, this should be estimated on the sample in the condition in which it is to be used. Congress considers the following tabulation desirable:—

Moisture.	
Nutritious nitrogenous substances.	
Non-nutritious nitrogenous substances.	
Carbohydrates, &c.	<ul style="list-style-type: none"> <li>{ Fatty substances.</li> <li>{ Starchy substances.</li> <li>{ Sugars.</li> <li>{ Cellulose.</li> <li>{ Non-nitrogenous extractive substances.</li> </ul>
Mineral matter	<ul style="list-style-type: none"> <li>{ Phosphoric acid.</li> <li>{ Other mineral matter.</li> </ul>

### Pharmaceutical Section.

Sulphate of quinine: It is considered advisable that a determination of the sulphuric acid be made, and also a polarimetric examination.

Cinchona bark:—An accurate and practicable method of extraction and separation of the various alkaloids is desirable, and a committee of investigation has been appointed.

Opium:—Chiefly two methods are used for determination of opium: (a) by ammonium chloride; (b) by alcohol and ammonia. In the first method the ammonium chloride should be reduced in quantity as far as possible, owing to the solubility of morphine therein; in the second method the amount of alcohol and ammonia should be kept as low as possible. As pure morphine is obtained in each case, the solubility of this latter in dilute caustic potash ought to be known. It is also desirable to fix a minimum valuation for opium extract. A commission was appointed to inquire into the question.

Chloroform:—Seeing that chloroform keeps better when mixed with alcohol, it is desirable to fix the amount of alcohol which may be added, and to ascertain methods for the estimation of the alcohol added.

Phenol and salicylic acid:—Accurate determination of the chief physical constants is desirable, as well as an alkalimetric method of examining salicylic acid, which is often mixed with higher homologous acids.

Analgesine:—A commission has been appointed to examine its physical constants and chief reactions.—T. L. B.

### Estimation of Formaldehyde by Titration with Ammonia.

W. Eschweiler. Ber. 22, 1929—1930.

The author finds that by titrating with litmus as indicator in place of methyl orange or cochineal, the compound hexamethylenamine which is formed has a neutral reaction, and that one drop of acid colours the solutions red. The same is the case with phenolphthalein. With methyl orange, cochineal, Congo red, and tropoline as indicators, the mono acid compound of hexamethylenamine has a neutral reaction, so that in this case six molecules of formaldehyde appear to combine with three in place of four molecules of ammonia. The author further finds that in order to produce the compound hexamethylenamine the solution of formaldehyde and ammonia must be heated for one hour to 100°—J. B. C.

### ERRATUM.

This Journal, 1889, 902, at the foot of table, for "Saponification equivalents" read "Saponification value."

## New Books.

TECHNISCH-CHEMISCHES JAHRBUCH, 1888—1889. Ein Bericht über die Fortschritte auf dem Gebiete der CHEMISCHEN TECHNOLOGIE, vom April 1888 bis April 1889. Herausgegeben von DR. RUDOLF BREDERMANN, 11<sup>ter</sup> Jahrgang. Berlin: W. Carl Heymann's Verlag, 1890. London: H. Grevel & Co., 33, King Street, Covent Garden, E.C.

Octavo volume strongly bound in cloth and gilt-lettered. It contains Preface, Table of Contents, List of Abbreviated Titles of Works referred to in the text, 614 pages of Subject-Matter, with which are interspersed 263 wood engravings. In the pages enumerated is included a review of New Books which have appeared during the session 1888-89, arranged as works on Physics, Chemistry, Chemical Technology, Mineralogy, Geology, and Botany, and works on miscellaneous subjects. There is also a carefully arranged Index of Names of Authors and another of Subject-Matter.

The arrangement of the Subject-Matter is as follows:—I. Eisen. II. Alkalimetalle, Magnesium, und Aluminium III. Gold und Silber. IV. Kupfer. V. Blei. VI. Zink. VII. Zinn. VIII. Nickel und Kobalt. IX. Chrom und Mangan. X. Antimon, Arsen, Quecksilber, und Platin. XI. Chlor, Salzsäure, Brom. XII. Schwefel, &c. XIII. Sauerstoff, Wasserstoff, &c. XIV. Soda. XV. Kaliumverbindungen. XVI. Ammoniak. XVII. Alkalische Erden. XVIII. Thonerde. XIX. Glas. XX. Thonwaaren. XXI. Cement. XXII. Künstliche Steine. XXIII. Explosivstoffe. XXIV. Leuchtstoffe. XXV. Heizstoffe. XXVI. Zucker. XXVII. Stärke und Stärkezucker. XXVIII. Wein. XXIX. Bier. XXX. Spiritus. XXXI. Fette, Harze, Seifen, &c.

XXXII. Wasser. XXXIII. Nahrungsmittel. XXXIV. Düngemittel, Abfälle, Disinfection. XXXV. Gerberei. XXXVI. Leim. XXXVII. Organische Säuren. XXXVIII. Alkaloide. XXXIX. Farbstoffe. XL. Gespinnstfasern. XLI. Papier. XLII. Photographie. XLIII. Apparate. XLIV. Bücherschau.

A TEXT-BOOK OF ASSAYING for the use of those connected with Mines. By C. BERINGER, F.C.S., F.I.C., late Chief Assayer to the Rio Tinto Copper Co., London, and J. J. BERINGER, F.C.S., F.I.C., Lecturer to the Mining Assoc. and Inst. of Cornwall, &c. London: Chas. Griffin & Co., Exeter Street, Strand. 1889.

This is an 8vo volume, bound in cloth, and containing 385 pages of Subject-Matter and an Appendix of six pages, followed by an Alphabetical Index; with the text are interspersed 82 wood engravings.

In their preface the authors state that "Under the more important methods they have given the results of a series of experiments showing the effects of varying conditions on the accuracy of the process." "Such experiments are often made by the assayer, but seldom recorded." Then they add, "Statements like those generally made—that 'this or that substance interferes'—are insufficient, 'it is necessary to know under what conditions and to what extent.'"

The authors consider "the practice of naming a process after its discoverer, which has recently much developed, is proving objectionable, and that though we may speak of a Bunsen's burner or a Liebig's condenser, yet when matters are pushed so far that the chemist is directed to 'Finkenerise,' a residue, or to use the 'Reichert-Meißl-Wollny process,' it is time to stop." Hence they have omitted to carry out such an association in referring to the processes they cite.

The work is subdivided as follows:—PART I.—I. Introductory. II. Methods of Assaying.—Dry Gravimetric Methods. III. Wet Gravimetric Methods. IV. Volumetric Assays. V. Weighing and Measuring. VI. Reagents. VII. Formulae, Equations, &c. VIII. Specific Gravity. PART II.—IX. Silver, Gold, Platinum, Mercury. X. Copper, Lead, Thallium, Bismuth, Antimony. XI. Iron, Nickel, Cobalt, Zinc, Cadmium. XII. Tin, Tungsten, Titanium. XIII. Manganese, Chromium, &c. XIV. Earths, Alkaline Earths, Alkalis. PART III.—XV. Oxygen and Oxides.—The Halogens. XVI. Sulphur and Sulphates. XVII. Arsenic, Phosphorus, Nitrogen. XVIII. Silicon, Carbon, Boron. APPENDIX.—Various tables of Atomic Weights and other Constants, Thermometric Scales, Strengths of Acids, and Ammonia, with Corresponding Specific Gravities.

The price of the work is 10s. 6d.

PROMETHEUS. ILLUSTRIRTE WOCHENSCHRIFT ÜBER DIE FORTSCHRITTE DER ANGEWANDTEN NATURWISSENSCHAFTEN. Herausgegeben von DR. OTTO N. WITT. Berlin: S.W. Rudolf Mückenberger. London: H. Grevel, 23, King Street, Covent Garden.

THIS new and elegantly arranged Journal of general and applied science made its first appearance in 1889. Judging from the first ten numbers that have appeared, it would seem, put briefly, that the leading idea running through the compilation is the popularisation of science by bringing it in contact with all the current events of the day in which it plays a part. For example, in the opening issue, the first article is one on "Brown Coal"; the second, illustrated with excellent engravings, on the "New Double-Screw Steamer of the Hamburg and American Steamship Company"; the third, on "New Metals and Alloys"; and the fourth, on the new "Automatic Electric Light Producer" of the Great Western Railway Company's service of trains (illustrated). In subsequent numbers many articles relating to Chemical Manufactures and Industry appear, and they are treated in all cases so as to combine accuracy, elegance of style, interesting reading, and admirable illustration. The Journal is a weekly one and the price is 3 marks (3s.) per quarter.

CHEMISCH-TECHNISCHE ANALYSE. Handbuch der Analytischen Untersuchungen zur Beaufsichtigung des Chemischen Grossbetriebes und zum Unterrichte. Herausgegeben von Dr. JULIUS POST, Professor an der Technischen Hochschule zu Hannover. Zweite vermehrte und verbesserte Auflage. Erster Band, Vierte Lieferung. Zweiter Band, Erste Lieferung. 1890. Braunschweig: Druck und Verlag von Friedrich Vieweg und Sohn. London: H. Grevel and Co., 33, King Street, Covent Garden.

PART IV. of the first volume of this work with Part I. of the second volume have just made their appearance. For notices of the earlier issues of Vol. I. (see this Journal, 1888, 872). The prices of the two parts now noticed are, Part IV., 5s. 6d.; and Part I. of Vol. II., 3s.

PART IV. (conclusion of Vol. I.).—This volume treats of Inorganic Acids, Alkalis and their salts, and Chloride of Lime, and is mainly devoted to the products of the Sulphuric Acid and Alkali manufacture with the allied industries. It contains 195 pages of subject-matter, and is illustrated with numerous excellent wood engravings.

As regards the distribution of the text, 56 pages are devoted to Sulphuric Acid and Sulphates; 3 to Nitric Acid; 15 to various Salts and the Stassfurt Industry; 4 to Ammonia and its Salts; 7 to Hydrochloric Acid; 26 to Soda and its Salts; 4 to the Regeneration of Sulphur from Alkali Waste; 9 to the Potassium Salts and Potash; 13 to Chloride of Lime, and 23 to Mannures.

PART I., VOL. II.—This is a treatise on (1) Lime, Gypsum, and Cements; (2.) Porcelain and Earthenware, and (3.) Glass. To Group 1, about 30 pages are devoted; to Group 2, 50 pages, and to Group 3, 22 pages.

A HANDBOOK OF QUANTITATIVE ANALYSIS by JOHN MILLS and BARKER NORTH. London: Chapman and Hall, Ltd., Agents to the Science and Art Department. 1889.

SMALL 8vo. volume, bound in cloth, containing Preface, Table of Contents, 203 pages of Subject-Matter, an Appendix of 5 pages, and an Alphabetical Index.

The scope of the book is stated in the Preface to be such as to meet the requirements of students entering for the Honours Examination in Inorganic and Organic Chemistry of the Science and Art Department. It is also stated to be sufficient for the Associateship of the Institute of Chemistry, the B.Sc. and M.B. Practical Examinations of the University of London.

The work is classified as follows:—I. Introduction. II. Simple Examples of Gravimetric Analysis. III. Simple Examples in Volumetric Analysis. IV. Simple Examples of Quantitative Separation. V. Analysis of Alloys. VI. Analysis of Minerals, Ores, &c. VII. Analysis of Commercial Products. VIII. Assaying by Cupellation. IX. Analysis of Water. X. Gas Analysis. XI. Ultimate Organic Analysis. Appendix, with useful explanation and description of Filter Pumps; Modes of Preserving Platinum and Silver Vessels; Mending Platinum Vessels; Cleaning Platinum Vessels; Recovery of Platinum and Silver Residues, &c.

The text is illustrated with 31 wood engravings. The price of the book is 3s. 6d.

## Trade Report.

### TARIFF CHANGES AND CUSTOMS REGULATIONS.

#### SWITZERLAND.

##### *Classification of Bauxite in Customs Tariff.*

Bauxite (mixture of hydrated alumina, hydrated oxide of iron, and silicic acid), raw or ground.—Category 160. Duty free.—*Board of Trade Journal.*

#### AUSTRIA-HUNGARY.

##### *Customs Classification of Hydrosulphite of Indigo.*

According to the *Moniteur Belge* for the 23rd November, the Austro-Hungarian Customs authorities have decided that the substance called *cure d'hydrosulfite* or hydrosulphite of indigo is classified under Category 324 of the tariff, and pays a duty of 4 florins per 100 kilogrammes.—*Ibid.*

#### BULGARIA.

##### *Provisional Commercial Arrangement with United Kingdom.*

The following is a copy of a notice issued by the Foreign Office on the 31st December last:—

An arrangement has been negotiated by Mr. O'Connor, Her Majesty's Agent and Consul-General at Sofia, by which British goods will be admitted into Bulgaria on and after the 1st January 1890 upon payment of Customs duties at the rate of 8 per cent. *ad valorem*.

Alcohol, gunpowder, salt, and tobacco are not included in this arrangement, but these goods of British origin will receive most favoured nation treatment.—*Ibid.*

### EXTRACTS FROM DIPLOMATIC AND CONSULAR REPORTS.

#### SULPHUR MINING IN SICILY.

The following information respecting sulphur mining in Sicily is extracted from the last report of the United States Consul at Catania:—

"The mine district in Sicily comprises the provinces of Catania, Caltanissetta, Girgenti, and the commune of Lercara, in the province of Palermo. There are in all 567 mines, 191 of which are closed, on account of being remote from the railway lines, and carrying the mineral on the back of mules or donkeys does not pay to work them.

"On the commercial point of view I may give some summary details on the working and on the method of smelting in use, whilst on the scientific point the matter has been splendidly treated by illustrious geologists of Italy, Great Britain, Germany, and France.

"The interior of a mine is carried with pillars and vaults (here commonly called 'colonne, pasture, and tetti') in the same manner as a coal mine. There you see the same galleries, the same portable underground rails, the same small waggons by means of which the rock (brimstone ore) is carried to the vertical or inclined shaft and through this by means of steam engines is lifted up and piled on the open soil.

"The galleries in the mines are sustained by pillars of rocks superposed one upon the other, and there are galleries one under the other until the stratum is exhausted. When the mine has surpassed the depth of 40 metres, water is generally found, and the hauling engine is used also for pumping. The air of the interior of a mine is generally impregnated with gases, often inflammable, and the first care

in opening a new mine is in getting a current of air. This is obtained by opening a second pit-hole communicating with the first.

"Safety-lamps have been introduced everywhere in order to prevent explosion. By a recent law every mine is to be supervised by a mineralogical master (*capominatore*) in order to prevent disasters, and to hold the fitters, owners, or contractors responsible for same, or for accidents happening to workmen when labouring. On the other side, owners or fitters, by means of a small premium paid to the 'Cassa Nazionale sugli Infortuni' (National Insurance Bank) against accidents happening to labourers are able to stand such losses. In those mines where there are no engines the mineral is carried in bags by boys on stairs on the inclined shaft up to the open soil. When the engines are in use it is hoisted up in waggons.

"The melting of brimstone is only allowed by the Italian Government from the 24th of June till the end of October, as the evaporating acids injure the grain fields.

The smelting process is very simple and takes place at mines. No machinery, no engines are to be seen; the ore is simply put in kilns (*calcaroni*) of the capacity of from 100 to 1,000 cubic metres, filled with lumps of ore and covered with earth, in order to prevent excessive evaporation. The mass is then lighted on the highest point of the inclined line at the bottom of the kiln. When fire has made a good progress in the kiln it is covered completely, and slow internal combustion commences, a good quantity of the mineral acting as fuel for heating and smelting the whole mass. In about five days a hole is made at the lowest point of the inclined bottom of the kiln, and the sulphur flows out in a liquid state into small wooden forms of the capacity of about 65 kilogrammes, which are filled continuously one day after the other until the kiln has ceased to flow. The liquid brimstone is then kept for about three hours in the forms, just sufficient time for cooling and changing into a solid state. In cooling, the sulphur contracts, and falls easily out of the forms. It is then ready to be sent to the nearest harbour for shipping. It is graded into seven different qualities, from the finest yellow to the darkest brown, and denominated: Lieata, first quality; best, second unmixed; good, second unmixed; current, second unmixed; best, third unmixed; good, third unmixed; current, third unmixed. When the ore is very rich, steam is used instead of fuel, and different systems, covered by patents, are adopted. The work is paid by cubic measure of mineral (ore) brought out of the mine. The work in the mine is considered unwholesome; as proof thereof it is to be remarked that in those provinces of Sicily where mines are abundant, the greatest number of young men are deformed and unfit for military service.

"The number of workmen employed in a mine is, according to the importance of the same, from 4 to 400 men and boys, working, on an average, eight hours, and earning about 3 lire for men and 1½ lire for boys (1 lira = 9 *d.*). The miner, with his small earning to support his family, has to live very economically. Miners pay no personal taxes except the commune tax, which is included in the price of victuals. Every mine has a gang of boys to carry the ore out of the mine where the work is not done by machinery.

"In the last years the prices of sulphur have gone down sensibly, owing to the adoption of steam-engines in the mines, to the opening of the railroads, which have reduced the cost of transportation to the harbours, once so costly because done by carts, mules, and donkeys, and to the superabundance of production in comparison with consumption and export.

"I think prices will keep low for many years, unless speculation on the markets should bring a fictitious rise of a short duration. It is my belief that few, if any, countries can compete in cheapness of hand-labour with the Italian workmen, well known everywhere for their sobriety.

"The refinement of brimstone is made with different systems (patented), but on the same principle. Brimstone is put into retorts and through a small fire, is evaporated, and condensed in cooling ovens. The grinding is done in different ways, all kept secret. Rolling, crushing, or vertical stone-mills are used; but I should never advise American buyers to import ground brimstone, as very often

it is adulterated with sundry stuffs, such as chalk, earth, strontia, &c., and dyed with chromate of yellow. Many sulphur mines have been worked in Sicily by foreigners, but very few of these have succeeded in getting any profit.

"Sicilians work the mines very economically, and only for large and rich mines steam-engines are used, whilst foreigners begin always with many expenses, without ascertaining the thickness and extent of the stratum. Owners very seldom sell their mines, except at exorbitant prices. The general usage is to lease the mine to fitters (*gabellotti*), getting from 10 to 40 per. cent. of the yield of brimstone during the time of the lease, which runs from 2 to 40 years. On the mines there is only one tax, the land tax. This is paid to the Government, not on the value of the over-soil, but on that of the under-soil, according to the formed working mine."—*Ibid.*

#### THE CAPE COLONY.

The statistical returns show that the trade of the Cape Colony has increased considerably in the year 1888. The prosperity of the Cape Colony and Natal has been greatly stimulated by the opening up of the Transvaal goldfields, with its accompanying large influx of foreign population. Kimberley, with its diamond mines, saved Cape Town; and the Transvaal, with its gold mines, will save the Colony. The rapid advancement of South African wealth and commercial prosperity, and in the development of various manufacturing industries, hinges entirely on the prosperity of the mines of the Transvaal. Any hindrance or check to them would react most fatally on colonial prosperity.

The declared value of pharmaceutical goods imported into Cape Colony increased from 59,595*l.* in 1887, to 76,764*l.* in 1888; oilman's stores, from 140,988*l.* to 162,110*l.*; paints, from 18,312*l.* to 20,494*l.* The exports of aloes grew from 152,367 lb. to 657,785 lb.—*Chemist and Druggist*.

#### CHINA.

##### *Cassia Lignea.*

The principal cassia-growing district in China is Loting, in the province of Kwang-tong, and a good deal is also produced in the province of Kwang-si. About 80,000 piculs of cassia are exported yearly from Canton and Pakhoi. Until 1887 the trade was a fairly prosperous one, but since then the profits have fallen off, so far as the two centres named are concerned, because the junks carry the cassia at a very low rate from the growing districts directly to Hong Kong, and tranship it there in the European steamers, whereby the spice is less liable to deterioration than when it is handled more frequently.

##### *Musk.*

The exports of musk from Shanghai during the last two years have been as follows:—

	1888.	1887.
To London .....	Catties, 375	Catties, 602
Mar-elles .....	908	770
New York .....	575	285
Hamburgh .....	119	71
	2,103	1,728

About 75 per cent. of this was Tonquin, 20 per cent. Yunnan, and 5 per cent. Cabardine musk. Yunnan musk is only brought to market in small quantities nowadays.

##### *Galls.*

The most noteworthy feature of the trade in nutgalls in 1888 was the increase of the shipments to Germany from 6,300 piculs in 1887 to 13,438 piculs in 1888, and the decrease of the exports to London, which were 12,680 piculs in 1887, and only 9,094 piculs in 1888.—*Ibid.*

#### GUATEMALA.

##### *Drug Cultivation.*

The Government is offering a good many inducements to agriculturists to take up the cultivation of economic plants, including many varieties of drugs. Anyone desirous of cultivating sarsaparilla, rubber, or cacao, and not having land for the purpose, can, on requisition, be put in possession of a suitable lot. The following rewards are offered to agriculturists who at the end of eight years can show healthy plantations of cinchona (*calisaya* or *succirabra*):—154*l.* for 1,000 plants eight years old to the first cultivator, 77*l.* to the second, 36*l.* 10*s.* to the third, and 15*l.* 10*s.* to all others. A premium of 4*l.* for a term of three years is given for every 150*l.* of indigo grown and made in the country, and at the end of ten years after commencing the cultivation a reward of 11*l.* 10*s.* for every thousand gum trees planted out, of five years old.

##### *Cochineal.*

The industry of cochineal raising has almost entirely disappeared from the country. For thirty years cochineal was the principal article of export, but now what little is grown is used for native consumption.

##### *Coca.*

A quantity of coca seed was imported from Peru in 1888, for distribution among the people in a suitable zone for its growth; but the result was unsatisfactory, from the bad quality of the seed, and fresh means are being taken to extend the cultivation of this plant.

##### *Vanilla, Cinchona, &c.*

The vanilla plant grows wild on the mountains all over the country. The quality of the vanilla is good, but though it figures as an export it is not cultivated for that purpose. Peruvian bark has proved a failure, and the cost of introducing the tree has been practically lost. Indigo works are subsiding in the country; a few still exist in the east, but though means are taken to encourage them, only 93*l.* of indigo was exported in 1888.—*Ibid.*

#### JAPAN.

##### *Camphor Adulteration.*

In camphor the quantity exported from Nagasaki was some 300,000 lb. less than the previous year, the figures being:—1887, 2,008,028 lb., value 38,200 *dols.*; 1888, 1,689,452 lb., value 39,276 *dols.* Prices ruled high, but the trade is being gradually injured by the systematic adulteration carried on. Owing to the higher prices ruling in 1888, salt, rice, flour, and water were used in considerable quantities, in some cases amounting to nearly 40 per cent. of the whole.

##### *Drug Imports.*

The imports of foreign drugs into Nagasaki increased from 1,823*l.* in 1887 to 6,827*l.* in 1888.

##### *The Trade for 1888.*

The foreign trade of Japan for 1888 shows a far greater expansion than has been obtained in any previous year, the figures being:—

	1888.	1887.
Imports .....	£ 11,395,353	£ 8,905,320
Exports .....	10,667,266	8,184,322

By far the greater portion of this increase falls to the United Kingdom. The following figures are of interest to the drug trade:—



*Imports.*

	1888.	1887.
	£	£
Druzs .....	282,540	256,949
Dyes and paints .....	117,750	110,315
Glassware .....	56,965	42,719
Scientific instruments .....	70,915	47,491

*Exports.*

	1888.	1887.
Camphor .....	Tons 2,549	3,816
Japan wax .....	" 1,495	1,805
Antimony .....	" 1,300	2,026
Drugs .....	Value £ 29,399	12,743

—*Ibid.**Exports in 1888.*

	England.	Germany.	America.	Austria.	France.	Other Countries.	Total Value.
Antimony.....	Sacks 7,961	..	..	..	..	..	£ 28,659
Chrome.....	Tons ..	..	2,480	..	152	..	19,528
Sponges .....	Bales, sacks, cases 6,493	..	..	3,978	579	497	83,138
Galls.....	Sacks 998	604	10	209	174	579	19,296
Gum.....	Cases 73	..	140	86	316	82	4,182
Mastic .....	" 41	..	..	..	..	..	1,230
Olive oil .....	Barrels 128	..	1,128	276	654	225	24,688
Opium.....	Cases 719	4	1,459	55	239	552	242,249
Liquorice root .....	Bales 7,346	..	47,859	..	2,098	10	50,135
" paste .....	Cases 5,859	..	..	189	..	187	19,923

—*Ibid.*

## UNITED STATES OF COLOMBIA.

*Drugs and Patents.*

French and American patent medicines have a very large sale at Cúcuta, as drugs are much needed, and the medical profession is largely in the hands of quacks. Drugs and chemicals are also paying articles of import. The excessive and irrational duties almost preclude the importation of many foreign articles; toilet soaps, tooth-brushes, india-rubber goods, &c., for instance, are excessively dear.—*Ibid.*

## VENEZUELA.

*Copaiba.*

The copaiba tree is so universally distributed throughout the forest in the region of which Maracaibo is the principal port, that it alone should produce a revenue sufficient to sustain the population of the province.

*Copaiba Balsam.*

From Ciudad Bolívar, one of the principal trading centres in Venezuela, it is reported that the advance in the price of copaiba balsam has caused much greater attention to be paid to the collection of this article than was the case before. During the year 1888 17,488 kilos. of this article were exported.

*Touquin Beans.*

The export of Touquin beans has also increased, having been 21,772 kilos. A large part of the 1888 crop was destroyed by the excessive heat.—*Ibid.*

## TURKEY IN EUROPE.

*Antimony.*

Three antimony mines have been recently opened in the Salonica district, and, on the whole, are successfully worked—two by British subjects and one by an Austrian company. The ore, of which about 300 tons have already been exported—chiefly to the United Kingdom—is of very good quality, and costs about 10*l.* per ton "F.o.b." at Salonica.—*Ibid.*

## TURKEY IN ASIA.

*The Drug Trade.*

A very large trade in druggists' goods is carried on from Smyrna, as may be seen from the following figures. Of drugs last year the following quantities were imported:—From Great Britain, 1,043 cases; Germany, 57 cases; Austria (Trieste), 624 cases; Belgium, 47 cases; France, 1,545 cases; Italy, 287 cases; other countries, 178 cases; total, 6,988 cases; value 63,904*l.*

## WEST COAST OF AFRICA.

*Export Duties.*

Certain export duties are still kept up in the British colony of Sierra Leone, in the Niger Territories, and in Gambia. In the French and German settlements on that coast, however, there are none, nor in Lagos, the Gold Coast, and Liberia. The principal duties are:—

Articles.	Gambia.	Sierra Leone.	Niger Territories.
	£ s. d.	£ s. d.	£ s. d.
Palm oil ..... per gallon	Nil.	0 0 1	0 0 1
Palm kernels ..... per cwt.	"	0 0 2	0 0 2
Ground nuts, shelled ..	0 0 4	0 0 3	Nil.
" " unshelled ..	Nil.	0 0 2	"
Beniseed.....	"	0 0 2	"
Kola nuts .....	"	0 5 0	"
Gum .....	"	0 2 0	"

—*Ibid.*

## MEXICO.

*Drug Exports.*

The bulk of the drugs exported from Mexico is shipped to the United States, though from there a large part finds its way to the United Kingdom. The following figures are of interest as showing the comparative value of the exports during the last five years:—

Articles.	Annual Average, Five Years ending June 30, 1884.	Year ending June 30, 1886.	Year ending June 30, 1887.	Weight.
	Dollars.	Dollars.	Dollars.	Tons.
Aloes (essential oil)...	1,576	..	10,121	14.5
Cascarilla.....	..	..	1,264	8.0
Chicle (chewing gum).	73,288	127,906	285,930	570.4
Honey.....	74,559	47,564	35,719	630.8
Indigo.....	64,984	95,269	50,289	34.7
Jalap.....	30,324	19,541	10,926	63.5
Sarsaparilla.....	49,569	95,870	55,669	482.6
Vanilla.....	425,150	419,178	555,113	49.5

The essential oil of aloes is exported from Vera Cruz in about equal proportions to Great Britain and France; the chicle gum, which goes all to the United States, where it has obtained an enormous popularity, lately comes nearly all from Tuxpan, with an occasional shipment from Tampico and other ports. Honey is also shipped to the States from Tuxpan and Tampico. The principal port for indigo is Tonala, but Salina Cruz and Vera Cruz also participate to some extent in the business. About two-thirds of the Mexican indigo are sent to France, and most of the remainder to Germany. As regards sarsaparilla the export figures bear witness to a heavy decline in the output, but with jalap the case is rather the reverse. Of this root more than four-fifths come from Tampico, about one-eighth from Tuxpan, and the inconsiderable residue from Vera Cruz and Matamoros. The United States receive one-half of the root, France a quarter, and the rest goes to England and Germany. Of the Mexican vanilla, which is brought into commerce mostly *via* Tuxpan, and partly through Vera Cruz, nearly all goes to the States, and a little to France.—*Ibid.*

## SIAM.

*Dyeing Materials.*

The export from Bangkok of dye-woods, gamboge, indigo and other materials used in dyes, shows no increase, the fact being that chemical dyes are now rapidly supplanting the vegetable. The smallness of the amount of cutch exported is remarkable. In Burmah this article of jungle produce has risen to a very important item in the export, and there is every reason to believe that there is plenty of cutch in Siam also. It would be worth the while of the Siamese Government to investigate the reasons why more is not made of this source of natural wealth in the country.—*Ibid.*

## SPAIN.

*The Cochineal Industry.*

Cochineal still continues to hold its place as the principal article of export from the Canary Islands. During 1888 the following quantities were shipped:—To England, 220,339 kilos.; France, 132,127 kilos.; Germany, 117,460 kilos.; Spain, 6,912 kilos.; Africa, 5,432 kilos.; total, 482,260 kilos.; equal to 1,048,110 English lb. This trade is gradually declining every year, owing to the unremunerative prices offered in Europe, and endeavours are constantly being directed to the finding of a substitute for this product.

*Sulphate of Copper.*

The application of sulphate of copper has been found to be useless against the mildew, and it is believed in some quarters that this remedy has only a preventive and not a curative character, though others assert that the remedy failed because it was applied too late, and, besides, not only is the unfortunate vine attacked by mildew, but it is also exposed to the ravages of four other phases of destruction, well known to all acquainted with vine-growing.

## GERMANY.

*Profits in Chemical Industry.*

The German chemical industry has been on the whole in a very flourishing condition during the year 1888. According to recently published statistics the number of factories falling under this description has increased from 4,235 in 1887, to 4,464 in 1888, that of the average number of hands employed from 82,211 to 84,315, and the total of the wages paid from 62,710,380 to 65,644,013 m., the average wages of each hand having been 757.13 m. in 1887, and 773 m. in 1888. The dividends declared by the chemical factories carrying on business as limited liability concerns during the last five years have been as follows:—

	1884.	1885.	1886.	1887.	1888.
	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Alkali and heavy chemicals.....	6.8	5.77	5.79	6.02	7.45
Pharmaceutical and fine chemicals.....	13.26	12.63	14.14	16.28	13.81
Coal tar products.....	11.05	7.03	9.94	13.25	15.44
Explosives.....	7.84	10.8	16.46	14.33	15.58
Artificial manures.....	6.21	3.07	2.51	5.32	7.97

The following table shows the proportion of profits of the limited companies during the last five years:—

Year.	No Dividend Declared.	Under 5 per Cent. Dividend.	5 to 10 per Cent. Dividend.	Over 10 per Cent. Dividend.
1884	Per Cent. 25	Per Cent. 23	Per Cent. 33	Per Cent. 19
1885	34	22	28	16
1886	33	24	13	29
1887	33	12	30	20
1888	32.5	16.25	23.25	25

## MISCELLANEOUS TRADE NOTICES.

## TRADE WITH THE BLACK SEA.

The *Deutsches Handels-Archiv* informs its readers that the articles which find the most ready market in the Turkish ports of the Black Sea are Turkey-red cotton thread, cloth stuff, whether wholly or partly of wool, cheap hardware, chemical products, dyes, and beer, the consumption of which is rapidly on the increase. There is room, says the German paper, for a very great development of trade in the Black Sea, and merchants would do well to let themselves be represented by active and capable agents who have made themselves acquainted with the character of Turkish trade and the requirements of the inhabitants.—*Board of Trade Journal.*

## THE DEVELOPMENT OF YUCATAN.

The Belgian Consul at Merida has forwarded to his Government a report on the commercial position of the State of Yucatan, from which we extract the following particulars:—

After a long period of decline and impoverishment, the last two or three years have seen a very remarkable revival of prosperity throughout the State of Yucatan, and in particular in Merida, its capital, and this division of Central America must now be regarded as offering a market to European goods. Merida, which is united to the sea at the port of Progreso by a short line of railway, now contains upwards of 50,000 inhabitants, and publishes 10 newspapers. The principal squares and the Hidalgo Park are lighted with electricity, and a considerable number of fine public buildings have recently given to the city an air of importance. It was the year 1888 in which the condition of Yucatan became to a marked degree ameliorated. The main industry of the country is the manufacture of the textile hemp called henequen, the price of which rose rather suddenly to the unprecedented figure of three piastres the arroba. In consequence, very large commercial transactions were effected in 1888 and 1889 by the capitalists resident in Merida.

The product which occupies the second place after henequen is bluewood or campéchy wood, the amount of which exported in 1889 has greatly exceeded previous returns. The whole import trade of 1888 showed a considerable increase, but the merchants of Europe seem as yet not to have waked up to the idea that a market for their goods has opened up in Yucatan. At present the only countries from which goods are imported into the State are the United States, England, Germany, France, and Spain.—*Ibid.*

## CINCHONA IN MADRAS.

The Calcutta *Englishman* (overland mail) for the 3rd December says:—

"From the report on the Government cinchona plantations in the Nilgeries we gather that 110,162 lb. of bark were taken during the past year; and adding this to the balance on hand from the previous year, there was a total of 254,130 lb., of this 18,350 lb. were disposed of by using 17,300 lb. for the manufacture of febrifuge, and the remainder was sold to the Medical Stores Department. The actual money realised by sale of bark, &c. amounted to Rs. 3,601, while the value of febrifuge supplied free of cost to medical stores was about Rs. 3,000; and that supplied to the Mysore Government Rs. 2,417, making in all a total revenue of about Rs. 9,000. The actual expenditure amounted to Rs. 69,493, so that the Government suffered a clear loss of something over Rs. 60,000 on the working of the cinchona plantations during the past year.—*Ibid.*

## CINCHONA PLANTING.

*Van Romunde. Nederl. Tijdsch. Pharm. Chem. en Toxicol.* 1889, 1, 259.

During the year 1888 the quantity of bark collected in Java was 380,899·5 kilos., of which 1,109 kilos. were used in the country itself, the remainder being sent to Holland. The amount for the first quarter of 1889 was 87,500 kilos.

—T. L. B.

## PRODUCTION OF COBALTFEROUS MATT OR CHEEP IN NEPAL.

Communications have been received at the Board of Trade from the India Office enclosing certain memoranda from the Revenue and Agricultural Department of the Government of India at Simla relating to the production and analysis of a substance called "cheep" which is obtained close by the copper mines in Nepal. The name of the locality is "Kachipatar" Argah Zillah, Sowrobhar, about 80 miles north of Deolha, on the estate of Mr. D. Ricketts.

A sample of this substance was sent to the Director, Geological Survey of India, for analysis, and in a report by the latter he says:—"The sample of cheep has been analysed with the following results:—Loss at 100° C. 0·40; insoluble in acids 0·70; sulphur, 20·41; cobalt, 13·97; iron, 62·82; total, 98·30.

"Cobalt is principally used in the state of oxide for colouring pottery and glass, and 13·97 of the metal is equivalent to 19·69 of oxide. Assuming therefore that the specimen analysed is a fair average sample, the cheep would yield nearly a fifth of its weight of cobalt oxide. It appears from the 'Mineral Resources of the United States' for 1886 (the last available) that 14,215 lb. of cobalt oxide were imported in that year, valued at 22,757 dols., which is at the rate of 717l. per ton. According to Mr. Ricketts's letter of the 23rd January 1888, to your address, the 'cheep' is sold at Kachipatar, the place of production, at Rs. 30 to Rs. 35 per maund, and at Deolha at Rs. 40 to Rs. 50. These prices are equivalent (taking the rupee at 1s. 4d., and 27·22 maunds as equivalent to one ton) to 54l. to 64l., and 73l. to 91l. a ton. As five tons of 'cheep' therefore (containing one ton of cobalt oxide) would be worth at the above prices 270l., 320l., 365l., and 455l., and the cobalt oxide would be worth, after extraction, about 700l., there would appear to be a fair margin to allow of the 'cheep' being exported to England and treated there. That is to say, if it is obtainable in sufficient quantity. The metallurgical treatment of cobalt ores being rather difficult, it does not seem probable that the natives of Nepal are acquainted with any process by which a fairly pure oxide can be obtained.

"In reply to a communication respecting the probable supply of cobaltiferous matt from the Secretary to the Government of India, Foreign Department, Simla, to the President in Nepal, the latter states:—"In reply to your letter asking for a report as to the probable supply of 'cobaltiferous matt,' which would be obtainable if a demand were to arise for the substance among cobalt refiners in England, I have the honour to state that Mr. Ricketts, of Deolha Estate, Gorakhpur, though unable to estimate in figures the quantity that would be procurable if the above contingency were to arise, is of opinion that the supply would, in all probability, increase with the demand, which is at present about 400 maunds annually."—*Board of Trade Journal.*

## INDIA-RUBBER MONOPOLY.

The *Provincia do Para*, a journal of Para, throws some light on the new tax of 20 reis per kilo. laid on india-rubber. It will be remembered that since January 1st that tax is paid, not to the State, nor to the province, but to the Companhia Mercantil. The Governor of the Province of Para received on November 30th a petition signed by numerous commercial men of Para, begging that a tax of 20 reis per kilo. should be laid on india-rubber for a period of five years, and that it should be paid to the Companhia Mercantil "as a compensation for the efforts, and especially for the sacrifices, that company is making to support our market, and to keep up the price of our principal product for exportation, which is threatened with a permanent depreciation of its value, owing to the manœuvres of a group of powerful speculators for the fall, who have, during the last few years, sought to absorb our market almost completely."

The petition also declared that, in supporting the price of india-rubber, the Companhia Mercantil effectually aided in insuring a progressive increase of the public revenue. The Companhia Mercantil on the same day addressed a similar petition to the Governor, and assured him that the company would employ the proceeds of the tax solely to cover any loss that might arise from its operations for supporting the price of india-rubber, and that any balance that might remain would be handed to the State.

Notwithstanding the protests of numerous delegates from the opposition group of commercial men at Para, who had an interview with the Governor, that functionary decreed the levying of the additional tax of 20 reis per kilo., to be paid to the Companhia Mercantil. M. Justo Leido Chermont, Governor of Para, who signed this decree, is, it is stated, a large shareholder and President of the Council of Administration of the Companhia Mercantil. The merchants who protested against the tax have sent a petition to General Deodoro da Fonseca, Chief of the Provisional Government, demanding its abolition, and they seem to be pretty confident that the decree will be repealed and the sums paid by them to the Companhia Mercantil shortly returned to them.—*Standard.*

## THE BRAZILIAN INDIA-RUBBER INDUSTRY.

A newspaper in Providence, U.S.A., where extensive rubber works are established, has received advices from Para stating that the concession recently obtained from the Provincial Government by the Companhia Mercantil de Para, permitting it to levy a duty of 20 reis per pound upon the entire export of Para india-rubber, has been cancelled.—*Standard*.

## RECENT UNITED STATES STATISTICS.

## COPPER PRODUCTION BY STATES.

	1888.	1889.	
	Lb.	Lb.	Long Tons.
Lake Superior .....	86,581,124	86,000,000	38,333
Arizona .....	31,497,300	32,000,000	14,286
Montana .....	27,897,968	105,000,000	46,875
New Mexico .....	1,631,271	3,400,000	1,518
California .....	1,570,021	1,500,000	750
Colorado .....	3,300,000	3,000,000	1,330
Utah .....	2,131,047	2,400,000	1,071
All other sources .....	3,241,725	3,000,000	1,330
Domestic production....	227,853,456	236,500,000	105,580
From imported ores....	5,000,000	5,100,000	2,277
Total production .....	232,853,456	241,600,000	107,857
Stocks, beginning of year....	40,000,000	75,000,000	37,482
Available supply .....	272,853,456	316,600,000	144,337
Deducts exports in ore, matte, ingots .....	18,000,000	82,000,000	36,607
Consumption .....	119,853,456	169,600,000	75,714
Stocks on hand end of year ..	75,000,000	75,000,000	29,016

## HEAVY CHEMICALS.

The United States Bureau of Statistics reports the following importations for the first 10 months of 1889 and 1888:—

Article.	1889.		1888.	
	Quantity.	Value.	Quantity.	Value.
Bleaching powder .....	Lb. 79,575,222	Dols. 1,260,204	Lb. 80,403,166	Dols. 1,287,670
Soda bicarbonate .....	1,115,742	17,487	1,354,525	21,045
Soda ash and sal soda ...	250,270,100	238,488	240,000,391	2,336,545
Branstone .....	Tons 120,064	1,823,497	97,015	1,551,299

## METALLIC PRODUCTS OF THE UNITED STATES.

	1888.	
	Quantity.	Value.
Pig iron, spot value, long tons.....	6,489,738	Dols. 167,000,000
Silver, coining value, troy oz. ....	45,783,632	59,195,000
Gold, coining value, troy oz.....	1,604,927	33,175,000
Copper, value at New York, lb. ....	231,270,622	33,853,954
Lead, value at New York, tons of 2,000 lb. ....	180,555	15,924,951
Zinc, at New York, tons of 2,000 lb.....	55,903	5,500,855
Quicksilver, at San Francisco, flasks....	33,250	1,413,125
Nickel, at Philadelphia, lb. ....	195,182	115,518
Aluminium, at Philadelphia, lb.....	19,000	65,000
Antimony, at San Francisco, tons of 2,000 lb.....	100	20,000
Platinum (crude), at New York, troy oz. .	500	2,000
Total .....	..	250,245,493

## THE PRODUCTION OF CALIFORNIA QUICK-SILVER MINES.

The following table shows the production of the several mines for six years past:—

Mines.	1884.	1885.	1886.	1887.	1888.	1889.
New Almaden .....	Flasks. 20,400	Flasks. 21,400	Flasks. 18,900	Flasks. 20,000	Flasks. 18,000	Flasks. 13,100
Etna .....	2,931	1,309	3,478	2,694	950	..
Napa Consolidated.....	1,373	2,197	1,769	2,880	4,035	4,500
Great Western.....	3,292	3,469	1,949	1,446	625	550
Sulphur Bank .....	890	1,296	1,449	1,890	2,164	2,150
New Idria .....	1,025	1,144	1,106	1,490	1,320	1,000
Great Eastern .....	332	446	735	689	1,151	1,350
Redington .....	881	385	490	673	123	800
Guadalupe.....	1,179	35	..	..	..	..
Brafford Consolidated .....	..	..	..	1,543	3,848	1,700
Various .....	7	392	756	455	992	500
Total flasks .....	34,913	32,973	29,081	33,760	33,250	25,650*
Lowest price per flask.....	Dols. 26'00	Dols. 28'50	Dols. 32'00	Dols. 33'50	Dols. 37'00	Dols. 40'00
Highest price per flask .....	35'00	32'00	39'00	48'00	48'00	50'00
Average per flask .....	30'50	30'25	35'50	42'25	42'50	45'00
Total Value at average price.... Dols.	975,000	970,000	1,039,000	1,425,000	1,415,000	1,154,000

\* The total production for 1889 is a near approximation.

## NON-METALLIC MINERAL PRODUCTS OF THE UNITED STATES.

	1888.	
	Quantity.	Value.
Bituminous coal, tons of 2,240 lb.....	91,106,995	Dols. 122,107,311
Anthracite, tons of 2,240 lb.....	41,624,610	89,020,183
Building stone.....	..	25,500,000
Lime, barrels.....	49,087,900	24,543,500
Petroleum, barrels.....	27,316,018	24,508,559
Natural gas.....	..	22,662,123
Cement, barrels.....	6,253,895	4,533,639
Salt, barrels.....	8,055,881	4,377,204
Limestone for iron flux, tons of 2,240 lb. ..	5,438,900	2,719,000
Phosphate rock, long tons.....	423,705	1,951,673
Zinc-white, short tons.....	20,000	1,600,000
Mineral waters, gallons sold.....	9,028,568	1,709,202
Borax, lb.....	7,580,609	453,349
Gypsum, short tons.....	96,009	430,009
Manganese ore, long tons.....	25,500	255,000
Mineral paints, long tons.....	24,000	380,000
New Jersey marls, short tons.....	600,000	300,000
Pyrites, long tons.....	54,531	167,658
Flint, long tons.....	30,000	175,000
Mica, lb.....	48,000	70,000
Corundum, short tons.....	589	91,620
Sulphur, short tons.....	..	..
Precious stones.....	..	64,850
Gold quartz, souvenirs, jewellery.....	..	75,000
Crude barytes, long tons.....	20,000	110,000
Bromine, lb.....	307,386	95,290
Feldspar, long tons.....	8,700	50,000
Chrome iron ore, long tons.....	1,500	20,000
Graphite, lb.....	400,000	32,000
Fluorspar, short tons.....	6,000	30,000
Slate, ground, long tons.....	2,500	25,000
Cobalt oxide, lb.....	12,265	18,411
Novaculite, lb.....	1,500,000	18,000
Asphaltum, short tons.....	53,800	331,590
Asbestos, short tons.....	100	3,000
Rutile, lb.....	1,000	3,000
Total.....	..	328,914,528

## RÉSUMÉ.

	1888.	
	Quantity.	Value.
Metals.....	..	Dols. 256,245,103
Mineral substances.....	..	328,914,518
Mineral products, unspecified.....	..	6,500,000
Grand total.....	..	591,659,631

## FERTILISERS.

The importations of kainit during the year aggregated 12,600 tons. This compares with the receipts during the three preceding years as follows:—1888, 17,500 tons; 1887, 6,000 tons; 1886, 16,900 tons. The syndicate price throughout the year has been 10 dols. per ton actual weight, and 9.75 dols. per ton foreign invoice weight.

The importations of muriate of potash during the year aggregated 28,445 tons. This compares with the receipts for the two preceding years as follows:—1888, 27,922 tons; 1887, 17,905 tons. The importations at the port of New York for the three years were respectively 18,030, 16,560 and 9,945 tons. The syndicate price averaged 1.80 dols. per 100 lb.

Early in the year a prominent firm of importers endeavoured to secure the free entry of high-grade sulphate of potash, claiming that the duty enforced upon it practically prohibited its use in this country, and therefore was prejudicial to the interests of importers and consumers. A final decision has not been reached on the subject as yet, and it is expected that when Congress next considers the question of the tariff it will be formally asked to place sulphate of potash upon the free list.

A notable feature of the year was the falling off in the Southern consumption of Northern fertilisers. Acid factories are being established all through the Southern States, and there is certainly no reason why the South should be dependent upon the North for its fertilisers. During 1889 the consumption of acidulated phosphate rock in the South materially increased, but Northern dealers had little or no part in it.

## NITRATE OF SODA.

The shipments to the States were 667,500 bags, against 517,500 the year before; 555,000 in 1887; 522,750 in 1886; and 270,323 in 1885.

The quantity to arrive for Atlantic ports is 320,000 bags, against 221,000 in 1889; 239,000 in 1888; and 238,500 in 1887. The total visible supply is 371,726 bags, against 310,000 in 1889; 301,940 in 1888; and 311,266 in 1887.

The quantity to arrive in Europe is 2,835 in bags, making the visible supply then 4,312,509 bags, against 3,112,000 last year and 2,927,500 in 1888.

The deliveries at San Francisco in 1889 were 80,060 bags, making total deliveries in this country 618,358 bags, against 515,000 in 1888; 534,347 in 1887; and 454,760 in 1886.

In Europe the deliveries were 4,920,000 bags, making total for the world 5,538,358 bags, against 5,227,000 in 1888; 4,164,317 in 1887; 3,522,260 in 1886; and 3,278,686 in 1885.—*Engineering and Mining Journal*.

## EXPORTS OF MINERAL OILS FROM THE UNITED STATES FOR NOVEMBER 1889.

S. G. Brock, Chief of the Bureau of Statistics, submits the following statement of the exports of domestic mineral oils from the United States for the month of November, 1889:—

*Mineral Oils, Crude.*

(Including all natural oils, without regard to gravity.)

	Gallons.	Dols.
Total for November 1889.....	8,742,471	565,968
“ “ 1888.....	9,869,357	694,416
Total for 11 months ending November 30, 1889.....	77,068,808	5,534,347
Total for 11 months ending November 30, 1888.....	73,912,428	5,185,851

*Naphthas.*

(Including all lighter products of distillation.)

—	Gallons.	Dols.
Total for November 1889 .....	1,801,399	158,124
" " 1888 .....	1,921,097	153,975
Total for 11 months ending November 30, 1889 .....	13,506,696	1,161,642
Total for 11 months ending November 30, 1888 .....	12,273,998	986,027

*Illuminating Oils.*

—	Gallons.	Dols.
Total for November 1889 .....	41,336,976	3,963,109
" " 1888 .....	39,443,136	3,177,012
Total for 11 months ending November 30, 1889 .....	504,696,921	37,372,850
Total for 11 months ending November 30, 1888 .....	414,845,229	33,880,346

—*Chemical Trade Journal.*

## THE COPPER INDUSTRY.

The Spanish Government having recently ordered the Rio Tinto Company to diminish by one-half the quantity of copper ore it was burning near Huelva, on the ground that the smoke from the furnaces was injurious to the health of that town, there has been a very general protest in the Peninsula against the edict, largely upon the ground that it may have the effect of frightening away foreign capital.—*Ironmonger.*

## THE GOLD DEPOSITS OF NOVA SCOTIA.

*E. Gilpin. Trans. Royal Soc. of Canada, 1889.*

Gold is present in two distinct formations:—

(1.) The lower graphite and ferruginous micaceous schists group, and (2.) the lower quartzite group. The former extends 1,300 m., the latter 3,500 m. The veins of ore vary from 0.32 m. to 2 m., and are very numerous at Mount Uniacke, where no fewer than 31 have been discovered; their depth has been estimated as 100—200 m. According to the latest researches their presence is not, as was formerly thought, in any way dependent on that of the neighbouring granite, which appears more recent than that of the gold formations in the vicinity of Morseland, County Harbour Narrows, &c. The granite usually carries no metal of any kind, though copper has been observed by itself near Dalhousie, Queen's County. The gold is usually accompanied by various pyrites, including copper pyrites and arsenical pyrites. At the Dufferin gold mine, on Salmon River, Halifax County, the vein varies from 0.6 to 4 m., and of 400 m. 80 m. have been worked out, yielding 55,483 tons of quartz containing 27,814 oz. of gold. The gold is widely distributed, but only here and there in paying quantities. It is now well established that the gold is always obtained from, and associated with, schists. As regards the depth to which the lodes extend, it seems probable that those rich in gold deteriorate as they descend, and on a vein suddenly ceasing to yield rich ore, one running parallel to it will be found to carry more gold with equal abruptness, but no direct proof is yet forthcoming.

—B. B.

## TRADE BETWEEN SPAIN AND THE UNITED KINGDOM.

IMPORTS INTO THE UNITED KINGDOM FROM SPAIN FOR THE THREE MONTHS ENDED 31ST DECEMBER.

Principal Articles.	Quantities.		Values.	
	1888.	1889.	1888.	1889.
Chem. products. Value £	..	..	£ 12,811	£ 12,364
Copper ore and regulus ..... Tons	25,528	19,327	573,974	424,201
Copper, unwrought ..... "	5	..	300	..
Iron ore ..... "	615,476	890,693	414,268	679,932
Lead ore ..... "	596	10	3,374	90
Lead, pig and sheet ..... "	20,868	20,881	285,552	271,067
Manganese ore.. "	1,810	3,004	5,520	7,423
Pyrites ..... "	136,588	120,050	282,582	223,235
Quicksilver ..... Lb.	403,784	412,631	50,619	43,561
Rags, esparto ... Tons	12,723	16,316	78,346	95,967
Zinc ore ..... "	220	..	550	..
Total Value.....	..	..	3,186,235	3,512,345

EXPORTS OF HOME PRODUCE FROM THE UNITED KINGDOM TO SPAIN FOR THE THREE MONTHS ENDED 31ST DECEMBER.

Principal Articles.	Quantities.		Values.	
	1888.	1889.	1888.	1889.
Alkali..... Cwt.	76,910	74,926	£ 23,828	£ 23,627
Caoutchouc ma- nufactures.... Value £	..	..	5,578	4,974
Cement..... Tons	2,715	2,131	4,711	4,190
Chemicals..... Value £	..	..	11,078	37,350
Naphtha and petroleum.... „	..	..	5,871	19,961
Glass..... „	..	..	3,093	2,229
Grease and tallow Cwt.	4,671	3,171	4,665	2,854
Manure..... Value £	..	..	26,257	51,528
Oil, seed..... Tons	67	129	1,354	2,879
„ other sorts.. Value £	..	..	1,500	3,454
Oil and floor cloth..... Sq. yds.	76,500	67,600	3,293	2,854
Painters' colours and materials. Value £	..	..	10,577	10,982
Paper of all sorts Cwt.	718	1,623	1,993	3,800
Sap..... „	679	192	684	848
Total Value.....	..	..	785,767	1,075,551

## BOARD OF TRADE RETURNS.

## SUMMARY OF IMPORTS.

	Year ended 31st December	
	1888.	1889.
	£	£
Metals.....	23,242,958	22,081,845
Chemicals, dyestuffs, and tanning materials.....	8,111,439	8,635,378
Oils.....	6,432,871	7,122,998
Raw materials for non-textile industries.....	34,722,501	43,091,671
Total value of all imports....	380,582,026	427,210,839

## SUMMARY OF EXPORTS.

	Year ended 31st December	
	1888.	1889.
	£	£
Metals (other than machinery) ....	37,155,581	40,945,735
Chemicals and medicines .....	7,436,071	7,933,519
Miscellaneous articles.....	50,989,042	33,073,187
Total value of all exports.....	233,812,607	248,094,959

## IMPORTS OF METALS FOR YEAR ENDED 31ST DECEMBER.

Articles.	Quantities.		Values.	
	1888.	1889.	1888.	1889.
			£	£
Copper:—				
Ore..... Tons	120,807	136,514	1,035,512	1,050,348
Regulus and precipitate .....	102,519	113,219	3,849,135	3,163,088
Unwrought ....	44,652	38,639	3,498,199	2,035,036
Iron and steel:—				
Iron ore .....	3,552,408	4,023,620	2,462,231	3,121,357
Iron bolt, bar, &c. ..	113,362	141,803	1,027,186	1,034,102
Steel, unwrought ..	12,058	10,870	111,024	90,865
Lead, pig and sheet ..	132,941	145,451	1,860,609	1,878,850
Pyrites .....	617,232	643,879	1,176,916	1,210,061
Quicksilver .....	5,532,774	5,074,813	616,437	562,227
Tin .....	560,989	605,735	3,520,309	2,815,049
Zinc .....	61,045	56,842	1,046,106	1,084,101
Other articles ... Value £	..	..	2,978,974	4,062,861
Total value of metals	..	..	23,242,958	22,081,845

## IMPORTS OF CHEMICALS AND DYESTUFFS FOR YEAR ENDED 31ST DECEMBER.

Articles.	Quantities.		Values.	
	1888.	1889.	1888.	1889.
			£	£
Alkali..... Cwt.	56,753	47,105	59,567	31,383
Bark (for tanners, &c.) .....	339,685	463,766	135,018	207,347
Brimstone .....	782,854	796,055	175,046	172,976
Chemicals..... Value £	..	..	1,310,194	1,416,426
Cochineal .....	7,340	8,995	48,310	50,297
Cutch and gambier Tons	28,135	25,107	704,731	678,548
Dyes:—				
Aniline .....	..	..	265,794	272,329
Alizarine .....	..	..	283,862	317,644
Other .....	..	..	11,889	21,936
Indigo .....	78,188	90,483	1,703,682	1,783,256
Madder .....	15,034	14,199	13,292	17,139
Nitrate of soda....	2,053,282	2,351,310	981,894	1,102,583
Nitrate of potash ..	353,601	328,533	500,663	287,922
Valonia .....	32,017	31,361	457,634	454,405
Other articles... Value £	..	..	1,659,833	1,822,347
Total value of chemicals	..	..	8,111,439	8,635,378

## IMPORTS OF OILS FOR YEAR ENDED 31ST DECEMBER.

Articles.	Quantities.		Values.	
	1888.	1889.	1888.	1889.
			£	£
Cocoa-nut..... Cwt.	197,773	213,470	251,327	278,057
Olive .....	18,580	22,954	674,472	818,352
Palm .....	955,369	1,019,077	547,839	1,078,605
Petroleum .....	94,477,807	102,647,478	2,558,753	2,593,173
Seed .....	16,311	17,127	413,109	463,554
Train, &c..... Tons	16,861	21,051	323,579	442,339
Turpentine .....	339,067	408,074	517,897	662,681
Other oils..... Value £	..	..	746,895	785,877
Total value of oils ...	..	..	6,432,871	7,122,998

## IMPORTS OF RAW MATERIALS FOR NON-TEXTILE INDUSTRIES FOR YEAR ENDED 31ST DECEMBER.

Articles.	Quantities.		Values.	
	1888.	1889.	1888.	1889.
			£	£
Bark, Peruvian .. Cwt.	145,216	129,600	553,411	430,563
Bristles..... Lb.	2,847,591	3,337,443	384,569	471,451
Caoutchouc..... Cwt.	218,171	236,274	2,529,436	2,612,704
Gum:—				
Arabic.....	83,371	65,368	329,036	243,117
Lac, &c.....	103,364	78,602	271,946	276,274



IMPORTS OF RAW MATERIALS FOR NON-TEXTILE INDUSTRIES FOR YEAR ENDED 31ST DECEMBER—*cont.*

Articles.	Quantities.		Values.	
	1888.	1889.	1888.	1889.
Gutta-percha .... Cwt.	22,483	18,042	£ 181,660	£ 576,893
Hides, raw:—				
Dry..... "	585,254	575,158	1,618,318	1,573,132
Wet..... "	576,176	647,250	1,353,663	1,500,155
Ivory..... "	11,757	12,051	537,671	573,170
Manures:—				
Guano..... Tons	25,952	26,894	202,131	190,783
Bones..... "	65,651	62,855	310,983	310,263
Paraffin..... Cwt.	350,927	311,086	458,553	373,185
Linon rags..... Tons	11,104	42,170	470,833	426,611
Esparto, &c..... "	217,996	215,723	1,265,815	1,083,518
Pulp of wood .... "	116,010	121,531	677,866	688,571
Rosin..... Cwt.	1,314,719	1,337,841	238,199	205,451
Tallow and stearin "	1,146,070	1,243,006	1,132,595	1,614,946
Tar..... Barrels	121,884	131,524	69,079	99,819
Wood:—				
Hewn..... Loads	1,985,219	2,989,191	1,042,107	5,635,318
Sawn..... "	4,236,084	5,918,750	9,638,077	13,112,333
Staves..... "	113,872	170,155	599,112	694,115
Mahogany .... Tons	42,620	39,858	871,738	354,179
Other articles.... Value £	..	..	9,130,779	10,197,384
Total value .....	..	..	16,722,501	46,694,971

In addition to above, drugs value \$16,700, were imported, as against \$87,122, in 1888.

## EXPORTS OF METALS OTHER THAN MACHINERY FOR YEAR ENDED 31ST DECEMBER.

Articles.	Quantities.		Values.	
	1888.	1889.	1888.	1889.
Brass..... Cwt.	75,603	107,812	£ 370,759	£ 455,909
Copper:—				
Unwrought..... "	503,932	650,318	1,956,218	1,547,151
Wrought..... "	157,932	313,123	568,557	967,973
Mixed metal.... "	159,136	306,500	494,873	816,130
Hardware..... Value £	..	..	3,168,103	2,988,202
Iron and steel.... Tons	3,906,533	4,188,588	26,416,656	29,153,161
Lead..... "	48,616	52,153	728,618	759,170
Plated wares... Value £	..	..	391,089	437,840
Telegraph wires... "	..	..	521,655	1,040,982
Tin..... Cwt.	120,826	108,477	701,776	522,759
Zinc..... "	111,768	131,616	87,653	105,239
Other articles... Value £	..	..	1,718,684	2,151,807
Total value .....	..	..	27,155,581	40,915,735

## EXPORTS OF DRUGS AND CHEMICALS FOR YEAR ENDED 31ST DECEMBER.

Articles.	Quantities.		Values.	
	1888.	1889.	1888.	1889.
Alkali..... Cwt.	6,315,000	6,635,096	£ 1,638,770	£ 1,575,319
Bleaching materials "	1,603,000	1,522,166	615,381	562,598
Chemical manures. Tons	..	320,181	1,849,035	2,043,765
Medicines..... Value £	..	..	962,154	971,117
Other articles... "	..	..	2,499,731	2,771,120
Total value .....	..	..	7,496,071	7,933,519

## EXPORTS OF MISCELLANEOUS ARTICLES FOR YEAR ENDED 31ST DECEMBER.

Articles.	Quantities.		Values.	
	1888.	1889.	1888.	1889.
Gunpowder..... Lb.	13,523,600	10,671,500	£ 363,270	£ 274,200
Military stores.. Value £	..	..	1,319,661	1,167,980
Candles..... Lb.	10,850,000	12,564,600	198,298	231,115
Caoutchouc..... Value £	..	..	1,163,271	1,121,787
Cement..... Tons	612,702	632,707	1,165,900	1,233,624
Earthenware ... Value £	..	..	1,997,225	2,092,236
Stoneware..... "	..	..	101,298	105,703
Glass:—				
Plate..... Sq. Ft.	1,319,198	3,604,125	260,867	213,991
Flint..... Cwt.	128,886	121,105	283,674	274,919
Bottles..... "	833,613	981,258	495,166	464,228
Other kinds.... "	294,884	217,358	139,631	164,553
Leather:—				
Unwrought .... "	159,138	143,119	1,393,880	1,313,681
Wrought ..... Value £	..	..	372,309	413,600
Seed oil..... Tons	78,608	68,657	1,593,912	1,490,217
Floor cloth..... Sq. Yds.	16,292,200	17,192,100	779,538	818,617
Painters' materials..... Value £	..	..	1,418,189	1,559,186
Paper..... Cwt.	1,075,919	988,765	1,837,326	1,763,527
Rags..... Tons	58,330	58,827	460,319	473,287
Soap..... Cwt.	499,808	492,593	482,399	502,850
Total value .....	..	..	30,989,012	33,073,187

## EXPORTS OF SPANISH HEMATITE IRON ORE.

The *Bilbao Maritimo y Comercial* shows that the exports of Spanish hematite iron ore from that port alone last year were nearly up to the heaviest on record. The only year in which they were exceeded was 1887, at which

period the price of the mineral ruled very much lower than in 1889:—

	Tonn.
1878 .....	1,221,730
1879 .....	1,117,836
1880 .....	2,315,598
1881 .....	2,500,532
1882 .....	3,692,512
1883 .....	3,378,234
1884 .....	3,155,432
1885 .....	3,295,382
1886 .....	3,160,917
1887 .....	4,179,122
1888 .....	3,589,425
1889 .....	3,851,635

—Ironmonger.

### OUR FOREIGN TRADE IN 1889.

Sir Rawson W. Rawson, chairman of the Commercial Committee of the Imperial Federation League, has compiled the following particulars concerning our foreign trade in 1889:—

#### YEAR 1889 COMPARED WITH THE YEAR 1888.

##### A.—Imports.

1. Tonnage of vessels entering United Kingdom has increased from—  
27,977,000 to 28,518,000 = 5.0 per cent.
2. Value of imports has increased from—  
386,582,000*l.* to 427,211,000*l.* = 10.5 per cent.
3. Average price per ton has increased from—  
14*l.* 6*s.* to 14*l.* 18*s.* = 4.2 per cent.

##### B.—Exports.

1. Tonnage of vessels clearing from United Kingdom has increased from—  
31,664,000 to 33,049,000 = 4.3 per cent.
2. Value of exports has increased from—  
297,885,000*l.* to 313,031,000*l.* = 5.1 per cent.
3. Average price per ton has remained stationary at 9*l.* 8*s.*

#### READINGS FOR THE YEAR 1889 COMPARED WITH 1888.

1. The character of the year 1889 is one of uniformly moderate increase with the exception of—  
(a.) Price of coal, which has advanced 21.3 per cent.  
(b.) Price of gross exports, which has remained stationary.  
(c.) Tonnage of exports, exclusive of coal, which is almost the same.

2. Imports have increased in volume 5.0 per cent.

“ “ “ value 10.5 “  
“ “ “ price 4.2 “

3. Total exports have increased in volume 4.3 per cent.

“ “ “ value 5.1 “  
“ “ “ remained in price stationary.

4. Tonnage employed in the export of coal formed 58.4 per cent. of the total tonnage cleared; in 1888 it formed 56.8 per cent. The quantity of coal exported showed an increase of 7.4 per cent. compared with an increase of 10.2 per cent. in 1888. The increase in the first five months was sixfold that in the last seven. There has thus been a check to the previous rapid increase of this exportation.

5. The quantity of bunker coal showed an increase of 8.6 per cent. This affords some (a minimum) measure of the increase in the tonnage of steam—compared with sailing—vessels which cleared in 1889, as the increase of total tonnage clearing was only one-half—viz., 4.3 per cent.

6. The increased price of coal advanced 21.3 per cent., but this affected the total value of exports by less than 1 per cent.

7. The tonnage clearing with exports, exclusive of coal, increased only 0.3 per cent., but the value of exports increased 4.1 per cent., which was caused by an advance in price, which figures as 3.9 per cent.

8. The value of United Kingdom produce exported increased 6.5 per cent.; that of foreign and colonial merchandise, 1.4 per cent.

#### AS REGARDS MONTHLY FLUCTUATIONS.

The first four months and the last three have been the periods of greatest activity in the export trade both of British and foreign and colonial merchandise.

#### EXPORTS IN 1889 COMPARED WITH 1888.

	British.	Foreign and Colonial.
	Per Cent.	Per Cent.
January to April .....	Increase, 8.4	Increase, 16.8
May to September .....	“ 2.0	Decrease, 16.9
October to December ...	“ 11.1	Increase, 11.6

#### AS REGARDS DESCRIPTION OF MERCHANDISE.

1. Of the ten groups of imports, as classified by the Board of Trade, all have increased except metals. This is shown in the following table, in which the groups are arranged according to the proportionate importance of each:—

##### Imports in 1889.

	Proportion of Total Value.	Increase in Value of each Group.
	Per Cent.	Per Cent.
1. Articles of food and drink .....	37.7	7.9
2. Raw materials for textiles .....	21.4	13.4
3. Manufactured articles .....	15.3	11.2
4. Raw materials, other than above .....	10.2	19.0
5. Metals .....	5.0	Decrease, 5.0
6. Miscellaneous articles .....	3.4	Increase, 4.8
7. Animals, living, for food .....	2.4	31.0
8. Chemicals .....	2.0	6.4
9. Oils .....	1.7	10.7
10. Tobacco .....	0.9	40.8
Total .....	100.0	10.5

2. Of the ten groups of exports all have increased, as shown in a similar manner in the following table:—

##### Exports in 1889.

	Proportion of Total Value.	Increase in Value of each Group.
	Per Cent.	Per Cent.
Produce of United Kingdom:—		
1. Yarns and textile fabrics .....	35.2	1.2
2. Metals and manufactures thereof, except machinery, &c. ....	13.1	10.2
3. Miscellaneous manufactures .....	10.5	6.6
4. Raw materials (chiefly coal) .....	5.5	24.1
5. Machinery and millwork .....	5.0	17.9
6. Apparel, &c. ....	3.7	2.0
7. Articles of food and drink .....	3.4	4.7
8. Chemicals .....	2.5	6.7
9. Animals, living .....	0.4	12.6
10. Foreign and Colonial .....		
Total .....	100.0	5.1

## METALS IN 1889.

(From W. T. Sargant and Son's Annual Circular.)

## LEAD.

—	1889.	1888.	1887.	1886.	1885.	1884.
	Tons.	Tons.	Tons.	Tons.	Tons.	Tons.
Production of British Mines .....	45,000*	45,000*	44,000	39,599	37,687	39,000
Importation of Foreign into United Kingdom.....	145,451	132,911	114,382	107,878	108,063	109,914
Exports from United Kingdom .....	52,153	48,653	44,367	42,388	38,578	35,539
Highest price of soft pig.....	£ s. d. 14 15 0	£ s. d. 15 15 0	£ s. d. 15 15 0	£ s. d. 13 12 6	£ s. d. 12 10 0	£ s. d. 11 10 0
Lowest price of soft pig .....	12 5 0	12 5 0	11 17 6	12 0 0	10 5 0	10 2 6

## SHEET ZINC.

—	1889.	1888.	1887.	1886.	1885.	1884.
	Tons.	Tons.	Tons.	Tons.	Tons.	Tons.
Importations of foreign into United Kingdom.....	19,261	18,262	19,555	18,322	19,663	20,138
Highest price.....	£ s. d. 25 2 6	£ s. d. 22 10 0	£ s. d. 19 5 0	£ s. d. 17 15 0	£ s. d. 17 0 0	£ s. d. 18 5 0
Lowest price.....	18 15 0	16 17 6	16 0 0	16 0 0	16 0 0	16 15 0

## SPELTER.

—	1889.	1888.	1887.	1886.	1885.	1884.
	Tons.	Tons.	Tons.	Tons.	Tons.	Tons.
Importations into United Kingdom.....	56,842	61,045	56,187	51,508	60,229	47,647
Highest price for Silesian .....	£ s. d. 24 5 0	£ s. d. 22 0 0	£ s. d. 21 10 0	£ s. d. 15 5 0	£ s. d. 15 10 0	£ s. d. 15 5 0
Lowest price for Silesian.....	16 10 0	14 10 0	14 0 0	13 10 0	14 0 0	14 0 0

## QUICKSILVER.

—	1889.	1888.	1887.	1886.	1885.	1884.
	Bottles.	Bottles.	Bottles.	Bottles.	Bottles.	Bottles.
Production of Spanish† .....	49,778	53,243	51,011	48,537	45,813	48,998
Italian† .....	10,498	10,460	7,400	8,000	7,888	8,040
Austrian.....	14,000	14,000	14,000	14,900	14,370	14,680
Californian .....	28,000	33,250	33,760	29,981	32,073	31,913
Borneo‡ .....	1,559	1,559	1,559	1,559	1,559	1,559
Total.....	103,835	112,512	107,720	102,167	101,703	104,290
Exports from United Kingdom†.....	55,366	45,806	61,049	66,974	50,942	50,634
Highest price of Spanish .....	£ s. d. 9 15 0	£ s. d. 10 10 0	£ s. d. 11 5 0	£ s. d. 7 10 0	£ s. d. 6 17 6	£ s. d. 6 15 0
Lowest price of Spanish.....	7 7 6	6 15 0	6 10 0	5 13 0	5 10 0	5 1 6

\* Estimated.

† Calculated November to November.

‡ Average production.

**THE SULPHATE OF AMMONIA MARKET.**  
(From Messrs. Bradbury and Hirsch's Annual Circular.)

IMPORTS OF NITRATE OF SODA AND PRODUCTION OF  
SULPHATE OF AMMONIA FOR 20 YEARS.

	Nitrate of Soda.		Sulphate of Ammonia.	
	Imports into Europe.	Average Price per Cwt.	Production U.K.	Average Price per Ton.
	Tons.	s. d.	Tons.	£ s. d.
1870	100,000	15 3	40,000	16 0 0
1871	128,000	15 5	41,000	19 0 0
1872	175,000	15 1½	42,000	21 0 0
1873	225,000	14 3	43,000	18 8 9
1874	230,000	11 11	45,000	17 2 6
1875	280,000	11 9	46,000	18 10 0
1876	300,000	21 8	48,000	18 12 6
1877	208,000	13 10	52,000	19 16 3
1878	250,000	15 0	55,000	20 3 0
1879	205,000	14 9	57,000	18 8 9
1880	140,000	15 8	60,000	19 0 0
1881	230,000	14 8	65,000	20 4 6
1882	835,000	13 1	72,000	20 8 6
1883	440,000	11 2	75,000	16 11 0
1884	505,000	9 9	87,000	14 9 3
1885	380,000	10 2	97,000	11 9 1½
1886	330,000	9 9	106,500	11 3 7½
1887	440,000	9 6	113,700	11 17 8
1888	640,000	9 9	122,800	11 18 0
1889	760,000	9 2	132,000	12 1 4½

**SULPHATE OF AMMONIA.**

*Production, Deliveries, and Exports for 1889 as compared with 1888.*

	1889.	1888.
	Tons.	Tons.
Production:—		
England, Scotland, and Ireland .....	132,000	122,800
Deliveries and exports:—		
Germany, Denmark, Sweden, Russia, &c....	32,000	32,000
France, Spain, and Italy .....	18,000	19,000
Belgium and Holland .....	20,000	18,000
America and colonies .....	17,000	14,000
Home consumption for agricultural and chemical purposes .....	40,000	31,300
Stocks .....	5,000	8,500
	132,000	122,800

**THE SOURCES OF SULPHATE OF AMMONIA.**

	1889.	1888.
	Tons.	Tons.
Gas works .....	100,000	93,000
Iron works .....	6,000	5,300
Shale works .....	23,000	22,000
Coke and carb. works .....	3,000	2,500
	132,000	122,800

**Monthly Patent List.**

\* The dates given are the dates of the Official Journals in which acceptances of the Complete Specifications are advertised. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the said dates.

**1.—GENERAL PLANT, APPARATUS, AND MACHINERY.**

**APPLICATIONS.**

1889.

20,530. R. Tervet and G. Reid. Filter presses. December 21.

20,548. J. R. Watson. Improved retort and filter stand. December 21.

20,569. G. H. Bolton. Improvement in or appertaining to caustic soda drum lids and the like, and to the attachment of such lids, metallic labels, or the like to caustic soda drums, or other sheet metal structures. December 21.

20,739. A. H. Hobson. Dessicating apparatus. December 24.

20,784. R. A. Robertson and W. J. Murreles. Apparatus for evaporating, concentrating, and distilling liquids. December 27.

20,792. A. Lusuardi and A. Faleo. Pressure and vacuum gauges. December 27.

20,925. J. E. Bedford. Apparatus for exposing liquids to the action of gases. December 31.

20,954. J. Wright. Apparatus for distilling, evaporating, or concentrating liquids or solutions and the like, and for condensing exhaust steam. December 31.

1890.

206. J. F. H. Gronwald and E. H. C. Oehlmann. Sterilising apparatus. Complete Specification. January 6.

210. T. Thorp. A frictionless metallic gas-pressure gauge. January 6.

266. A. Brock and H. A. Romero. Improved air pump. Complete Specification. January 7.

341. C. Nield. Improved water filter. January 8.

386. A. Simpson. Improvements in salt pans. January 9.

398. W. Wild. Apparatus for the evaporation of liquids. January 9.

474. C. Blagburn. Apparatus for treating liquids and solids with gases. January 10.

510. T. E. Wilson. The concentration of liquids by evaporation. January 11.

## COMPLETE SPECIFICATIONS ACCEPTED.\*

1889.

499. F. H. Danehell. Means or apparatus for separating solid matters from liquids. December 25.

1078. H. Green and A. Green. Purifying water and removing crustation from steam boilers. January 8.

1345. B. Nicholson and T. Palmer. Rotating vessels for use in tanning, washing, emulsifying, churning, &c.; applicable for treating materials in vacuo or under fluid pressure. December 31.

1424. C. F. H. Hayes. Apparatus for compressing air, gases, or other elastic fluids. January 8.

2603. W. Beaumont. Centrifugal pumps. January 8.

2806. A. Waldbauer. Centrifugal apparatus for continuously washing, dyeing, impregnating, &c. various materials and fabrics. December 31.

2837. Clement, Baron of Bechtolsheim. Centrifugal separators. December 25.

3016. Sir B. Samuelson, Bart.—From G. Maffei. Separating liquids of two different densities. December 31.

3736. M. J. E. Vasscl. Apparatus for filtering liquids, or for preparing extracts from substances. January 15.

4340. H. Halpin and P. W. Willans. Means and apparatus for condensing steam. January 15.

5197. J. C. Mewburn.—From La Société des Generateurs à Vaporisation Instantanée (Système Serpollet). Apparatus for generating steam. December 31.

12,973. The Water Softening Company, Limited, E. Easton and H. Carrod. Apparatus for softening and filtering water or other liquids under pressure. January 15.

13,761. J. Wach. Improvements in thermostats and apparatus operated by differences in the thermal expansion of metals. December 25.

16,763. J. Popper. Condensing vapours and cooling gases or liquids. December 31.

## II.—FUEL, GAS, AND LIGHT.

## APPLICATIONS.

1889.

20,174. J. J. Robins and W. Shakespeare. An improved self sealing door for gas retorts and other purposes. December 16.

20,243. D. McDonald. Improved coal bricks. December 17.

20,255. T. S. Brown and A. W. Brown. Increasing the illuminating power of gas, called the "Aeme gas-saver." December 17.

20,368. W. L. Wise.—From L. Semet, Belgium. Improvements in coke ovens. December 18.

20,370. J. Morley. Improvements in preparing material for and in compounding firelighters, and in means therefor. December 18.

20,432. T. H. Gray and S. S. Bromhead. Improvements in the preparation of coal gas for combustion, and in apparatus for the same. December 19.

20,551. J. West. Improvements in machinery for charging gas retorts. December 21.

20,679. A. Myall.—From J. H. Laneaster, United States. Improvements in smokeless and gasless fuel. December 23.

20,980. H. E. Newton.—From the Fuel, Gas, and Light Improvement Co. Incorp., United States. Improvements in the process of and apparatus for manufacturing gas. December 31.

20,981. H. E. Newton.—From the Fuel, Gas, and Light Improvement Co. Incorp. Improvements in apparatus for the manufacture of gas. December 31.

1890.

27. A. M. Clark.—From D. Dull, United States. Improvements in distilling heavy crude petroleum oils. January 1.

44. S. J. Bull. Improvements in scoops for charging gas retorts. January 1.

67. T. Parker. Improvements in the distillation of coal, shale and the like, the invention comprising the production of a special description of coke. January 2.

260. J. Stears. Improvement in preventing deposit of carbonaceous matter in ascension pipes used in gas-making. January 7.

343. W. T. Cotton and E. F. B. Crowther. Improvements in making gas from coal by vertical fireclay or metal retorts, which are tapered internally, and fitted with slide valves which are encased in iron or steel boxes underneath the retorts to empty them when required, and circular pans at the outlet containing water, to make the valve-boxes and retorts air-tight. January 8.

344. G. Craig. Improvements in the coking of coal and in the ovens employed therefor. January 8.

373. G. H. Hunt. An improved compound of materials for economising smoke in furnaces and other fireplaces. January 9.

502. J. K. Thompson. Improvements in blocks of briquettes made from coal or other material. January 11.

552. W. C. Sellar. Improvements in gas producers. January 11.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

50. C. F. Claus. Production of mixtures of hydrogen and carbonic oxide; and obtaining hydrogen, carbonic oxide, or carbonic acid severally, and certain by-products. December 25.

1806. C. H. Mowll. Improved composition of artificial or block fuel. December 25.

1955. W. Wells. Burner for converting oils or spirits into vapour or gas to obtain light or heat. December 25.

2796. H. Darby. Apparatus for generating and burning gases from hydrocarbon oil and water. December 25.

3394. G. R. Hislop. Apparatus for charging gas producers and gas or shale oil retort furnaces with hot residual coke. December 31.

3936. J. Channon. Firelighters. January 15.

4168. W. Pariby. Instrument for clearing gas-retort ascension pipes. January 15.

6090. F. H. Briggs. Apparatus for the manufacture of water-gas. December 25.

7539. J. T. Ellis. Improvements in apparatus for condensing smoke in the furnaces of steam and other boilers. December 25.

12,460. S. Elliott. Improved method of and apparatus for the treatment of smoke. January 15.

14,287. M. A. Morse. Improvements relating to the manufacture of gas, and to apparatus therefor. December 11.

16,486. A. Coze. Improvements in gas retorts and in apparatus connected therewith. December 25.

18,502. E. Fales. Improvements in and relating to the method of and apparatus for burning coal and other fuel in furnaces. December 25.

### III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

#### APPLICATIONS.

1890.

67. T. Parker. *See* Class II.

178. P. Richin. Process for separating phenol and the cresols from cresote, and for obtaining the said substances in a pure condition. January 4.

### IV.—COLOURING MATTERS AND DYES.

#### APPLICATIONS.

1889.

20,219. J. Imray.—From La Société Anonyme des Matières Colorantes et Produits Chimiques de St. Denis, France. Improved manufacture of azoxyaniline and of colouring matters derived therefrom. December 16.

20,292. J. Imray.—From La Société Anonyme des Matières Colorantes et Produits Chimiques de St. Denis. The production of a substituted meta-diamine and of colouring matter derived therefrom. December 17.

20,497. O. Imray.—From The Farbwerke vorm. Meister, Lucius, und Brüning, Germany. Manufacture of non-plumbiferous lakes. December 20.

20,668. J. Y. Johnson.—From The Badische Aniline und Soda Fabrik, Germany. Improvements in and relating to the manufacture of colouring matters suitable for dyeing and printing. December 23.

20,678. O. Imray.—From The Farbwerke vorm. Meister, Lucius, und Brüning. Production of compounds of the diphenylmethane group and the rosaniline series. December 23.

20,924. J. E. Bedford. Improvements in the treatment of logwood and logwood extract for dyeing and printing. December 31.

1890.

39. R. J. Friswell. Improvements in the manufacture of raw materials for dyestuffs. January 1.

134. R. J. Friswell and A. G. Green. Improvements in the production of azo-colouring matters. January 3.

569. Kern and Sandoz. Improvements in the manufacture of blue colouring matter. January 11.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1888.

18,709. E. Edwards.—From Standaert Brothers. A new or improved blue dye. December 25.

1889.

1771. B. Willeox.—From The Farbenfabriken vorm. F. Bayer and Co.—Manufacture of para-amido-phenol ethers. January 8.

3333. T. R. Shillito.—From J. R. Geigy and Co. Production of triphenylmethane colours dyeing with the aid of mordants. December 31.

3529. B. Willeox.—From The Farbenfabriken vorm. F. Bayer and Co. Production of indulines or induline-like dyestuffs. January 8.

4825. J. Imray.—From La Société Anonyme des Matières Colorantes et Produits Chimiques de St. Denis, A.F. Poirrier and D. A. Rosenstiehl. Manufacture of azo-colouring matters. December 25.

5032. J. Imray.—From La Société Anonyme des Matières Colorantes et Produits Chimiques de St. Denis. Manufacture of colouring matters giving grey, brown, and analogous tints. December 25.

11,795. H. H. Leigh.—From R. G. Williams, United States. Colouring matters. December 25.

14,294. H. H. Leigh.—From R. G. Williams, United States. Improvements in dye colours. December 25.

18,901. C. Dreyfus. Manufacture and production of colouring matters for dyeing and printing. January 8.

### V.—TEXTILES, COTTON, WOOL, SILK, Etc.

#### APPLICATIONS.

1889.

20,314. H. A. Lowe. Improvements in the treatment of or finishing of manufactured or partly manufactured materials from cotton or similar fibres or other substance containing cellulose. December 17.

20,665. C. Baswitz. Improvements in the treatment of textiles and similar fabrics by means of ammoniacal oxide of copper, to increase the waterproof and non-combustible character of the fabrics. Complete Specification. December 23.

20,771. R. J. Maclean. An improved method or process of producing mixed coloured silk for obtaining mixed coloured silk fibre or mixed coloured yarn. December 27.

20,936. L. Pinagel. A process and apparatus for freeing wool from grease. Complete Specification. December 31.

1890.

200. J. Walker and W. Brown. Improvements in apparatus for drying or carbonising fabrics composed of animal and vegetable fibres, wool, silk, cotton, flax, rags, or other like fibrous substances. January 6.

#### COMPLETE SPECIFICATION ACCEPTED.

1889.

7715. C. H. Hine and J. H. Noad. Process for waterproofing and preserving textures and other materials. December 31.

### VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

#### APPLICATION.

1890.

142. Improvements relating to the dyeing and waterproofing of textile fabrics. January 3.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

415. A. G. Salamon. Bleaching fibre. January 8.  
 2668. J. Walker and H. Carver. Printing "sanitary" or washable paper-hangings. December 25.  
 2806. A. Waldbauer. See Class I.  
 2984. T. Ingham. Dyeing mixed woven fabrics. December 25.  
 3934. B. Willcox.—From The Farbenfabriken vorm. F. Bayer and Co. Dyeing and printing animal and vegetable fibres. January 15.  
 18,973. L. Weldon. Improvements in machines for dyeing cotton and analogous materials. December 31.

## VII.—ACIDS, ALKALIS, AND SALTS.

## APPLICATIONS.

1889.

20,722. J. C. Fell.—From the American Ammonia Manufacturing Co., United States. A method of and apparatus for obtaining ammonia from atmospheric nitrogen and the hydrogen of decomposed steam. December 24.

20,787. J. Longshaw. The treatment of spent or partially spent acid. December 27.

20,862. W. Smith and C. G. Cresswell. Improvements in the manufacture of nitrate of ammonia and bicarbonate of soda in conjunction therewith. December 28.

20,908. M. B. Mason, S. D. Warren, and F. Warren. Improved method of and apparatus for recovering chemicals from solutions, or when mixed with substances which are volatilisable or destructible by heat, especially adapted for recovering soda from waste liquors of pulp digesters. Complete Specification. December 31.

1890.

283. A. T. Smith, jun. Improvements in the treatment of ammonium chloride, ammonium sulphate, calcium chloride, or calcium sulphate for the obtaining of hydrochloric acid and other valuable products therefrom or thereby. January 7.

381. O. C. Townsend. Improvements in processes and apparatus for the manufacture or preparation of nitrate of potash and other salts. January 9.

393. R. C. Wilson. Improvements in and connected with the evaporation of brine and the manufacture of salt therefrom. January 9.

411. H. J. E. Hemebutte. Improvements in the manufacture of sulphate of copper, and apparatus therefor. January 9.

419. P. R. de Lambilly and E. L. Chabrier. A new or improved process for fixing atmospheric nitrogen, and obtaining ammonia and cyanogen compounds. January 9.

448. F. M. Spence, D. D. Spence, and A. Esilman. Improvements in the manufacture of alum. January 10.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

94. J. Hargreaves, T. Robinson, and J. Hargreaves. See Class IX.

2160. L. Mond. Apparatus for volatilising ammonium chloride. December 25.

2575. L. Mond. Volatilising ammonium chloride, and obtaining ammonia, chlorine, and hydrochloric acid therefrom; also obtaining chlorine from hydrochloric acid. December 25.

2786. E. K. Muspratt and G. Eschellman. Manufacture of chlorates, and of magnesium hydrate for use therein. December 25.

3071. J. S. MacArthur. Obtaining cyanides from residuary liquors of chemical processes. December 25.

3072. J. S. MacArthur. Obtaining cyanides from residuary liquors. December 25.

3407. A. Campbell and W. Boyd. Obtainment of chlorine and sulphate of lime. January 8.

3757. T. B. Saunders. Treating or utilising the spent or residual liquor formed in the process of cleaning iron or steel with hydrochloric acid. January 8.

4037. J. B. Hannay. Process and apparatus for manufacture and transport of liquid chlorine. January 15.

4316. E. Carez. Manufacture of ammonium nitrate. January 8.

## VIII.—GLASS, POTTERY, AND EARTHENWARE.

## APPLICATIONS.

1889.

20,176. W. H. Hollinshead and H. Griffiths. Improvements in appliances to be used for printing on, or ornamenting ceramic ware. December 16.

20,233. J. J. Candlish and W. Hyslop. Improvements in the manufacture of glass bottles and other hollow glassware, and in apparatus relating thereto. December 16.

20,234. J. J. Candlish and W. Hall. Improvements in the manufacture of glass bottles and other hollow glassware, and in apparatus relating thereto. December 16.

20,328. D. Webb. Improvements in the "posts" or "pontils" used in the manufacture of articles of "pressed glass." December 18.

20,470. J. Campbell. Improvements in drying kilns. December 20.

20,572. A. Griffiths. Improvements in the manufacture of fire-clay pipes, called "swan-necks." December 21.

20,599. G. Waller. An improved method of producing or making pictorial or decorative earthenware tiles by photographic means. December 23.

20,619. J. G. Sowerby. An improved method of cutting or dividing glass cylinders and other forms or articles of glass ware. December 23.

20,687. S. Washington. Improvements in tanks or furnaces used for melting or founding glass. December 24.

1890.

211. T. T. Liddle. Improvements in or relating to the manufacture of glass bottles. January 6.

342. J. Gimson and Challinor. An improved appliance for supporting china and earthenware cups whilst being fired. January 8.

352. E. Robson. An improved production of porcelain tablets or letters. January 8.



## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

153. J. B. Adams. Method and apparatus for making solid or hollow articles from molten glass or other plastic material. January 8.

161. T. Willett. Process and apparatus for making plates, &c. in earthenware, china, &c. December 31.

2776. J. Thorne. Manufacture of coloured lenses for signal lamps. December 25.

16,264. W. West. Improvements in the mode and means employed for the manufacture of flower or plant pots or other articles from clay or other semi-plastic material. January 15.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

## APPLICATIONS.

1889.

20,303. C. Smith.—From G. P. V. Neilsen, Denmark. A new or improved artificial asphalt. December 17.

20,848. A. Smith. A manufacture of artificial stone. December 28.

20,914. A. Klapperstuck and A. H. Meyer. Improvements in fireproof ceilings and walls. December 31.

1890.

428. A. Wright. An improved weathered facing brick for building purposes. January 9.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

91. J. Hargreaves, T. Robinson, and J. Hargreaves. Treating lime refuse from alkali works to render it suitable for manufacture of cement, and apparatus therefor. January 8.

116. R. Stone. Manufacture and utilisation of cement and plaster, and appliances therefor. January 15.

1262. A. McLean. Method and means for manufacturing blocks of artificial stone. December 31.

1952. J. C. Merryweather. Improvements in paving. January 15.

2718. A. C. Ponton, B. L. Mosely, and C. Chambers. Preparation of siliceous materials used in the manufacture of artificial stone. December 25.

2780. A. C. Ponton, B. L. Mosely, and C. Chambers. Artificial stone. January 15.

2781. A. C. Ponton, B. L. Mosely, and C. Chambers. Manufacture of artificial stone. January 15.

3354. A. C. Ponton, B. L. Mosely, and C. Chambers. Blocks or bricks of siliceous materials. December 31.

3355. W. Hubbard, B. L. Mosely, and J. S. Fairfax. Wall tiles or linings. December 31.

3357. H. H. Leigh.—From L. H. Clansen. Manufacturing artificial stone. December 31.

3378. C. Güttler. Method and apparatus for forming disintegrated or loose material, such as sugar, clay, cement, &c. into compact bodies. December 31.

4333. G. Batebelor. Drying slurry by waste heat from cement kilns. January 8.

4347. J. Homan. Fireproof structures and bricks for same. January 8.

10,997. J. B. King. Composition or plaster for the treatment of walls and the like. January 8.

17,640. W. P. Thompson.—From C. Kohn, Austria. Manufacture of artificial stone or of articles formed of the same. January 8.

## X.—METALLURGY, MINING, Etc.

## APPLICATIONS.

1889.

16,776A. J. B. Alzugaray. A new or improved universal furnace for the fusion and refining of metals, alloys, dross, &c. by the dry process or otherwise, called "the Baxeres universal furnace." Originally included in No. 16,776, dated October 24, 1889.

20,180. R. F. Neuninger. A new combined smelting and reverberatory furnace and apparatus for catching the dust and condensing the fumes from the furnace. Complete Specification. December 16.

20,181. T. Young. Improvements in treating ores to obtain precious metals, and in apparatus therefor. December 16.

20,210. J. Hobson. Improved means of separating particles of iron or of magnetic oxide of iron from admixture with other substances. December 16.

20,214. S. Pearson and J. H. Pratt. Improvements in the manufacture of alloys of aluminium. December 16.

20,223. E. Taussig. Improvements in and relating to casting. December 16.

20,264. A. Gutensohn. An improved method of and process for annealing metal plates and sheets. December 17.

20,313. W. P. Thompson.—From J. Brown, New Zealand. Improvements in washing out or separating gold dust and other heavy metallic particles from impurities, and in apparatus therefor. December 17.

20,380. J. E. Bott. Improvements in the manufacture of compound armour plates and similar articles. December 18.

20,395. T. Brady, T. C. Powers, M. J. Quinn, and A. J. Maher. Improvements in apparatus for casting ingots. December 18.

20,586. J. H. Darby. Improvements in the manufacture of steel or ingot iron. December 21.

20,587. J. Lysaght. Improvements in galvanising apparatus. December 21.

20,677. W. L. Wise.—From J. B. Walker, United States. Improvements in apparatus for treating finely-divided metalliferous material for the purposes of separation and amalgamation. December 23.

20,860. B. H. Thwaite. Improved open-hearth steel and high-temperature furnace. December 30.

1890.

70. I. Bang and A. Ruffin. Improved process for separating tin from other metals, more particularly applicable for the treatment of tin plate scrap and old tinned cases. January 2.

128. I. Bang and A. Ruffin. Improvements in the production of tin plates, and in apparatus for that purpose. January 3.

190. B. Talbot, jun., and J. Hardecastle. Improvements in the production of ingots or masses of steel and alloys thereof. January 4.

191. B. Talbot, jun., and J. Hardecastle. Improvements in or applicable to the manufacture of tubular, hollow, or dished ingots, and other articles of steel and alloys thereof. January 4.

213. J. Mackintire. Improvements in the manufacture of steel and iron. January 6.

237. R. Brown and G. H. Irvine. Improvements in the wet method of extracting gold from finely-divided auriferous quartz ores or other auriferous material, and in apparatus therefor. Complete Specification. January 6.

316. J. E. Chaster. Improvements in the extraction of metals from ore. January 8.

537. W. P. Thompson.—From H. Kromberg, Prussia. Improvements in tuyeres for cupola, &c. and other blast furnaces (hoehofenform). January 11.

560. T. Twynam. Improvements in the manufacture of copper. January 11.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1888.

14,400. H. Hutchinson. Treatment of complex gold and silver-bearing ores, &c., and recovery of metals therefrom. January 15.

14,445. P. Marino. See Class XI.

1889.

15,066. R. Hannan.—From J. J. C. Smith (New Jersey). A process for casting in aluminium bronze dies, to be used for the purpose of stamping metals or other substances for which dies are used. January 8.

19,145. F. A. Herbertz. Smelting furnaces. January 8.

19,621. D. Dennes and T. K. Rose. Improved process or means for extracting precious metals from their ores. January 15.

### XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

#### APPLICATIONS.

1889.

19,861A. C. H. Logan. Improvements in plates for secondary voltaic batteries. Complete Specification. December 10. Originally included in No. 19,861 of that date.

20,217. F. Marx. Process of manufacturing metallic electrodes. Complete Specification. December 16.

20,516. G. Philippart. Improvements in the construction of secondary batteries. December 20.

20,752. F. Marx. Improvements in the manufacture of electrodes for electric batteries. December 24.

20,856. D. G. Fitzgerald and A. H. Hough. Improvements in elements for voltaic batteries. December 28.

20,911. A. E. Woolf. Improved battery plates for storage batteries. Complete Specification. December 31.

1890.

29. S. U. Dardier. Improvements in the electrical coating of metals on non-metallic substances. January 1.

232. J. Hopkinson, E. Hopkinson, and G. A. Grindle. Improvements in electric motors and dynamo-electric machines. January 6.

249. W. F. King. Improvements in dynamo-electric machines. January 7.

302. H. H. Lake.—From P. H. Alexander, United States. Improvements in secondary or storage batteries, and in the manufacture of plates therefor. Complete Specification. January 7.

303. H. H. Lake.—From P. H. Alexander. Improvements relating to secondary batteries. January 7.

350. L. Epstein. Improvements in the formation of electrodes for use in primary and secondary electric batteries. January 8.

488. M. Sappey. Improvements in and connected with primary batteries. January 10.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1888.

14,445. P. Marino. The extraction and electro-deposition of aluminium and aluminium alloys. December 18.

1889.

1420. F. King. Secondary batteries. December 25.

2331. W. H. Dowland and W. Mills. Apparatus for economically effecting the double decomposition of certain substances by the aid of electricity. January 15.

3502. A. G. Greenway, B. F. Babcock, and B. Radcliffe. Improvements in processes for manufacturing iron, wherein the iron is subjected to an electric treatment. January 15.

18,365. J. B. Entz and W. A. Phillips. Plates for primary and secondary batteries. December 25.

18,953. Macanlay-Cruikshank.—From Johnson and Smith. An improved electrode for secondary batteries or accumulators. December 31.

18,966. O. A. Enholm. Primary or galvanic batteries. December 31.

18,989. W. F. Smith. Secondary or storage batteries. December 31.

19,861. C. H. Logan. Secondary batteries. January 15.

19,861A. C. H. Logan. Plates for secondary voltaic batteries. January 15.

19,883. J. K. Pumpelly and F. D. Thomason. Electrical secondary batteries. January 15.

19,899. W. P. Thompson.—From M. W. Parish, United States. Improvements in or relating to electric batteries. January 15.

### XII.—FATS, OILS, AND SOAP MANUFACTURE.

#### APPLICATIONS.

1889.

20,237. W. Rödiger. Improvements in and relating to the manufacture of soap. Complete Specification. December 16.

1890.

540. J. L. Wade. An improved non-freezing and lubricating oil. January 11.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1888.

18,857. H. H. Lake.—From La Société Anonyme des Parfums Naturels de Cannes. Purification of fatty substances and apparatus therefor. December 31. (Advertised in official Journal by error as accepted December 4, 1889.)

1889.

5353. W. G. Smith. Manufacture of soap. December 31.

9410. A. H. Parker. Improved cleansing, scouring, and bleaching compound for wool and other fabrics, also applicable to cleansing purposes generally. December 31.

18,592. J. Y. Johnson.—From L. Genevois, Italy. Improvements in the treatment or preparation for consumption of soft soaps and other like materials. December 25.

19,158. P. Heinz. A new or improved method for sweetening margarine butter and other oils and fats. December 31.

19,948. W. Grills and M. Schroeder. Improved process for the extraction of fats and oils from substances containing the same by means of sulphurous acid. January 15.

### XIII.—PAINTS, PIGMENTS, VARNISHES, AND RESINS.

#### APPLICATIONS.

1889.

20,289. J. Y. Johnson.—From N. C. Mitchell, United States. Improvements in and apparatus for the treatment of waste rubber goods to recover the rubber therefrom, and the production of a new product by such treatment. December 17.

20,707. R. Mullard. An improved method of making paints and varnishes, and of the treatment of gums and resins for the same, for preservative, decorative, and artistic purposes; for preserving the plates of iron and steel ships from corrosion, and the bottoms of ships from fouling. December 24.

20,830. C. Huelser.—From G. Calmann, Germany. An improved process and composition for producing printing ink. December 28.

1890.

71. G. Bischof. Improvements in the manufacture of white lead and apparatus for that purpose. January 2.

280. J. Robson. Improvements in the method of protecting iron and steel surfaces from corrosion, and in coatings for the same. January 7.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

13,792. J. J. Carr. Material for making whitewash, colour-wash, and water-colour paints. January 8.

15,826. A. J. Boulton.—From W. Keil, United States. Manufacture of vulcanised plastic compounds. January 15.

18,076. H. H. Lake. From J. B. Tibbits, United States. Manufacture of white lead. January 15.

### XIV.—TANNING, LEATHER, GLUE, AND SIZE.

#### APPLICATIONS.

1889.

20,403. J. Hutchings and W. N. Hutchings. Improvements in the method of and apparatus for extracting the tannin from bark or other materials used for tanning purposes. December 18.

1890.

556. G. Rothel. An improved glue or cement. January 11.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

1345. B. Nicholson and T. Palmer. *See Class I.*

18,503. R. W. Johnson. Improvements in and relating to plastics, more especially designed for imparting to glue a permanent elasticity and adapted to be used for several purposes. December 25.

### XV.—AGRICULTURE AND MANURES.

#### COMPLETE SPECIFICATION ACCEPTED.

1889.

18,918. E. Saxl and H. Saxl. Fertilisers. December 31.

### XVI.—SUGARS, STARCHES, GUMS, Etc.

#### APPLICATIONS.

1889.

20,515. C. Steffen. An improved process for obtaining the entire quantity of crystallisable sugar contained in sugar mass. Complete Specification. December 20.

1890.

357. S. Wöhle, A. C. Irwin, and G. O. Jacob. Improvements in the preparation of dextrine. January 8.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

3378. C. Güttler. *See Class IX.*

4121. F. Demmin. Cleansing or whitening sugar in the centrifugal machine, and apparatus therefor. January 15.

19,728. W. Thompson, J. Myle, and J. B. Alliot. Apparatus for the expression of juice from sugar cane. January 15.

### XVII.—BREWING, WINES, SPIRITS, Etc.

#### APPLICATIONS.

1889.

19,796A. J. Fisher. Improvements in making bread and fermenting liquids. Previously included in No. 19,796, filed December 4, 1889.

20,298. E. Larsen. Utilisation of brewers' or distillers' yeast. December 17.

20,409. C. Gillett and H. Young. The utilisation of brewers' and distillers' grains in the manufacture of a cake or meal as a food for horses, cattle, pigs, and other animals. December 19.

20,434. B. Hoff and J. Frommel. Apparatus for use in rectifying spirits and other matters. December 19.

20,630. L. Haas. Apparatus for collecting for further utilisation the excess of carbonic acid generated during the manufacture of beer. (Date applied for June 27, 1889, being date of application in Switzerland.)

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

3468. B. Hoff. Improvements relating to distillation, and apparatus therefor. December 31.

5564. W. P. Thompson.—From La Société Generale de Maltose. Manufacture of maltose and fermentation of amylaceous matter. December 25.

15,401. A. J. Boulton.—From Otto and Schwerdtfeger, Saxony. Preservation of beer and the like. December 31.

17,523. H. H. Lake.—From J. Barnes, United States. Yeast cakes. January 15.

19,376. J. F. H. Gronwald and E. H. C. Oehlmann. Process for treating beer and making it keep permanently in good condition. January 15.

## XVIII.—CHEMISTRY OF FOODS, SANITARY CHEMISTRY, AND DISINFECTANTS.

## APPLICATIONS.

*A.—Chemistry of Foods.*

1889.

20,182. E. Sonstadt. Improvements in processes and apparatus for the preparation of an extract of tea, and in the utilisation of the by-products. December 16.

20,336. E. Sonstadt. Improvements in processes and apparatus for the preparation of an extract of coffee, and in the utilisation of the by-products. December 18.

20,614. W. Douglas and J. Donald. Improvements in preserving fish, birds, poultry, or other analogous articles of food. Complete Specification. December 23.

1890.

192. J. H. Hooker. A new or improved alimentary liquid. January 4.

*B.—Sanitary Chemistry.*

1890.

16. J. Smillie. Treating water for preventing action on lead. January 1.

149. H. Guthrie. Improvements in treating sewage, and in the construction and arrangement of means therefor. January 4.

206. J. F. H. Gronwald and E. H. C. Oehlmann. See Class I.

309. T. Kay. Improved economical processes in the treatment of sewage. January 7.

472. C. Jones. Improvements in or connected with destructors and other furnaces for treating refuse. January 10.

480. E. Devonshire. Improvements in purifying water. January 10.

*C.—Disinfectants.*

1889.

20,231. A. Artmann and H. W. Kufeke. Improvements in the process of producing disinfectants. December 16.

20,426. A. Muirhead. Improvements in apparatus for the production of ozone. December 19.

## COMPLETE SPECIFICATIONS ACCEPTED.

*A.—Chemistry of Foods.*

1889.

5052. F. McIntyre. Improvements in the making of butter for the purpose of preserving the same. January 15.

14,670. A. A. Freeman. Preservation of shell fish. January 15.

*B.—Sanitary Chemistry.*

1889.

15,190. H. Rohrbeck. Method and apparatus for disinfecting by means of saturated steam of a high temperature. December 25.

## XIX.—PAPER, PASTEBOARD, ETC.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

16,342. D. C. Simpson. A new or improved composition for treating paper and similar material to render writings or other markings indelible thereon. December 25.

18,090. I. Appletree. A new composition to be used as a substitute for ivory and for other purposes. December 25.

## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES, AND EXTRACTS.

## APPLICATION.

1889.

20,392. J. A. Kendall. Improvements in the manufacture of hypophosphites. December 18.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

3745. A. Domeier. Fixing perfumes. January 15.

10,280. H. H. Lake.—From la Société Anonyme des Parfums Naturels de Cannes. Process of recovering volatile solvents retained in substances treated therewith. January 8.

## XXI.—PHOTOGRAPHIC PROCESSES AND MATERIALS.

## APPLICATIONS.

1889.

20,526. O. Levens. Improvements in the production by photography of lined images in the preparation of surfaces to be etched. December 20.

20,662. W. H. Caldwell. Improvements in sensitising and developing chloride of silver for photographic purposes. December 23.

1890.

401. T. H. Redwood. Improvements in means or apparatus for producing a flash light, more especially applicable to photography. January 9.

547. J. J. E. Mayall. A new or improved manner of colouring photographic impressions with oil colours. January 11.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

2623. J. Leisk. Flash lamp for the rapid combustion of magnesium powder for photographic purposes. January 8.

2723. F. W. Hart. Apparatus for controlling the exposure of sensitive plates when using flash lamps. January 15.

## XXII.—EXPLOSIVES, MATCHES, ETC.

## APPLICATIONS.

1889.

20,286. E. W. Anderson. Apparatus for cutting into lengths and delivering smokeless explosive or other plastic material which has been moulded through a die. December 17.

20,514. E. Davies. Improvements connected with explosive and non-explosive projectiles, and in an explosive compound to be used with the explosive projectiles. December 20.

## COMPLETE SPECIFICATION ACCEPTED.

1889.

1988. A. V. Newton.—From A. Nobel. Preparing explosive compounds for use. January 8.

## XXIII.—ANALYTICAL CHEMISTRY.

## APPLICATIONS.

1889.

20,755. M. Gehre. New or improved process and apparatus for determining the amount of water contained in steam. December 24.

20,859. I. K. Lindström. An improved method of and apparatus for determining the quantity of fat in milk. Complete Specification. December 28.

## Society of Chemical Industry:

A MONTHLY RECORD

FOR ALL INTERESTED IN CHEMICAL MANUFACTURES.

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### SESSION 1889-90.

1890.

March 3rd:—

Dr. H. Schlichter. "Analysis of Textile Fibres and Fabrics."  
Dr. L. T. Thorne. "A Review of the Methods of producing Oxygen commercially."

March 31st (instead of April 7th—Bank Holiday):—

Election of Committee and Officers of Section. Nominations (signed by 10 members) to be sent to the Honorary Secretary on or before Monday, March 17th. No member shall sign more than one paper.

Dr. W. S. Squire. "Note on a Machine for a Continuous Separation of Solids and Liquids."

Mr. H. W. Langbeek. "Wool-Fat, and Processes for obtaining it, with special relation to Wool-Washing."

Mr. A. W. Eastlake. "The Petroleum Industry in Galicia."

April 21st (extra meeting):—

Professor Meldola, F.R.S., and Dr. E. R. Moritz. "Note on the Expansion of Ammoniacal Compounds from Sulphuric Acid used in Kjeldahl determinations."

Professor Dewar, F.R.S., and Mr. Boverton Redwood. "A Process for the Conversion of Heavy Mineral Oils into Lighter Hydrocarbons suitable for illuminating and other purposes."

May 5th:—Mr. Wm. Webster. "The Electrical Treatment of Sewage."

June 2nd:—Dr. W. S. Squire. "The Artificial Production of Cold theoretically and practically considered."

July:—Annual General Meeting in Nottingham.

Meeting held Monday, 3rd February, 1890.

MR. DAVID HOWARD IN THE CHAIR.

## NOTES ON COMMERCIAL CRESYLIC ACID AND ALLIED PRODUCTS.

BY ALFRED H. ALLEN.

IN conjunction with Mr. W. W. Staveley I have been for some time past experimenting with a view of learning more than has been placed on record respecting the metallic compounds or derivatives of the phenols and allied bodies. It had been our intention to place our results before you this evening, but unfortunately our work has met with repeated and unexpected interruptions, and unforeseen circumstances compel a postponement of our paper. Under these circumstances, and with the permission of the Chairman, I propose to bring before you some notes on certain classes of commercial phenols as now manufactured, which notes may really be regarded as an introduction to the postponed paper.

It is well known that carbolic acid and other phenols are manufactured by treating the fraction of coal tar known as "creosote oil" with caustic soda, separating the insoluble hydrocarbons, and then decomposing the phenolate of soda by means of an acid. Sulphuric acid is the acid most generally employed for this purpose, though hydrochloric and sulphurous acids have also been used. Decomposition of the phenolate of soda by carbonic acid, a reaction

associated with the names of Cliff and Hardman, is now practised in one and probably in several works. West-Knights and Gall have patented (No. 5824, 1887) the extraction of carbolic acid from creosote oil by a mixture of sulphate of sodium and lime, whereby sulphate of calcium and carbolate of sodium are obtained. The liquid is separated from the precipitate of calcium sulphate and the carbolate of sodium treated with sulphuric acid, whereby sulphate of sodium is reproduced and the phenols are liberated. Here the inventors seem to have missed an opportunity, for by employing carbonic acid instead of sulphuric, carbonate of sodium is obtained, and this reaction forms the foundation of Mr. W. W. Staveley's ingenious process of alkali manufacture (Patent No. 17,657, 1887), described in a paper read by him before the Liverpool Section of this Society (Journal, VII., 807). I believe this process would be found very suitable for dealing with waste forms of sulphate of sodium, whether solid or in solution, as the plant is comparatively simple and inexpensive, and the reactions are said to be very complete.

The carbolic acid required for decomposing the soda compounds of the phenols is usually obtained by the combustion of coke containing as little sulphur as possible. But in a patent obtained by Mr. R. Angus and myself (No. 11,689, 1887), we described the use of blast-furnace gases to precipitate the phenoloid constituents of blast-furnace tar from their solutions in alkalis. This process is now carried out on a large scale at the Lurgar Iron Works by the same company that own the Gartsherrie Works.

The crude blast-furnace tar is first distilled, whereby it is separated into pitch and creosote oil, and only trifling quantities of more volatile products. The creosote oil is quite different from gas works creosote, being free from naphthalene and anthracene and lighter than water. It contains from 20 to 30 per cent. of phenoloid bodies removable by alkali, and the extraction of these renders the residual hydrocarbons far more suitable for use with the Lueigen and similar lights, though the high market price obtainable at present for crude creosote oil does not afford much encouragement for the extraction of the phenols. The process employed at Lurgar consists in treating the creosote oil with caustic soda solution of sp. gr. 1.11 to 1.15 in quantity insufficient to remove the whole of the phenoloid bodies present. By operating in this way the higher homologues and oxy-phenols chiefly remain with the insoluble hydrocarbons. The alkaline liquid is separated from the oily layer containing the hydrocarbons, and the excess of phenols, and is decomposed by passing through it blast-furnace gases, whereby the phenols are liberated and a solution of carbonate of sodium obtained. This solution, without either concentration or dilution, is causticised by means of lime, when it is ready for treating another quantity of creosote oil, and thus no further alkali is required, except that requisite to supply actual waste. The phenols thus obtained constitute what the patentees have called crude "neosote," a name suggested by its similarity in composition to wood creosote, but which signifies "new preserver." As originally made, the neosote contained a larger proportion of the oxy-phenols characteristic of wood-tar creosote than is at present the case. In some cases the neosote has been purified by partial resolution in alkali, which removes phenoloids of acid character and high boiling point, which properties give them a special value for certain applications. Redistillation is also sometimes practised, as is treatment with steam or air, which removes a notable quantity of sulphur compounds. When newly manufactured, neosote has the appearance of sherry, but darkens on keeping and exposure to light to that of mahogany. When freshly distilled it is perfectly colourless and scarcely distinguishable from the similar fractions of the phenols from gas tar.

The chemistry of the phenols from blast-furnace tar has been investigated by Professor Watson Smith. On treating 14 gallons of blast-furnace creosote oil (sp. gr. 1.088) with caustic soda of 1.07 sp. gr., and decomposing the alkaline solution with sulphuric acid, he obtained two gallons of phenoloids of 1.07 sp. gr. On fractionally distilling this

he obtained 1.33 per cent. of phenol, against a yield of 65 per cent. from Lancashire gas tars. The fraction which would contain the cresols amounted to only  $4\frac{1}{2}$  per cent. of the total phenoloids. The large fraction (19.4 per cent.) distilling between 210 and 230°, Mr. Smith considered probably consisted mainly of phlorol (mixture of the xlenols) with guaiacol and creosol. The results show that a close similarity exists between the phenoloids of blast-furnace creosote and those contained in wood tar. A large proportion of the creosote examined by Watson Smith distilled above 230°, and the portion distilling above 360° gave, on treatment with soda and exposure to air, unstable colouring matters which are probably allied to the eupitomic acid obtained from wood tar.

It is probable that the extraction of the phenoloids from the sample examined by Mr. Smith was not complete. By repeatedly treating a sample of blast-furnace creosote oil of 1.956 sp. gr. with strong caustic soda (sp. gr. 1.21) I extracted 34 per cent. by measure of crude phenoloids having a density of 1.0355. On distillation these gave very little over below 200°, and 59.5 per cent. below 250°, the largest fraction distilling between 220 and 230°.

As already stated, the blast-furnace phenols now manufactured at Lugar differ considerably from those formerly extracted, as is evident by the figures in the table at the top of the next column obtained by the fractional distillation of a sample of commercial neosote with a Glyusky's fractionator.

It will be observed that 27.8 per cent. passed over between 195° and 200°, and an additional 19.8 per cent. between 200 and 205°. The regular decrease in the specific gravity of the fractions is interesting. The original sample gave 3.15 per cent. of hydrocarbon as the result of a very careful determination.

	Per Cent. by Measure.	Sp. Gr. of Fraction.
Below 174° (Water) .....	1.0	.. .. .
From 174° to 190° .....	4.4	.. .. .
From 190° to 195° .....	10.8	1.0435
From 195° to 197½° .....	21.2	1.0450
From 197½° to 200° .....	6.6	1.0390
From 200° to 202½° .....	11.2	1.0340
From 202½° to 205° .....	8.6	1.0290
From 205° to 210° .....	10.9	1.0255
From 210° to 215° .....	7.8	1.0185
From 215° to 220° .....	8.2	1.0125
From 220° to 225° .....	1.0	.. .. .
Total below 225° C. ....	90.7	

Residue (by difference) 9.3 containing 1.05 of hydrocarbon.

The three isomeric cresols are stated by Schulze (Ber. 20, 419) to exist in gas tar approximately in the proportions of 35 per cent. of orthocresol, 40 of metacresol, and 25 per cent. of paracresol. 1:3:5 and 1:2:4 xlenols have been found in the fraction of gas tar phenols boiling between 170° and 210°.

The following table shows the leading physical and chemical distinctions between the three isomeric cresols:—

	Melting Point. °C.	Boiling Point. °C.	Specific Gravity at °C.	Coefficient of Expansion.	Highest Bromo- Derivative.	Highest Nitro- Derivative.
Orthocresol .....	30; 31–31.8	188; 190.8	1.0578	.00072	Br <sub>2</sub> ; m.p. 52°	(NO <sub>2</sub> ) <sub>2</sub> ; m.p. 85.8°
Metacresol .....	4; 3–4	204; 202.8	1.0498	.00078	Br <sub>3</sub> ; m.p. 82°	(NO <sub>2</sub> ) <sub>3</sub>
Paracresol .....	36; 36–37	201.8; 198–199	1.0522	.00086	Br <sub>2</sub> ; m.p. 49°	(NO <sub>2</sub> ) <sub>2</sub> ; m.p. 82.5°

The melting and boiling points recorded by different observers are not in very perfect concordance. The specific gravities and coefficients of expansion are figures by Pinette (Annalen 243, 43), and apparently refer to the liquid cresols, which exhibit, in a marked manner, the phenomenon of superfusion. The "higher bromo-derivative" means the most highly brominated substitution compound which does not react with potassium iodide. Thus, in the case of phenol it would be C<sub>6</sub>H<sub>2</sub>Br<sub>3</sub>OH, and not C<sub>6</sub>H<sub>2</sub>Br<sub>3</sub>OB.

Metacresol forms a tribromo- and trinitro-derivative, whereas the ortho- and para-isomerides form only di-substitution products.

In fractionally distilling neosote several very marked breaks occur. Thus, with the sample in question, the temperature remained absolutely constant for a considerable time at 197° and again at 204°.

By fractional neutralisation and distillation of the lower boiling portions of neosote, crystals of impure carboic acid may be obtained by immersing the liquid in a freezing mixture and adding a minute crystal of phenol. Orthocresol did not induce a similar solidification. The crystalline needles melted at 26.0°C., and reacted with 5.02 atoms of Br, the liquid drained from the crystals reacting with 4.54 Br. Calvert's No. 2 crystallised carboic acid reacted with 6.04 Br.

The examination of other samples of neosote has given much the same results as the above, and shows that the article consists chiefly of cresols and their homologues. The presence of the higher homologues has some practical interest, for as a consequence neosote is considerably less volatile and soluble in water than is cresylic acid. The following experiments were made exposing known weights

of neosote and Calvert's No. 5 carboic acid side by side in shallow vessels.

Time of Exposure in Days.	Loss by Volatilisation.	
	Neosote.	No. 5 Carboic Acid.
	Per Cent.	Per Cent.
1	0.84	5.45
2	1.95	7.99
3	3.24	9.39
4	4.55	12.11
5	5.25	13.30
7	5.95	14.18
8	6.65	14.43
11	10.57	19.82
14	14.20	25.02
17	16.68	30.07
21	18.02	35.02
24	19.10	38.46

This moderate volatility of neosote may be an advantage or the contrary, according to the circumstances under which it is intended to be employed.

For comparison I may quote the following results obtained by the distillation with fractionating bulbs of a sample of

crude commercial cresylic acid of 1·049 sp. gr. manufactured from gas-tar :—

—	Measure of Distillate.	Specific Gravity of Fraction.
	Per Cent.	
First drop over at 90° C., water..	8·5	.. ..
Below 180° C. ....	2·5	.. ..
From 180° to 190° .....	4·0	.. ..
From 190° to 195° .....	10·5	1·045
From 195° to 200° .....	20·0	1·043
From 200° to 205° .....	10·0	1·037
From 205° to 210° .....	9·5	1·032
From 210° to 215° .....	6·0	.. ..
From 215° to 225° .....	6·6	.. ..
Total below 225° .....	89·5	.. ..

A sample of "once-run" cresylic acid had a specific gravity of 1·038, and gave on distillation with fractionating bulbs the following figures :—

—	Measure of Distillate.	Specific Gravity of Fraction.
	Per Cent.	
First drop over at 95° C., water..	9·5	.. ..
Below 190° .....	1·5	.. ..
From 190° to 195° .....	15·5	.. ..
From 195° to 200° .....	36·0	1·041
From 200° to 205° .....	19·5	1·034
From 205° to 210° .....	11·0	1·029
From 210° to 215° .....	6·0	.. ..
From 215° to 228° .....	6·5	.. ..
Total below 228° .....	96·5	.. ..

Another sample of commercial cresylic acid of sp. gr. 1·041 gave in the same apparatus :—

—	Measure of Distillate.	Specific Gravity of Fraction.
	Per Cent.	
First drop over at 120° C., water.	None	.. ..
Below 180° .....	0·2	.. ..
From 180° to 190° .....	4·6	.. ..
From 190° to 195° .....	56·6	1·046
From 195° to 200° .....	23·0	1·039
From 200° to 205° .....	10·0	1·030
Total below 205° .....	96·4	.. ..

Calvert's "No. 5 carbolic acid" gave the following results when fractionated :—

	Per Cent.
Below 170° .....	6·8 containing 5·5 per cent. of water.
Between 170° and 183° .....	2·0
Between 183° and 186° .....	3·6
Between 186° and 190° .....	46·8 of 1·0541 sp. gr.
Between 190° and 195° .....	28·8 of 1·0430 sp. gr.
Between 195° and 200° .....	7·0 of 1·0370 sp. gr.
Total below 200° .....	95·0

The antiseptic properties of cresylic acid are generally regarded as being fully equal to, if not superior, to those of carbolic acid, and direct experiment has proved this to be true also of the higher homologues, while if the acrid body of high boiling point already referred to be removed, the effect of the higher homologues on the skin is far less caustic than that of carbolic acid.

I must leave a detailed consideration of the metallic compounds or derivatives of the higher phenols to a future occasion, but may say that I have met with great difficulty in preparing them of definite composition. Of the heavy metals, the phenolates of lead and mercury are the most readily prepared.

I may point out that the phenols present one peculiarity which is capable of several interesting applications. I refer to the fact that their calcium salts are readily soluble in water, but decomposable readily and completely by carbonic acid. With the exception of the compound of lime with cane sugar, and the sulphhydrate of calcium, no other calcium salts occur to me of which the same can be said; and in these cases there is not the same precipitation of the acid in a separate and readily available form that occurs with the phenolates.

#### DISCUSSION.

The CHAIRMAN said that the Section had reason to thank Mr. Allen heartily for the fraction of his paper which he had been able to give them. Some halves were greater than some other wholes; and the matter which Mr. Allen had brought before them was deeply interesting as throwing light upon a most important modern development of chemistry. He would call upon Professor Smith to give the meeting the benefit of his special experience in this direction.

Mr. WATSON SMITH had listened with great pleasure to the particulars brought forward by Mr. Allen, and especially to his promise of further details on this interesting subject. He had started the work in this direction himself, and might mention that it was by isolating in as pure a condition as possible a number of the phenoloid constituents of blast-furnace tar, and afterwards passing them over red-hot zinc dust and obtaining the corresponding hydrocarbons, that he had obtained to some extent the clue to the nature of the phenols which gave rise to them. He did not trouble to pass the carbolic acid over, as it was easy to identify that by crystallisation. But he obtained the cresol in the shape of toluene, and some xylenes afterwards, as also pseudocumene and mesitylene, and a quantity of naphthalene derived from the naphthols present. In his first communication on the subject he had said that probably guaiacol and cresol would be present also; but in his later experiments he had found no indications of their existence in this blast-furnace tar. He could not conceive that guaiacol, especially, could be present and not be detected, for it had a peculiarly strong odour—indeed, it was to the presence of guaiacol in wood tar that the extremely powerful smell of that body was due. In the samples of phenols presented by Mr. Allen, the peculiar odour of guaiacol was not perceptible to him, and they thus so far corroborated the observations made upon the fractions isolated by himself. It was probable, therefore, that what they had before them were merely a mixture of the homologues of phenol, with possibly some hydroxyphenols, but none of the methylated derivatives. Mr. Allen had stated that his cresol contained 1 per cent. of water, and had referred to that as being lower than the usual percentage in commercial cresol. No doubt that would be due to its being freshly distilled.

Mr. A. H. ALLEN, interposing, said that the cresol to which he referred was two years old. It was to be observed that it was a product of distillation of blast-furnace tar, whereas ordinary commercial cresol was a by-product of the manufacture of carbolic acid.

Mr. WATSON SMITH, continuing, said it was characteristic of the cresols that they absorbed or contained less water than phenol. In fact, that characteristic was used as a distinctive proof of identity, and a tar distiller would know that his crude carbolic acid was good if water were present in considerable quantity (about 13 per cent.); whereas if

only a small quantity, say 5 or 6 per cent. were present, he would expect to find a considerable proportion of crude cresol. The small percentage of water contained in Mr. Allen's cresol, therefore, went far to show that it was an unmixed product. The reason for this small percentage as compared with phenol was to be found in the fact that, in the case of cresol, hydrates were not formed, whereas in the case of phenol two hydrates are known, and this accounted for the corresponding absorption of water. The discovery of these hydrates had been the result of special study by Mr. Charles Lowe, of Manchester.

### FEODOSSIEFF'S NEW METHOD OF TEMPERING AND HARDENING STEEL FOR CANNON AND PROJECTILES.

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PROFESSOR W. C. ROBERTS-AUSTEN, in his very able lecture before the British Association at Newcastle-on-Tyne last year, pointed out the confusion at present existing in the use of the words "hardening," "tempering," and "annealing" as applied to steel. He gave the following definitions: "*Hardening*" is the result of rapidly cooling a strongly heated mass of steel; "*Tempering*" that of reheating the hardened steel to a temperature far short of that to which it was raised before hardening—this heating being followed or not being followed by rapid cooling; whilst "*Annealing*" consists in heating the mass to a temperature higher than that used for tempering, and allowing it to cool slowly.

As I shall hereafter show, the method to be described can either be used for hardening steel or may be modified for tempering or annealing. This method has been invented by Captain G. Feodossieff, of the Imperial Russian Navy (this Journal, 1889, 548). Glycerin is the substance or medium used, and it is employed for the tempering or hardening of steel, cast steel or cast iron. It is proposed to vary the specific gravity of the glycerin from 1.08 to 1.26 at 15° C. by the addition of water according to the composition of the steel and the effects desired. The quantity of glycerin is to be from one to six times greater in weight than that of the pieces to be plunged into it, and in accordance with the hardness of the metal its temperature is to be varied from 15 to 200° C., a higher temperature being employed for the tempering of the harder steels, whilst a lower temperature is used for tempering the milder steels.

Additions of various salts to the glycerin baths are recommended to increase their quenching power. Thus, for a *hard temper*:—Manganous sulphate may be added in quantity varying from 1 up to 34 per cent. of the liquid, or from  $\frac{1}{4}$  to 4 per cent. of potassium sulphate. For a "*softer temper*," 1 to 10 per cent. of manganese chloride and 1 to 4 per cent. of potassium chloride may be added.

The principal advantages claimed for these processes are as follows:—

- (1.) The temperature of the aqueous solutions of glycerin may be varied within wide limits, the boiling point of pure glycerin being 290° C. (By a misprint our Journal, 1889, 548, gives 29° instead of 290° C.)
- (2.) Owing to the fact that solutions of glycerin in water dissolve most salts that are soluble in water, the quenching powers of the glycerin may be readily varied by dissolving such salts in the bath to suit the kind of metal to be tempered and the degree of temper required.

It will be observed that in the directions given in the patent, just those are defined which are necessary for the manufacturer or workman, who already possesses a general knowledge of the subject of "hardening" and "tempering." However, since the date of this patent, Captain Feodossieff writes me that he has made new and further improvements,

and has succeeded in tempering heavy projectiles, armour-plate and railway tyres by his glycerin process, and has obtained very satisfactory results. I have now much pleasure in exhibiting some specimens of steel treated by Feodossieff's process. The first is a 4-inch cast-steel ingot which had been made red hot and then immersed in glycerin. An examination of the upper part of it will show that a peculiar shrinkage has taken place, which proves that the metal has not been forged. This ingot, sent me by Captain Feodossieff from St. Petersburg for exhibition with the other specimens at this meeting, was specially sent for the purpose of showing us that the qualities of the metal are derived from the manner of hardening and not from forging.

Specimen No. 2 is a part of the same ingot as No. 1, but which has been forged, raised to a low heat and then plunged into glycerin. After cooling it was bent at an angle of 180°. The tensile strength of this piece of annealed steel is 65 tons, elongation 15 to 18 per cent. The specimen before us was bent cold. Specimen No. 3 is of forged steel, tempered in glycerin. All three pieces now exhibited, Nos. 1, 2, and 3, are fragments of the same 4-inch ingot.

With regard to the specimen 2, which was bent upon itself cold, analysis proved it to have the following composition: carbon, 0.7 per cent.; chromium, 2.2 per cent.; and silicon, 0.15 per cent. Captain Feodossieff has experimented with unforced cast-steel 12-inch projectiles. One of these penetrated 12-inch armour-plate made by Cammell; the head part of the projectile fell at 10,000 feet behind the target. He now makes 12-inch projectiles which penetrate 16-inch armour-plate. Various experiments have proved that Feodossieff's method of hardening produces an intrinsic change in the molecular structure of the metal.

The inventor proposes that experiments should be made now with guns, which are so difficult to forge, but such experiments ought to be made on a large scale in works like those of Lord Armstrong and Co., Limited, at Elswick, or of Sir Joseph Whitworth and Co. I need hardly point out the great importance of this suggestion if it should be found that by such a uniform method of treatment the absolute uniformity of structure of the strongest and toughest kind so much desired in large ordnance will be insured, and that thereby, as one would *a priori* conclude, a great decrease in the cases of the bursting of cannon be the result, or that such cases be reduced to as nearly zero as possible. When one thinks of the operation of forging it is difficult to believe that, even with the best mechanical contrivances, the result, can be absolutely uniform in all parts of so large a piece of metal as that serving for the construction of a large piece of cannon. If not uniform we must expect to have strong and also weaker places. When it comes to a process of treatment by immersion in a liquid like glycerin, it becomes then difficult to discover why a uniform process with uniform results should not be attainable.

### DISCUSSION.

The CHAIRMAN said that the subject of the tempering and hardening of steel was one of those obscure questions lying on the border-land of chemistry and physics, which recent discoveries had thrown some light on, but which were still most difficult to deal with. The consideration of how far this particular process was likely to be successful in overcoming the difficulties of the problem, he must leave to those members, several of whom were present, having that special experience which would enable them to decide.

MR. A. H. ALLEN found it difficult to understand why there should be any great difference in the behaviour of steel when cooled by immersion in glycerin and when cooled by immersion in oil, which was the ordinary method of proceeding. He would have expected that all that was required was a viscous liquid with a high boiling point; and that so long as the necessary condition of viscosity or mobility was secured, it would not matter much whether the steel was hardened in glycerin or oil, or other suitable liquid. He desired to protest against what he considered a misuse of the term tempering. He understood Professor Smith to use that term to express the process of dipping or immersing the hot steel in glycerin or other liquid with a

view to produce a *hardening* effect. It was usual, however, to restrict the term tempering to that *softening* by moderate heating, which was carried out *after* the hardening—to *reduce* the hardness. It might be that the tempering or softening process was prevented from going too far by again immersing the steel in water or other liquid, but this could not be considered essential except in a supplementary sense, and was not tempering. If the author had made his steel as hard as he could by sudden cooling, and had then heated it up in glycerin, instead of relying on the film produced on the surface or other indication that the requisite temperature had been attained, that might have been called tempering. As used in the paper, the term was likely to cause confusion, and he hoped, therefore, that Mr. Smith would modify his use of it before it was printed.

Mr. WATSON SMITH desired to remind the meeting that, in communicating Captain Feodosieff's results, he was in a position of some difficulty, especially with respect to the terms employed. For that reason he had begun the paper with the definition of hardening and tempering given by Professor Roberts-Austen. He was at a loss to know how he could, with propriety, make any radical alterations in Captain Feodosieff's own statements.

Mr. BERTRAM BLOUNT thought that some attention should be paid to the fact that the steel treated by the author of the paper was not of normal composition, but contained chromium. Reference to the peculiar properties of such steels had been made by Mr. Arnold, of Sheffield, in a paper read before the Institution of Civil Engineers last year, on the subject of the use of steel for railway tyres. Mr. Arnold had, if he remembered rightly, found that for producing a steel moderately hard, and free from tendency to failure, it was safer to use chromium than to increase the percentage of carbon. But such a steel as that could hardly be compared with ordinary steel. Another point that had occurred to him was that these highly conductive—or, rather, high boiling point—liquids, though distinctly of use, should not be necessary in the hands of any workman of ability. It had been said that a man who could not temper steel with fire and water merely ought to abandon the trade.

Mr. JOS. BERNAYS said that he had had some experience in the hardening and tempering of steel. Tempering could be done in various ways; and he agreed with Mr. Smith's definition of it as a slighter reheating of the steel, and plunging it into a liquid. The process was so carried out in every smith's shop in the making of tools, and similar articles of steel. They were first heated and made thoroughly hard; then they were heated again to a lower temperature, and were then again plunged into water for thorough cooling. He would not say that tempering might not be done in other ways; but the method he had described was the one usually followed in an engineer's shop. He had done the operation frequently himself, and had by it produced a proper temper in steel tools, springs, &c.

Mr. A. H. ALLEN inquired which operation was considered by Mr. Bernays to be tempering—the warming up after hardening, or the plunging into water afterwards?

Mr. BERNAYS replied that both were necessary, that the steel was first heated to redness, and plunged into cold water or oil for making it hard. It was, however, not quite cooled down. The inside of the steel was left hot, and a bright spot was then filed at the part to be tempered. As the surfaces were getting reheated from the inside, the bright part assumed various colours, first, pale straw, then pale blue, and lastly dark blue, according to the heat transmitted from the interior. As soon as the right degree was arrived at, the steel was again plunged into water and thoroughly cooled, and was then in the condition of "tempered" steel.

Mr. ALLEN asked whether the last operation was considered essential for any purpose besides getting the metal cooled quickly and conveniently.

Mr. BERNAYS said it was necessary to prevent the metal getting still hotter.

Mr. ALLEN feared that Mr. Bernays and he were speaking of different things. Mr. Bernays was probably referring to large bodies of metal, whereas he was considering small articles, in which case any final artificial cooling to prevent spoiling of the temper already attained was unnecessary.

Mr. BERNAYS replied that he was speaking of articles of the size of the samples before them, which had a considerable thickness. They might be reheated in a different way, by bringing them to an external source of heat.

Dr. SQUIRE wished to point out, with respect to Mr. Allen's remark, that the process described in the paper was not under the same conditions as the method of tempering by means of oil. There was a difference which might perhaps be of importance. The specific heat of glycerin was higher than that of oil; and, moreover, he understood the author to say that the glycerin might be mixed with more or less water, by which its specific heat would be further increased. The greater the specific heat of the liquid into which the steel was plunged the more rapid would be the cooling; so that the dipping into glycerin more or less diluted appeared to be a sort of halfway method between dipping into water and dipping into oil.

Mr. ALLEN said that was true.

Mr. E. RILEY said that Mr. Hadfield, who was present, would probably be able to tell the meeting something about the effect of adding chromium to steel. He might say, however, that its use had been known for 12 years past. Hundreds of tons of chromium iron had been used for making axles at Messrs. Brown, Bayley, and Dixon's works, because it was found that the addition of a little chromium pig to the steel enabled it to stand a very severe falling weight test. That was in 1878 or 1879, and the fact was then a trade secret.

Mr. R. A. HADFIELD feared that he could not give the meeting much information as to the effect of chromium on steel. His own experiments indicated that combination of chromium and iron only did not give hardness, and that the prevailing impression that they did was a wrong one. Carbon alone seemed capable of giving the hardness to iron, that is to the material termed "steel"; and unless carbon was present, chromium was no more effective than manganese, silicon, or other element. Perhaps it might be well for the author to know that the results he had quoted as to tests of projectiles were very different from those required by our own War Office. A 12-in. shell penetrating a 16-in. plate would not be considered sufficient by our Government. They would require it to pierce something like 20 inches of compound steel and iron plates—which was a severe test. Thanks to the assistance which the Government was now giving to English manufacturers, they were accomplishing similar results, equal and superior to those of foreign makers. A short time ago his firm, Hadfield's Steel Foundry Co., Limited, had sent two 6-in. shells through 9-in. compound plates and 8 ft. into the wood backing, and both shells were practically undamaged. It was satisfactory to know that his own firm and other English makers had been able to fulfil the requirements of the War Office, and equal foreign productions. There was considerable misconception as to the definition of the word "tempering." He thought that Mr. Allen was perfectly right in his view, and that hardening must be defined as a distinct process and not mixed up with tempering. Probably the error was partly explained by the fact that the French always use the word "*tremper*" to indicate hardening; thus the English and French expressions had got mixed and led to this prevalent misconception. Hardening, he thought, must be defined as the turning of the carbon from the state in which it was found in unhardened metal into the hardened compound, in which form it would scratch glass; if this result was not attained then the term "hardening" could not be applied. Tempering in all Sheffield trades meant a subsequent and separate operation or reheating and reduction of hardness. The form of that compound was not known at present, but thanks to the experiments of scientists much was being

found out about it. In the meantime it would be best to stick to the good English word "hardening," which clearly defined the operation. Tempering was a different process altogether.

Mr. E. RILEY quite agreed with Mr. Hadfield as to the effect of chromium. His firm had used it in the manufacture of soft steel, because they had found that it increased the tensile strength of the metal without increasing its hardness. It was expensive to use, however, and so they did without it as far as possible.

Mr. R. A. HADFIELD said that it was a striking instance showing how men's minds in different parts of the world unconsciously worked on the same idea, that experiments had been made in America within the last two years in the same direction as the process now described by Professor Smith.

## BROMANIL.

BY S. M. LOSANITSCH.

*Professor of Chemistry in the Royal Serbian Academy.*

IN a communication on the action of nitric acid on ordinary tribromaniline, melting at  $119^{\circ}\text{C}$ , I have shown that in the reaction which takes place along with a certain amount of other products, bromanil is produced in considerable quantity (about 10 per cent.) (Ber 15, 471).

This reaction for the formation of bromanil is important, since by its aid the pure substance can be easily prepared. It is only necessary to boil the tribromaniline with strong nitric acid till the decomposition products (dibromdinitromethane and tetrabrombenzene) have distilled over, afterwards washing the residue with hot alcohol, in order to obtain bromanil in pure golden-yellow crystalline plates. In the investigation referred to, I have thus obtained several hundred grammes of pure bromanil as a by-product, and this material served for the further study of bromanil.

*Bromanil* is easily soluble in hot benzene, and tolerably soluble in glacial acetic acid, but almost insoluble in alcohol. According to an earlier report, it is considerably soluble in boiling alcohol (Ann. 143, 255; 91, 307; 205, 53), but this is to be ascribed to the presence of impurities.

In the analyses, the bromine found was in one case 75.30 per cent., and in another 75.45, the calculated amount for  $\text{C}_6\text{Br}_2\text{O}_2$  being 75.47 per cent. The product obtained was therefore a pure bromanil. It crystallised from glacial acid in shining rhombic plates of a sulphur-yellow colour, and from benzene in thick dark yellow plates. By letting hot under-saturated benzene solutions of the substance stand in beaker glasses for some time, I succeeded in obtaining perfectly formed crystals of bromanil.

From such a solution, after some days, beautifully developed crystals were obtained, and these were crystallographically examined by my friend Professor A. Arzruni. He states that "bromanil crystallises in the monoclinic system. The crystals are of an orange-yellow colour (corresponding to the tint 4 K. of Rüdke's colour scale by incidental, and 6 N. to 7 N. by transmitted light), and they are very lustrous.

"The forms observed are (001), (110), and (101), which all exhibit very even surfaces.

"The crystals are, without exception, twins upon the face (001).

"The fact has been deduced by repeated experience, and in such crystallisation no sensible disturbance in the formation and angle values were observed. This is shown in the following table:—

		Measured.	Calculated.
110	110	$73^{\circ} 29'$	..
110	001	$86^{\circ} 46'$	..
110	$\bar{1}\bar{1}0$	$18^{\circ} 24'$	$18^{\circ} 28'$
001	$\bar{1}01$	$77^{\circ} 58\frac{1}{2}'$	..
$\bar{1}01$	$10\bar{1}$	$24^{\circ} 8\frac{1}{2}'$	$24^{\circ} 7'$

"The values prefixed by an asterisk above lead on calculation to the axis relationship:— $a:b:c = 1.3905:1.2:9335$ , and the axial inclination  $\beta = 74^{\circ} 26\frac{1}{2}'$ . A very complete cleavage is manifested in a direction parallel to (001). The plane of the optic axis coincides with that of the symmetrical axis."

Bromanil is not soluble in concentrated sulphuric acid, but on long boiling it dissolves slowly with liberation of bromine, and is converted into a dirty red crystalline powder. This powder consists of bromanilic acid. I have investigated two salts of this acid. The silver salt,  $\text{C}_6\text{Br}_2\text{O}_2(\text{OAg})_2$ , separates as a dark red amorphous precipitate on the addition of nitrate of silver to the aqueous solution of the acid. The percentage of silver found in this salt was 42.19, whilst the formula given requires 42.19 per cent. The barium salt,  $\text{C}_6\text{Br}_2\text{O}_2\text{Ba}$ , separates as a brown crystalline powder when bromanil is boiled with an aqueous solution of barium hydrate. This powder dissolves in boiling water slightly acidulated with hydrochloric acid, to which it communicates a violet colour, and from this solution the barium salt separates in brown prisms grown together in the form of crosses. In this salt 31.15 per cent. of barium was found, the formula demanding 31.64.

*Anilide of Bromanilic Acid*,  $\text{C}_6\text{Br}_2\text{O}_2(\text{NH}\cdot\text{C}_6\text{H}_5)_2$ , separates in shining rhombic plates on adding aniline to a boiling solution of bromanil in benzene or glacial acetic acid. These crystals have a beautiful bluish-violet colour in reflected light, and appear yellow by transmitted light. The compound was first prepared by Stenhouse, and according to him, it crystallised in black tables (Ann. Suppl. 8, 22).

*Toluide of Bromanilic Acid* (ortho and para).—



Ortho and paratoluidine combine with bromanil to form these toluides when they are boiled with benzene or glacial acetic acid.

The *ortho-toluide* separates in dirty yellow, the *para-toluide* in dark red rhombic plates. The latter compound appears yellow by transmitted light. Both these toluides are dissolved by benzene or glacial acetic acid, yielding yellow solutions. The *ortho*-compound is more easily soluble than the *para*. The nitrogen was determined in these compounds, and found to be 5.92 and 5.89 per cent. respectively, the formula demanding 5.88 per cent.

*Xylide of Bromanilic Acid*,  $\text{C}_6\text{Br}_2\text{O}_2[\text{NH}\cdot\text{C}_6\text{H}_3(\text{CH}_3)_2]_2$ . The benzene solution of the bromanil turns brown on addition of xylidine (ordinary). This solution was evaporated to dryness, and the residue dissolved in glacial acetic acid. From the solution the xylide crystallises in yellow plates. Nitrogen found = 5.3 per cent.; the formula requiring 5.56 per cent.

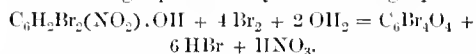
## DISCUSSION.

On finishing the reading of the communication, Mr. WATSON SMITH said that the only indication he could at present see of the practical application of bromanil was the possibility of its being used in the manufacture of colouring matters. But unless its use was found to constitute some considerable improvement on Chloranil blue, he feared that there was not much chance of its employment even in that direction. Chloranil was produced very cheaply at present by the reaction of potassium chlorate and hydrochloric acid on pure phenol. The chloranil thus formed combined on heating with the aromatic tertiary amines, yielding a fine blue colour, or by heating with benzyldiphenylamine, a fine green colouring matter was obtained. Whether it might be possible by substituting bromanil to produce an analogous variation of shades such as were obtained, for example, in the Eosin group, where, by brominating fluorescein the tetra-brominated product (ordinary Eosin) of yellow shade is formed, or by iodising the tetra-iodo-compound (erythrosin) of blue shade, would be obtained, only actual experiment could decide.

The CHAIRMAN said that he would be a bold man indeed who should say of any given process that it would never become valuable in technical chemistry. Remembering an instance

in his own student days, when some carbolic acid was required and there was nothing to prepare it from except the alkaline washings of some coal tar—he was never surprised to find chemical rarities of a few years back becoming the common productions of to-day. A purely scientific paper of to-day might in a few years hence be the source of a valuable chemical industry: and therefore such papers should be regarded with respectful, if not active, interest.

Mr. A. R. LING wished to draw attention to the fact that he had described a method in the Chemical Society's Journal, 1887, 148, by which bromanil could be obtained in theoretical quantity. This consisted in heating dibrom-*ortho*- or dibrom-*para*-nitrophenol with bromine and water, the reaction being represented by the following equation:—



Since dibrom-*ortho*-nitrophenol could be readily obtained by nitrating crude dibromophenol (from phenol and bromine), this method appeared to him to be worthy of attention, should bromanil become of technical importance. With regard to bromanilic acid the constitution of which had recently been determined by Hantzsch—to the other compounds mentioned in the paper, he thought that they had been already fully described by Stenhouse and others.

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SESSION 1889-90.

Notices of Papers and Communications for the Meetings to be sent to the Local Secretary.

Meeting held Wednesday, January 15th, 1890.

DR. F. HURTER IN THE CHAIR.

### NOTE ON A GLASS TOWER CONDENSER.

BY DOUGLAS HERMAN.

As having some relation to the principal business of this evening—the discussion of Dr. Hurter's masterly paper on the "Condensation of Hydrochloric Acid Gas"—I thought it might be of interest to the members to see how a tower condenser can be made practically entirely of glass.

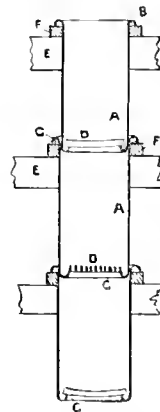
In the laboratory, for the condensation of acid or corrosive gases (except of course in the case of fluorine), no chemist probably would think of using any other material than glass, its superiority being so manifest. On the large scale,

however, especially where it is desired to employ a tower filled with some material exposing a large surface, the difficulties in the way of employing glass are great, as a fracture in the lower part of the tower caused by the pressure of any considerable height of packing material or otherwise, might empty the tower of its contents. If the glass walls of the tower were made sufficiently thick to be reasonably safe against such an accident they would be very costly, besides being liable to fly with changes of temperature. Moreover, one of the principal advantages to be derived from the use of glass, namely the cooling effect arising from the thinness of the material, would be to a considerable extent neutralised.

In the apparatus before you an attempt has been made to overcome these difficulties, with what probability of success I must leave it to members to determine who have far more practical acquaintance with the subject of the condensation of acids, &c., than I can pretend to.

The principle adopted is to divide the vertical height of the tower into sections of two or three feet, so arranged that each section shall carry the entire weight of packing material contained within it, without in the slightest degree pressing upon the portion of the tower below it. A dangerous pressure in the lower parts of the tower is thus avoided, and the tower may be built to any height that is considered desirable.

The principle of sub-dividing the weight is carried into effect by providing slightly tapering glass pipes, *a*, with an upper external and lower internal flange, of the form shown



in section at *b* and *c*. The upper flange, resting upon suitable supports, *e, f, g*, carries the whole weight of the pipe with its contents. A grid, *d*, supported by the lower internal flange, carries the packing material. The pipes being slightly taper, the lower portion of one pipe fits into the upper portion of the pipe immediately below it, and the joint is made good with any suitable cement. Flanges of the form shown, in addition to serving the purposes described, materially add to the strength of the pipe.

Bends to connect one tower with another can be made of glass, but it is practically impossible to make large tees of this material, and where such connexions are necessary, earthenware should be employed. If desired, the bottom pipe of each tower may be made funnel-shaped to facilitate the conveying away of the liquid product.

The grids, *d*, supporting the packing material are composed of strips of glass,  $\frac{3}{8}$  in. thick by  $1\frac{1}{2}$  in. deep, and to prevent the liquid trickling along these to the sides of the tower, they are cut with a slight curve, from the lowest part of which the liquid has a tendency to drop. By placing the grid in one pipe at right angles to that in the one immediately above it, the centre of distribution of the descending liquid is continually changed from pipe to pipe.

The packing material may be glass, and if in the twisted irregular form here shown, will weigh, with the supporting grid, about 1 cwt. per pipe.

If sufficient taper is given to the pipes, and care is taken not to overfill them with packing, a damaged or obstructed pipe may be lifted out and another one substituted, without interfering with the rest of the tower.



The pipes are made about  $\frac{1}{8}$  in. thick, and in point of price will compare favourably with earthenware, doing the same amount of work.

Whatever the practical value of such an apparatus may be—and it is perhaps better adapted for the preparation of pure acids, &c., than for the condensation of the crude hydrochloric acid of alkali works—it certainly satisfies one of the requirements insisted on by Dr. Hurter as necessary for successful condensation, namely, that of providing “ample cooling surface in that part of the apparatus in which the gas comes in contact with the condensing liquid.” Each pipe of the size shown has a capacity of about 3 cub. ft., and a surface area of over 9 sq. ft., or say 3 ft. of surface to one of capacity. In the stone condenser, 40 ft.  $\times$  8 ft.  $\times$  8 ft., employed by Dr. Hurter in some of his experiments, the relation of vertical surface to capacity is only as 0.5 : 1, and the sides of course are enormously thicker in the case of stone than in glass.

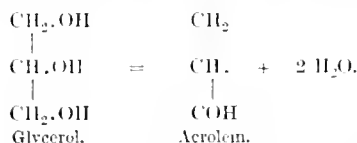
### A QUALITATIVE TEST FOR GLYCEROL.

BY CHARLES A. KOHN, B.Sc., PH.D.

*Demonstrator of Chemistry, University College, Liverpool.*

Is the following qualitative test for glycerol advantage is taken of two characteristic reactions:—

(1.) The formation of acrolein by the distillation of glycerol with acid potassium sulphate, according to the equation:—



(2.) The red colouration produced with a solution of rosaniline, decolourised by sulphur dioxide, by acrolein, as by all aldehydes (Schiff and Caro's reaction).

To carry out the test the solution containing the glycerol is evaporated down to about 5–10 cc. in a small porcelain dish, about 1 gm. of powdered acid potassium sulphate added, the whole well mixed together and then taken to dryness on the water-bath. The residue is transferred to a hard glass tube, 3–4 in. long, fitted with a cork and exit tube, the latter passing into a test tube containing water. On heating the tube the acrolein is readily evolved and is dissolved by the water in the test-tube; the contents of the latter can then be tested for the presence of an aldehyde in the usual way by the addition of a few drops of Schiff's reagent. Both before and after the addition of the reagent the solution in the test-tube should be well shaken; the colour only develops slowly and reaches its maximum after 15–20 minutes standing. If the aqueous solution happens to have got warm by the condensation of steam evolved during the distillation, it must be cooled before adding the reagent, since the latter turns red on warming in the absence of an aldehyde.

The delicacy of the reaction has been tested by Mr. J. T. Conroy, B.Sc., and Mr. B. W. Jones, with the result that it is possible to detect 0.015 gm. of glycerol in this way: or in round numbers, 1 part of glycerol in 650 parts of water, by evaporating down 10 cc. of the solution, and therefore 1 part of glycerol in 6,500 parts of water by evaporation down 100 cc.

The reaction is not given by any of the following substances: Mannite, cane sugar, grape sugar, milk sugar, starch, dextrin, albumen, gelatin, stearic acid and oleic acid; but the carbohydrates interfere with the delicacy of the test owing to the fact that their distillation products with acid potassium sulphate hinder the formation of the red colour with the rosaniline solution. To carry out the test in presence of sugar, the latter must first be removed by evaporating the solution in the presence of calcium

hydrate and sand or of calcium hydrate and calcium carbonate, and extracting the residue with a mixture of 2 parts of alcohol to 1 part of ether. On evaporating this extract with acid potassium sulphate and distilling the residue, the reaction is obtained as above, but it is not possible to detect less than 0.03 gm. of glycerol in the presence of much sugar, or 1 part of glycerol in 3,300 parts of water by evaporating down 100 cc. of the solution.

The reaction must be similarly applied in testing for glycerol in wine and in beer; the limit of its delicacy in these cases is the same as above. In testing milk for glycerol the casein, albumen, and sugar should be first removed.

Fatty acids do not interfere with the delicacy of the reaction.

Compared with other colour reactions for glycerol, this test appears more characteristic, though not quite so delicate as Reichel's phenol test (this Journal, 1882, 202). It is considerably more delicate than Reichel's pyrogallol test (this Journal, 1883, 356), and also more delicate than the borax test (Senier and Lowe, J. Chem. Soc. 1878, 401).

Mr. E. CAREY asked whether it would not be possible to apply the test for the quantitative determination of glycerol colorimetrically?

Dr. KOHN replied that this might be done as long as only small quantities of glycerol were concerned, but he did not consider that the depth of colour obtained would serve as a quantitative reaction when the tint first got was a fairly deep one, even if dilution were resorted to for comparison.

### ADJOURNED DISCUSSION ON DR. HURTER'S PAPER ON THE CONDENSATION OF HYDROCHLORIC ACID GAS.

(See Journal, VII., 861–67.)

Mr. A. E. FLETCHER said he had read the paper by Dr. Hurter with great interest. His own experience had led him to advocate largely what Dr. Hurter had pointed out, viz., that condensers should be kept cool. It was of great importance to abstract the heat which was generated by the act of condensation. A question might arise as to the wisdom of using capacious coke towers as condensers, as being likely to retain the heat of condensation, but probably the reason why such large vessels were used was that the supply of hydrochloric acid was intermittent. A large massive coke condenser acted both as a condenser and a water receiver. The supply of hydrochloric acid was intermittent, and at the time the gas approached the condenser the latter was nearly full of almost clean water. The gas attacked it at the bottom and gradually gained on the water supply, which was constant and slow. It might reach near the top of the tower, but before it did so the supply of acid diminished, because the charge in the decomposing pot was getting spent; then the water gained on the hydrochloric acid, thus preparing the tower to receive the next charge of acid gas. He thought that was the *raison d'être* of large condensers, otherwise it would be much better to have smaller vessels, such as pipes and tanks.

There was an interesting passage in the paper calling attention to the fact that air, passed through hydrochloric acid of 12° Tw. would come away with about 0.2 gm. HCl per cubic foot. This did not, however, affect the question of condensing, because the top of the condenser, being filled with clean water, the hydrochloric acid came in at the bottom and passed away from the top. The solution of HCl thus became weaker as it rose up in the tower.

A point of practical importance in the condensation of hydrochloric acid not mentioned in the paper was the difficulty of wetting particles of matter when disseminated through a gas, producing a sort of acid fog. The hydrochloric acid must be supposed to be condensed already, but the

difficulty was to precipitate it. If they put some water into a bottle containing vapour of HCl mixed with air, it needed much shaking of the bottle to induce the water to take up the fine particles of hydrochloric acid. So in condensing, the great difficulty was to produce contact between the fine particles of acid and water. If this did not exactly come within the scope of the paper, yet being closely associated with it, he thought some attention might be given to the point.

Mr. R. F. CARPENTER had read the paper with extreme interest, and had worked out the calculations in Dr. Hurter's examples, so that he might thoroughly grasp the points Dr. Hurter desired to emphasise. In doing so, he had found two or three things that required correction.

On p. 863, second column, the number of units of heat given out by the condensation of 501 lb. HCl was given as 192,384. The strength of the acid condensed was not given, but this must be, if the heat units are correctly stated, higher than 36° Tw., and it is certainly doubtful, if on the large scale an acid stronger than 36° Tw. can regularly be obtained. Following on to p. 864, second column, in the example where percentage of HCl is 14.1, H<sub>2</sub>O, 15.1, &c., and  $x_0$  of the formula is given as 0.206, this should certainly be 0.151, as given correctly a few lines lower down.

He was much interested in the examples given by Dr. Hurter of the amount of condensing work done in the cooling pipes, as he had taken part, 12 or 14 years ago, in experiments on the large scale, having for their object the production of dry hydrochloric acid gas. With that view the salt used was subjected to a preliminary drying, and as strong an acid as possible was used, the gases mixed with air being cooled in glass pipes to air temperature. He was much surprised at the amount of hydrochloric acid removed from the gaseous mixture, 25 to 30 per cent., confirming the example given on p. 863, second column.

Dr. Hurter had given a most valuable contribution to forming a theory of condensation of hydrochloric acid, and it seemed to him that he had not left much for anybody else to add. From what Dr. Hurter said on pp. 865, 866, the technological books treating of heat required corrections from the extraordinary differences that were to be found between the rates of conduction of stone stated by certain authors and that proved to have taken place by Dr. Hurter in his stone condensers.

Mr. HIGGINS said that, in accordance with Dr. Hurter's request, he had done his best to pick a few holes in the paper. In the second paragraph, on p. 864, he thought the amount of steam condensed,  $V_0r_0 - V_1r_1$ , was wrongly stated.

Again, in calculating the radiation from the condenser, its temperature was taken at 80° C., which was the temperature of the acid at the bottom. He should like to ask whether that represented the average temperature of the whole condenser. On reference to Table I., it would be seen that, when the acid reached 12° Tw., it would be about 80° C., but the question was how far down the condenser the acid had to go before it became 12° Tw.

Besides Dr. Hurter's question "What is the strongest acid that can be produced from a gas of a given composition?" there was another to be solved by the manufacturer, "What is the strongest acid that can be produced from a gas varying much in composition, at one time nearly pure, at another nearly free, at one time dry, at another drenched with steam?"

Dr. HURTER, in reply, said he was very much pleased with the attention which had been paid to his paper by various readers at home and abroad. He thanked Mr. Fletcher and Mr. Carpenter for their kind remarks and criticisms. Mr. Fletcher had said that the paper might have dealt somewhat with the difficulties of complete condensation, but that was not within the scope of the particular paper, which was written entirely from the manufacturer's point of view. He felt certain that there was no one so competent to write a paper on the difficulties of complete condensation as Mr. Fletcher, who had not only a much longer but also a very much wider experience.

With regard to the errors which Mr. Carpenter had pointed out, the first example was one transferred from his note-book to this paper without revision, and referred to 36° Tw. acid. It did not agree quite with his tables, because it was worked out before he possessed these tables. The error was of no great consequence. If the tower had made 30° Tw. acid instead of 36° Tw., the example would be altered as follows:—

For condensation of steam .....	41,808
" " 501 lb. acid .....	205,452
Total .....	247,260

and the pipes would have done 54.6 per cent. of the work instead of 56 per cent., as stated in the paper.

As regards the second error, p. 864, the value of  $x_0$  was incorrectly stated; instead of 0.151 it was given as 0.206, but the example was correctly calculated with 0.151.

In reply to Mr. Higgins, the amount of steam condensed was correctly stated on p. 864, and was  $V_0r_0 - V_1r_1$ . There was, however, an error of omission in the text. The symbol  $x$  was not correctly explained. He had said, "For, if the volume  $V_0$  of a gaseous mixture of hydrochloric acid, air and steam containing  $x$  volumes of steam," &c., but he ought to have said "containing  $x$  volumes of steam per unit volume of the gas." That would at once explain the formula, and indeed the subsequent examples did supply the omission.

With respect to taking the temperature of the liquid within as the average temperature of the condenser, he had done that after mature consideration. At first he used to average the temperature of the water going in and the acid coming out, but that proceeding gave such enormously high figures for the heat-conducting power of the stonework, that he was driven to the conclusion that the average temperature within the condenser was greater. To the same conclusion he was driven by the fact, that although the gases passed through cold water at the top of the condenser, they came out hot. In the first example he gave of a stone condenser the gases came out at 60° C. It was clear from that that a great deal of the condensation is effected only at the very top of the tower, and when considering Table I. it would be seen that water of 15° C. could be heated to 60° C. by forming acid of only 7° Tw. With some form of spray condensers the temperature of the gases was frequently greater on leaving the condenser than on entering. All these considerations led him to the conclusion that rapid condensation occurred at the top, and that the condensation in the lower portions tended to keep up and increase that temperature, and it was more probable that the average temperature of the condenser was higher than the temperature of the acid flowing out.

With regard to the varying conditions occurring in the works, he did not think he could be expected to give formulæ which would deal exactly with every variation. The figures in his examples were all averages over one or two charges. There was one more blunder in the examples which needed explanation. The example on p. 866, second column, was also one from actual practice, and was wrongly copied. The difference of temperature between air and condenser was stated as  $65 - 15 = 50$ . The real difference was obviously 50°, but the example was worked out with 40°, so that the amount of hydrochloric acid of 30° Tw. should be certainly obtained in practice. The use of 40 instead of 50 was simply the application of a factor of safety.

He said in his paper that he thought it would be difficult to test the formula by small scale experiments, but since then it struck him that it could be tested by passing air at different temperatures through acids of various concentration at various temperatures, and thus to obtain some verification. He had made two such experiments, of which he appended the results.

*Experiment 1.*—Air in fine bubbles was passed through liquid acid containing per gramme of water 0.419 grm. HCl, at a temperature of 55° C. and at 765 mm. barometric pressure. (Acid = 29.5° Tw.)

The temperature of the gas was kept constant by a water-bath, and the gas at that temperature brought into

contact with concentrated sulphuric acid to absorb the steam. The HCl gas was then absorbed in water, and estimated by titration. The increase of weight of the sulphuric acid, after deducting from it the HCl absorbed by the sulphuric acid, gave the water carried over.

The results of the analysis were:—

	By Formula.
Air.....	82.76
HCl.....	10.67
H <sub>2</sub> O.....	6.52

2.—A similar experiment, made with acid of 25 Tw., 0.333 grm. HCl to 1 grm. of water at 83° C. and 751 mm. barometric pressure, gave—

	By Formula.
Air.....	60.97
HCl.....	11.42
Steam.....	27.61

It would be seen that the greatest error lay in the steam, not in the hydrochloric acid; a slight error in temperature would seriously affect the amount of steam.

The calculated results were obtained from the formula—

$$P = \sqrt{\frac{0.15}{0.3040 - 0.0016 t}}$$

putting for C 0.419, and for t, the temperature, 55, they calculated the partial pressure of the hydrochloric acid to be 82.8 mm. and from Table II. the tension of steam was found to be 63 mm. The barometer being 765 mm., the partial pressures are—

	Per Cent.
Air.....	605.2 or 80.95
HCl.....	82.8 or 10.82
Steam.....	63 or 8.23

And a similar calculation would give the results as shown for the second experiment.

He wished, in conclusion, to remind them that whatever merit there might be in adapting Roscoe and Dittmar's results to the wants of the manufacturer, the great credit belonged to those investigators for supplying the accurate facts needed for the adaptation.



Meeting held on Wednesday February 5th, 1890.

DR. F. HURTER IN THE CHAIR.

## A NEW FORM OF SPRENGEL PUMP, AND APPARATUS IN CONNEXION THEREWITH.

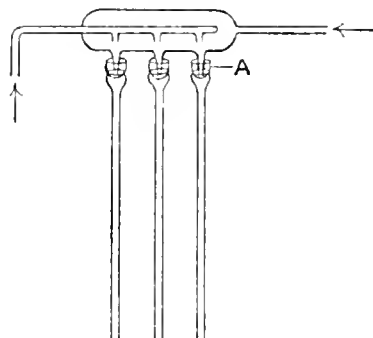
BY SIDNEY G. RAWSON, D.S.C., F.I.C.

*Demonstrator of Chemistry, University College, Liverpool.*

ANYONE who has used an ordinary Sprengel pump will know that the gas from the vessel which is to be exhausted is removed by small pistons of mercury. These rapidly succeed one another in the barometric fall tubes, and portions of the gas are trapped, and carried away between each two of these pistons. When the exhaustion is approaching completion, the volumes of gas so removed become exceedingly small, and the last portion of the process is then one of increasing slowness. The newer forms of pumps work on another principle. The mercury in them is caused to issue in the form of a fine jet or spray, and the molecules of the gas are, as it were, driven forward and caught by the mercurial bombardment to which they are subjected. They are then trapped by the pistons of mercury which form in the ordinary way lower down in the fall tubes.

This form of pump is divisible into two main kinds, viz., the one in which the jets of mercury issue in a horizontal plane as figured in Gillingham's paper (this Journal, III., 83), and the other in which the mercury flows from the commencement in a vertical direction, as in Fig. 1.

Fig. 1.

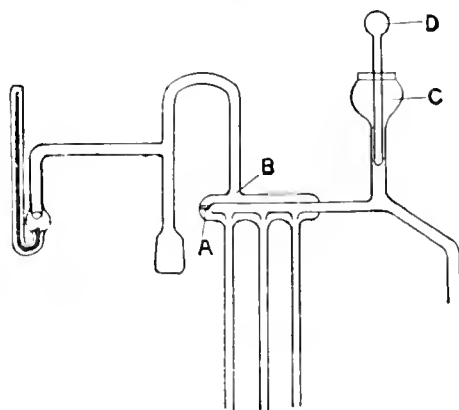


Of these two modifications, I prefer the latter, and for these reasons:—If the mercury issues horizontally, it is somewhat difficult to so place the jets that the streams of mercury shall exactly impinge on the mouths of the tubes. Unless the pump is well made, they will either strike too high or too low, and further, unless there is a considerable pressure of mercury, the jets may not carry even so far as the bounding walls of the exhaust chamber, then the lowest tube receives an excess of mercury, and becomes choked.

In the second form of pump, if the fall tube is slightly opened at its origin, the mercury can only run down that fall tube opposite to its own supply jet. This latter form is also more readily made, and especially is this the case as the number of fall tubes increases. The peculiar mercury distributor of this pump we owe to Nicol, though it had been used by myself some time before the publication of his paper in the British Association Reports for 1887.

My own form of this pump differs somewhat from Nicol's in the following respects. I seal the fall tubes to the pump

Fig. 2.



head, and use no india-rubber as a connexion (see Fig. 1, A), thus doing away with all chance of leakage. The mercurial supply tube, within the exhaust chamber in his pump, ends freely, and in the same horizontal plane with it is the exhaust tube. This, to my mind, would impose rather a strain on the tube from the leverage of the mercury, and I have therefore always sealed the tube to the wall (Fig. 2, A), and so placed my exhaust tube B that it opens in from above. The fall tubes are also sufficiently long to enable me to collect the gases which are exhausted. All these are, however, minor differences.

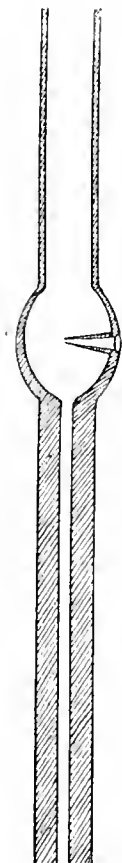
It will be found that if a pump has been working for some time the fall tubes always become coated inside with a thin

film, possibly of oxides of foreign metals. The mercury which I use I have had distilled two or three times, and one would feel inclined, therefore, to say that it was quite pure. But even with this pure mercury the deposit, though it formed very slowly, always made its appearance in the end. Small air bells or bubbles cling most obstinately to this film, and hence it becomes a matter of some importance to be able to remove it easily, and without stopping the pump. For this purpose the small funnel C, is attached to the pump. Usually this is kept filled with mercury, but when necessary, a little sulphuric acid is poured on the mercury, and the plug D, which is ground in, is gently raised, and the mercury, together with a portion of the acid, is allowed to run into the distributor, and thence into the fall tubes. The plug is replaced, and a little more mercury poured in. I have found sulphuric acid very good for this purpose. Professor Ramsay recommends the use of the vapour of glacial acetic acid as a cleansing agent.

This pump is a very excellent one and of great power. If only three fall tubes are required their free ends can be easily bent up so that they shall all deliver their gas together; this becomes more difficult as the number increases, but if mere exhaustion be required other tubes can be added with ease.

While experimenting with this pump I devised the very simple form which is shown in Fig. 3. A piece of ordinary pump-tubing some 50 ins. in length is taken, and about

Fig. 3.



10 ins. from the one end a small bulb is blown. There is then sealed into this and projecting into the cavity a small glass jet, about a quarter of an inch in length and of exceedingly fine bore, with the nozzle pointing inwards. It will be noticed that whilst in the other forms the feed-jets of mercury are detached from the fall tubes, here each fall tube carries its own jet, the arrangement being very compact.

The rate at which the feeding-jet will supply mercury entirely depends on the depth of the mercury in the exhaust chamber, and therefore on the length of the tube lying above the bulb. By regulating this depth the pump can be caused to work more or less rapidly as may be required, and this without any contrivances in the shape of taps, &c. There is further a very thorough division of the mercury in the little trapping bulb, for the fine jet of mercury, which is under considerable pressure, impinges strongly on the opposite wall, and is there broken up, filling the bulb with a shower of mercury drops. Possibly this effect might be increased if two feed-jets were sealed into the bulb. I have found it more advantageous to replace the piece of pump-tubing above the bulb by some of considerably wider bore and thinner walls; the difficulties of exhaustion are increased if the molecules of gas have to travel along capillary tubes.

Though two or three fall-tubes of this description can be sealed into a glass exhaust chamber, it is a somewhat difficult task. Unless an extreme amount of accuracy in the volume of gas to be collected is required, it is quite sufficient to take a piece of wide glass tubing which is strangled at either end so as to firmly hold the rubber corks with which it is to be closed. Through the lower cork passes the pump and fall tube combined, and through the upper the mercury feed-supply and the exhaust tube. From some experiments which I have made I agree with Gunningham (loc. cit.) that the diameter of bore of the fall tubes should be about 1.6 mm., and the length about 1 metre, reckoning from the level of the mercury in the lower reservoir to the mercury feeding-jet. In longer tubes than this the mercury falls with much force, and the incessant hammering which goes on is apt to cause the tubes to crack; several have been thus rendered useless in my hands.

In working with exhaust pumps there is usually some difficulty in making an air-tight joint between the pump and the apparatus which is to be exhausted. Of course the most effectual and certain way is to unite the two ends by an actual glass seal, but in many cases this is by no means easy, and when made successfully it has the disadvantage of converting all the different portions into one solid rigid whole. Professor Crookes uses very thin spiral glass tubes to unite the various pieces, and these from their elasticity do allow of there being a certain amount of play, but the glass seal, and with it considerable experimental skill, is still required.

India-rubber tubing is of very little or no use. All sorts of cements have been tried for this purpose. Thus Professor S. Thompson\* mentions a mixture of Burgundy pitch, 96 parts, with 4 parts of gutta-percha or 1 part of vaseline with 3 parts of paraffin wax. Others are equal parts of beeswax and resin, india-rubber, and many more. I have myself used beeswax and resin containing a little vaseline, but I cannot recommend it highly, though it is as good as any of the others. Paraffin wax, pure and simple, I have also tried and found to work well, but it contracts on solidification, and attaches itself more especially to the glass walls, leaving a fissure between when these are close together, and hence there is always the chance that air may get in. No organic compound can be said to be good, as at high *vacua* they all evolve vapour. As suggested by Professor Thompson in his paper, some experiments were made by me on fused chloride of lead. This only melts, however, at a high temperature, somewhat below a red heat, and at the same time emits fumes. On cooling it contracts considerably and cracks in all directions.

After trying many materials, the following is the cement I now employ, and which is of very easy application. It is nothing but fusible metal containing a considerable amount of mercury. Its percentage composition is as follows:—

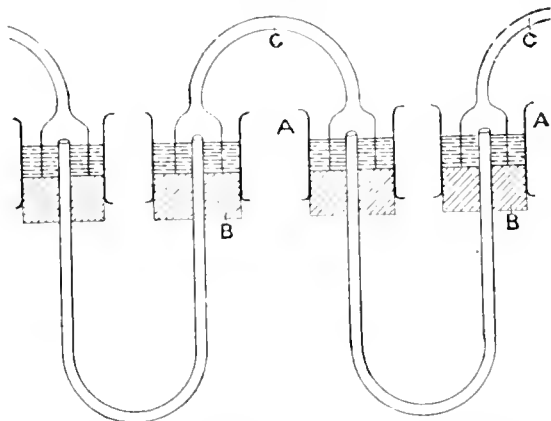
	Per Cent.
Bi .....	40
Pb .....	25
Sn .....	10
Cd .....	10
Hg .....	15

\* "The Development of the Mercurial Air Pump." *Journal of the Society of Arts*, 1888. From this paper Fig. 1 is copied.

If ordinary fusible metal be taken it will crack the glass, from the large amount of expansion which it undergoes on solidifying. The addition of a little mercury reduces its expansibility and at the same time lowers the melting point. As it still, however, expands considerably on cooling, it forces itself into every minute crevice, acting as a most efficient cement. It should be applied in a molten condition, and the glass should also be hot, and, before exhausting, the joint should stand for an hour to allow the alloy to become thoroughly set. Tubes cemented to the pump by this compound I have had exhausted for weeks without any sign of leakage. I wish here to express my thanks to Mr. H. S. Marsh for the trouble which he has taken in experimenting with me on these alloys.

I also employ a special form of joint. It is shown in Fig. 4 as in use between two U-tubes which have been

Fig. 4.

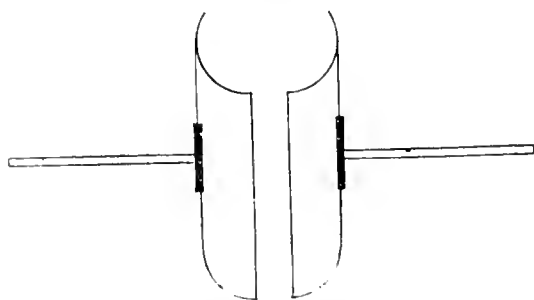


taken for drying the gases which are to be collected. Each limb bears a small glass cup A carried on a cork B, through which passes one end of the U-tube. A curved tube C, bell-mouthed at either end, fits in each case outside the free end of the tube but within the glass cup. The molten alloy is poured in almost to the level of the mouth of the U-tube, and on cooling grips the whole firmly together. Until the alloy is poured in there is any amount of play, as can easily be seen, between the different parts of the apparatus, for the bell tubes need not be placed in position until everything else has been finally adjusted.

Moreover, instead of there being a long stretch of U-tubes or other apparatus, these can be ranged in rows of twos or threes, &c., one behind the other, by placing the proper bell-tubes at right angles to the others in that row, and thus making connexion with those coming both before and after.

The apparatus can be readily disconnected by placing round each of the glass cups in turn a brass cup riveted to a rod of the same metal. When the rod is warmed

Fig. 5.

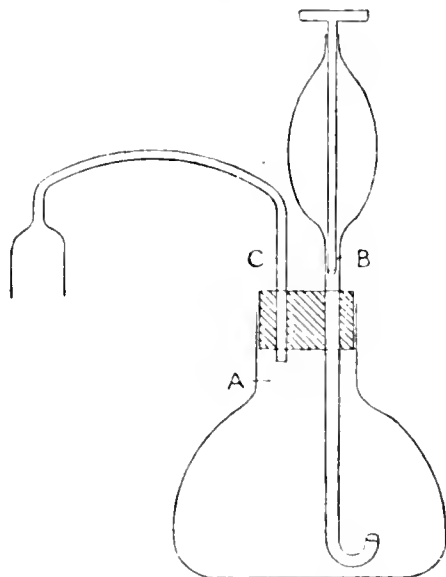


the brass cup rapidly becomes heated and the alloy, as it melts at such a low temperature, is easily liquefied, and the bell-tube can then be lifted out. It will be found more

convenient to have the brass cup in two halves attached to two brass rods as in Fig. 5; it is then more easy to remove it, otherwise, as it must fit close to the glass cup, it will be necessary to wait until the brass cup is cool before it can be bent open.

In some experiments on which I am engaged I have found it necessary to use a particularly formed flask for the preparation of certain gases. A short description of this may prove interesting.

Fig. 6.



It holds about 200 cc. and fitting to the neck and ground to it with the *greatest care* is a glass hollow stopper A perforated in two places. For cement I use a mixture of beeswax, resin, and a little vaseline. Through one perforation B in the stopper passes an inlet tube for acid, terminating within the flask in a tube bent up slightly on itself. Outside the flask it bears a glass bulb into which acid can be poured; the bulb is choked by a ground glass rod, cut into which is a slot, corresponding to one in the bulb. When the slots coincide the acid passes down, but by turning the glass rod through 180° the connexion is cut off. A little acid should always be left in the bulb. Through the other perforation C passes the exit tube, terminating in a bell mouth.

The whole apparatus, from pump to generating flask, it will be observed, is without a single tap of any kind, is air-tight, and yet is readily put together and disconnected, and requires, except in the case of the glass flask, no special skill in its manufacture.

#### DISCUSSION.

Dr. C. A. Koux said he had worked with Dr. Rawson's pump, and could testify to the value of the alloy, which gave very satisfactory results. He would like to ask Dr. Rawson what the efficiency of his simpler form of pump was, as compared with the ordinary forms of Sprengel pump.

Dr. HURTER said that in many of his own investigations he had had many of the difficulties to contend with which Dr. Rawson had so ingeniously overcome. The nuisance of corks and taps in researches where every trace of a gas had to be kept together was very great indeed. He recollected that a few years ago, when he was busy with the investigations of an ammonia-soda process, he had to avoid loss of every trace of ammonia which was evolved at a red heat. He could only do this by sealing the whole apparatus together into one piece, and when he had very nearly finished the apparatus he broke it and had to start again. Fusible metal he had also tried, and to his great grief it broke every

glass he put it into, and he was glad to learn that the addition of a little mercury would correct that property of expansion on cooling just sufficient to make a joint without breaking the glass.

Dr. Rawson said, in reply to Dr. Kohn, that as regarded the working of his new pump, he had tried it under various conditions, and had found that the exhaustion to be obtained with it was quite as good as that with any other pump with which he was acquainted. Fusible metal and glass tubes, though remaining intact for a time, were invariably broken sooner or later.

## HOME-GROWN SUGAR FOR GREAT BRITAIN AND IRELAND.

BY G. SCHACK-SOMMER, PH.D.

The agricultural question is undoubtedly one of the most pressing and vital importance both in England and Ireland. In which of these two countries it is of most immediate moment it is not necessary for my present purpose to inquire; and to go into the causes that have led to this state of things is equally beyond my province or intention. I would merely mention, in passing, that the British Government have thought it advisable to establish a Board of Agriculture, and to appoint Mr. Chaplin as its first president; sufficient proof, if any was needed, that the agricultural interests of the United Kingdom are of importance enough, even in their condition of decadence, to require the attention of a special department. To that special department and to you, members of the Society of Chemical Industry, I have something to say, which I think is worthy of the serious consideration of both.

I ask you why cannot we grow beetroot, and manufacture sugar from it, as successfully as they do in France, Germany, Austria, Russia, Holland, Belgium, and many other countries?

With the fiscal difficulties in the way of doing this I am not at present concerned. My attention has been concentrated principally upon what are supposed to be the natural difficulties of growing sugar beetroot in Ireland or England for the purpose of manufacture, and in this way creating a new industry for both countries. The popular impression has hitherto been that the climate of these islands is too cold and damp for the successful cultivation of sugar beetroot, but I have no hesitation in saying that practical experience has proved the popular impression in question to be entirely wrong and mistaken.

In 1879 a Select Committee of the House of Commons was appointed to inquire into the effect produced upon the home and colonial sugar industries of this country by what may be called, for the sake of brevity, the bounty system in those foreign countries which manufacture sugar for exportation. In the course of his evidence before the Committee, Mr. James Duncan was asked if he could give any explanation of the fact that the growth of beetroot was practically confined to the Continent; and if no part of this country was adapted to the growth of beetroot. Mr. Duncan's reply was that it was adapted, but that the disadvantage of growing was owing to the bounty system, foreign sugar having a bounty which acted as a preventative to the growth of sugar in England, and which might be taken to be in the average 2*l.* a ton. In answer to further questions, Mr. Duncan stated that the bounty rendered it practically impossible to grow any beetroot in this country, though it could be done in the east of England of quite as good quality, if not better, than in France; but that, in face of the bounty, it was impossible.

Mr. George Martineau, giving evidence before the same Committee, said that he had given a very considerable amount of attention to the subject of the growth of beetroot in this country. One of the questions asked Mr. Martineau was as to the relative richness of roots grown in England as compared with foreign countries. In answer to this it was stated that Dr. Voelcker, the chemist to the

Royal Agricultural Society, found 12 per cent. of sugar in roots from Suffolk; 12½ and 13 per cent. in roots from Berkshire; 11½ per cent. in roots from Surrey; 10½ to 13¼ per cent. in roots from Yorkshire; and 10, 12, and 13, up to 15 per cent. in roots from Kilkenny. That was in 1870. Since then Mr. Martineau himself had taken a considerable amount of trouble in the matter, and had roots grown both in Lincolnshire and the Isle of Thanet; and as far as the latter were concerned had in his own laboratory extracted 15 per cent. of sugar; an amount of richness in which England, in his opinion, could certainly compete with France, where the average was not more than 10½ per cent.

My object, however, in reading this paper is not to repeat evidence given 10 years ago before a Select Parliamentary Committee, but to bring before you the results of my own experiments made last year on the farms of Mr. John Ennis, Springwood, Ballymitty, county Wexford, Ireland, and Mr. John Gibbons, Dunbabin farm, Wavertree, near Liverpool. For the purpose of these experiments four different kinds of beetroot seed were sent to me by Dr. C. Scheibler, the eminent sugar chemist of Berlin, and to all three gentlemen I herewith tender my best thanks for their kind co-operation. Before proceeding to give the results of my experiments I may mention that in Germany and Austria the roots have been grown for years under an artificial system which causes them to be produced of an excessive richness.

### YIELD PER CENT. OF WEIGHT OF BEETS.

	Per Cent.
1850-51 .....	6'66
1860-61 .....	8'62
1870-71 .....	8'61
1871-72 .....	8'28
1872-73 .....	8'25
1873-74 .....	8'25
1874-75 .....	9'30
1875-76 .....	8'60
1876-77 .....	8'15
1877-78 .....	9'24
1878-79 .....	9'21
1879-80 .....	8'52
1880-81 .....	8'79
1881-82 .....	9'56
1882-83 .....	9'51
1883-84 .....	10'54
1884-85 .....	13'70
1885-86 .....	11'43
1886-87 .....	11'87
1887-88 .....	13'08
1888-89 .....	11'96

In this respect Germany surpasses other countries, but from the smallness of the size of the roots there are not so many tons produced to the acre. The seeds I obtained from Dr. Scheibler were:—

1. Dippe's Zuckerreichste Elitezüchtung.
2. Gebrüder Dippe's Zuckerreiche Imperial Elitezüchtung.
3. Gebrüder Dippe's verbesserte Zuckerreiche rothköpfige Elitezüchtung.
4. Gebrüder Dippe's verbesserte Zuckerreiche Kl. Wanzelhohnen Elitezüchtung.

Of course you must not forget that, though I obtained the best seed procurable, both with reference to choice of soil and manure, I had to leave the necessary care of the plant to quite inexperienced hands. Possibly it may be the intention of some members of this Society to make experiments as I have done—experiments which I have already been asked to repeat by several Irish farmers. In such a case a hint or two about soil and manure may not be unacceptable. As to the first point, viz., the soil, it must be noted that very wet or swampy ground is quite unsuited for beet growing. What this requires is a loose clay containing lime, with a well-drained bottom. As to the second point, a very good artificial manure for beetroot is one containing one part of nitrogen to two parts of phosphoric acid as superphosphate. They spend as much as 5*l.* to 6*l.* per acre for artificial manure in Germany. The proportion wanted is about 200

to 300 lb. nitrate and from 400 to 600 lb. superphosphate, or 400 to 500 lb. Peru guano, and 200 to 300 lb. superphosphate per acre. The soil has to be ploughed up deeply—the deeper the better—unless the bottom ground is very different from the top. The usual plough goes from 7 in. to 9 in. deep, but it is necessary to go from 5 in. to 6 in. deeper. Of course my friends, the farmers in Ireland, and at Wavertree, near Liverpool, did not take these precautions, nor did they take any care to thin the plants as they grew up. The results obtained, therefore, are all the more surprising, as they show beyond the shadow of a doubt that beetroots can be grown in this country as rich in sugar as even in Germany,

where the richest are produced under an artificial system of duty.

Comparison with German roots was possible through the circumstance that Mr. F. O. Licht, of Magdeburg, publishes during the summer months a *resumé* of the results obtained by analysis in his laboratory of a quantity of freshly drawn beetroots. The day that he did this was always known to me, and on the same day, on several occasions, I had beetroots drawn from the earth both in Ireland and Wavertree, and had them analysed in my laboratory. You will find in the following tables the results of these experiments, which are positively surprising.

Date of Experiment.....	F.O. Licht.	Wavertree.				Ireland.			
	26th Sept.	26th September.				26th September.			
	—	I.	II.	III.	IV.	I.	II.	III.	IV.
Number of beetroots drawn for experiment	88	10	10	10	10	10	10	10	10
Average weight with leaves, in grammes ...	886	948	946	1,052	1,249	676	762	792	1,016
Average weight without leaves, in grammes	503	358	342	373	539	309	305	368	395
Largest root with leaves, in grammes.....	1,650	1,400	1,285	1,505	2,075	920	965	1,430	1,330
Largest root without leaves, in grammes ...	1,144	401	405	635	624	250	330	690	490
Smallest root with leaves, in grammes .....	182	695	621	640	710	490	625	595	720
Smallest root without leaves, in grammes ...	275	310	135	216	236	190	275	265	295
Specific gravity of juice.....	1·0781	1·0716	1·0741	1·0744	1·0677	1·0787	1·0828	1·0902	1·0814
Degrees by Brix saccharometer.....	18·8	..	..	..	..	..	..	..	..
Quantity of sugar in 100 parts.....	16·22	15·0	15·6	14·7	14·2	16·7	17·0	18·2	16·8
Quantity of non-sugar in 100 parts .....	2·58	..	..	..	..	..	..	..	..
Quotient in 100 parts .....	8·62	..	..	..	..	..	..	..	..
Per cent. shown by Fehling's solution.....	..	·5	·4	·4	·4	·5	·5	·6	·5

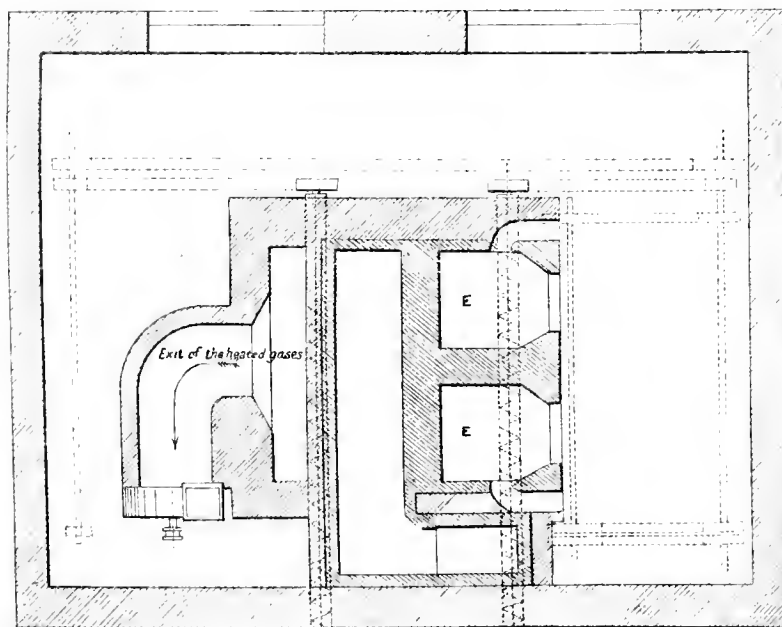
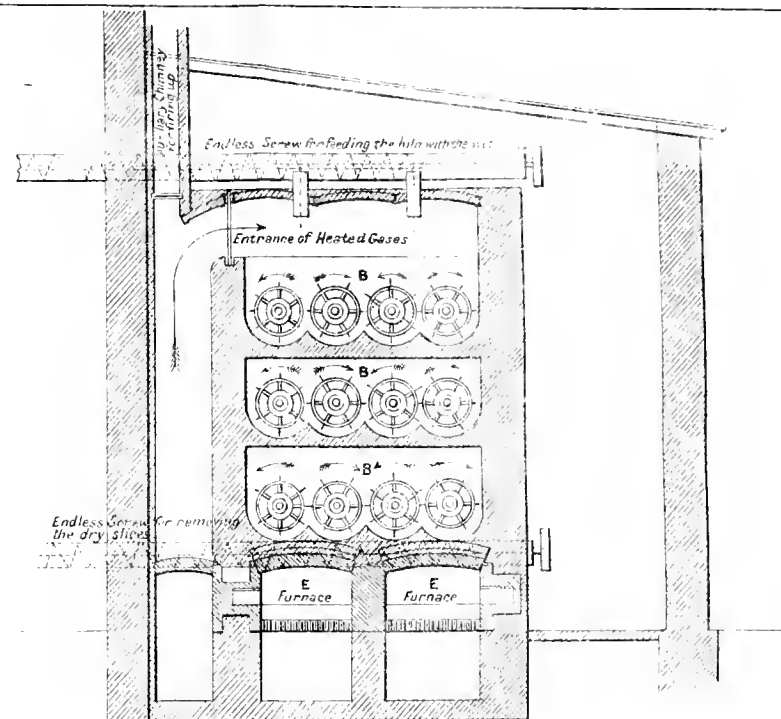
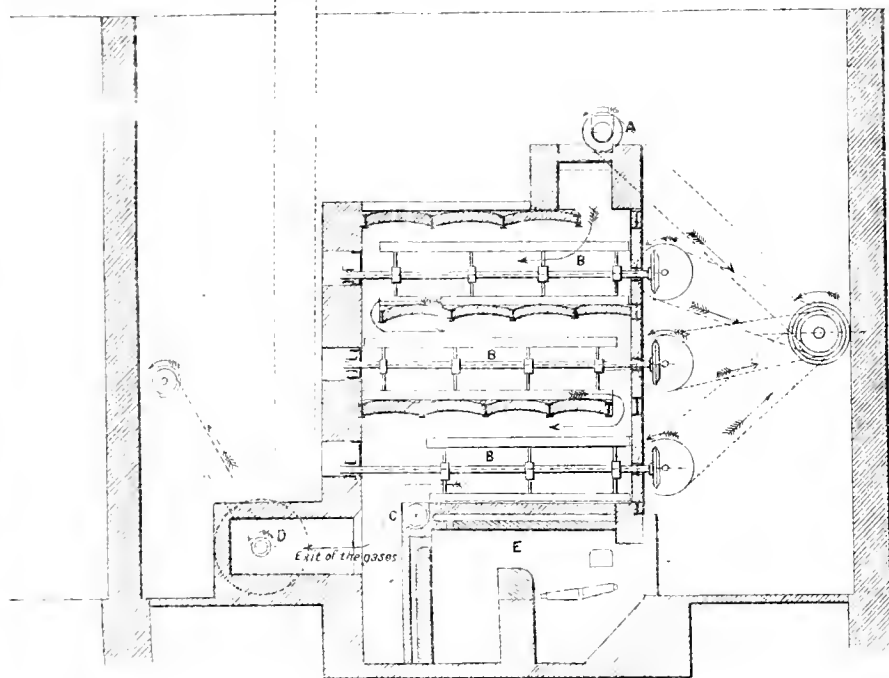
Date of Experiment .....	F.O. Licht.	Wavertree.				Ireland.			
	10th Oct.	10th October.				10th October.			
	—	I.	II.	III.	IV.	I.	II.	III.	IV.
Number of beetroots drawn for experiment	112	10	10	10	10	10	10	10	10
Average weight with leaves, in grammes ...	900	675	958	967	1,268	1,079	1,118	1,177	1,077
Average weight without leaves, in grammes	540	246	364	404	429	506	469	460	479
Largest root with leaves, in grammes.....	1,670	800	1,260	1,330	2,215	1,440	2,040	1,500	1,650
Largest root without leaves, in grammes ...	1,205	370	340	340	570	660	720	570	710
Smallest root with leaves, in grammes.....	490	400	590	510	770	780	820	780	740
Smallest root without leaves, in grammes...	295	130	255	200	310	380	320	310	330
Specific gravity of juice .....	1·0806	1·0799	1·0799	1·0784	1·0793	1·0867	1·0838	1·0821	1·0888
Degrees by Brix saccharometer.....	19·4	18·7	18·7	18·3	18·4	19·9	19·8	19·6	19·9
Quantity of sugar in 100 parts.....	16·38	16·5	17·0	16·7	16·4	18·2	17·5	17·2	18·8
Quantity of non-sugar in 100 parts .....	2·82	2·2	1·7	1·6	2·0	1·7	2·3	2·4	1·1
Quotient in 100 parts .....	85·5	88·2	90·9	91·2	89·1	91·5	88·4	87·8	94·4
Per cent. shown by Fehling's solution .....	..	·3	·4	·5	·4	·5	·7	·5	·6

1 kilogramme (1000 grammes) equals about 2 lb. English weight.

I will now explain to you how I arrived at these results. The beetroots were drawn carefully out of the earth and brought to me without the loss of a single leaf. I then

washed and dried them quickly and weighed them with the leaves. This gave the first result. Then I cut the top and the root and weighed again, imitating the German manu-





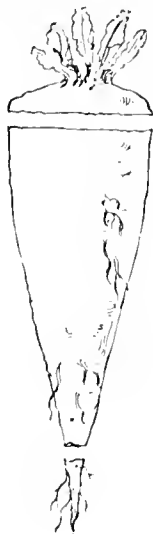
## PLANT FOR DRYING BEETROOT SLICES

- A Endless Screw for feeding the Kiln with wet slices
- B Agitator for moving and turning the slices
- C Endless Screw for removing the dry slices
- D Air propellor to draw the Gases from the Furnace through the Kiln
- E Furnaces

The long arrows show the direction of the slices & of the gases.



facturers as they prepare their roots to go over the Custom house scales, having found by experience that the top and the root contain very little sugar. I imagine that Mr. F. O.



Licht gets at his figures for the weight of roots without leaves in the same way, as when I communicated with him on this point he found no fault with my mode of proceeding. The third operation was to get the juice. This has to be a very quick process, as chemical changes begin almost immediately, and therefore all tests must be applied as soon as the juice is squeezed out of the roots. I had, therefore, prepared four small boxes, each 1½ ft. long by 1 ft. broad, with punctured tops, resembling a large-sized bread-grater. Before each of these a boy knelt, grating one root at once; and when the full quantity had passed through, I pressed the mass in cloth of a very close-woven material in a filter-press, and the juice came out almost entirely clear, though dark in colour. As it is generally found that there is a great difference in strength of saccharine matter between the first and last juice coming out of the filter-press, I was very careful to press the cakes quite dry. The juice was then treated with sugar of lead and tannin, to clarify it, and then polarised. Another portion was gauged with Brix saccharometer, and some filtered through filter-paper before taking the specific gravity by weight. The results are all those of analysis of the juice, no allowance being made for the pulp, which is generally from 4 to 5 per cent. of the weight of the beetroot. I tested two lots of roots on the 17th October. No. 1 was 4.36 per cent., and No. 2, 4.41 per cent. of fibre. This result was obtained by extracting a weighed quantity of grated roots, at first with cold and later with boiling water, and drawing off both with a Scheibler felt filter and suction pump, and then drying the fibre in a tared filter. This fibre is of the greatest importance to farmers who grow the root for manufacturers, for, just as oilcake is a valuable by-product of seed-crushing, so there is not a better food for cattle than the beetroot fibre, which has from 10 to 12 per cent. of solids, which is as much as the beet generally used for feeding purposes, and it is usually mixed with hay, chaff, or maize. As food for cattle, therefore, beetroot is quite as valuable after the juice has been extracted as before.

As the sugar has, in latter years, been extracted from the beetroot by the diffusion process in many places, the residue would afterwards contain 11.99 per cent. of dry substance, with 4.84 per cent. of ash, which would fill and overload the stomachs of the cattle.

A great improvement has been achieved by drying the beetroot slices by a patent drying kiln, the construction of which will be easily understood by the accompanying drawing. This kiln is the result of a prize competition, and the inventors are Dr. Meyer and Mr. Buettner, and the apparatus has been constructed by Messrs. Langen and Hundhausen, in Grevenbroich, near Cologne.

With this apparatus they are able to dry 225,000 kilos. of beetroot per day, at a cost of 7.63 pf. per 100 kilos., or equal to almost 4d. per 100 lb. This dried residue contains 94.20 per cent. dried substance which gives 8.95 per cent. ash.

7½ to 8 ctr. beetroot slices dried this way give 1 ctr., and in spite of a certain taste of smoke and sulphur acquired from the fuel gases, and a small addition of lime, the cattle have taken kindly to the food, and, what is rather remarkable, do not seem to have required a larger quantity than usual. The advantages of this invention are, first, a healthy cattle food, and secondly, a saving in the cost of transit. The food can also be easily kept.

Another point to which I would draw your attention is the proportion of non-saccharine matter in the table of comparative results already given. This is simply the difference between the quantity indicated by the Brix saccharometer, which is supposed to show what quantity of sugar there is in the liquid, and the quantity shown by the polariscope which is real sugar. In the beetroots I examined I found a reaction by Fehling's solution, and I wrote to several experts on the Continent to ask them how they accounted for it. One of the replies, from Dr. O. Follenius, was to the effect that he thought the precipitate must have been yellow, and not red, and that would be caused by pectin, which is found in nearly every juice. Director C. Krüger, however, writes that he finds invert sugar in beetroots either grown in poor soils or not properly attended to. That may well have been so in my own case, as I have already told you the soil had not been specially prepared. The result to be arrived at from a consideration of all the circumstances is, really to discover how much per cent. of pure sugar it is possible to extract from 100 parts of the dry substance of the juice, and the higher this percentage is, of course, the better the beetroot. To calculate this, the saccharometer of Brix is used to show the dry substance of the juice, and then a calculation is made as to how much is sugar. Take, for instance, a juice that has 12 per cent. of sugar and shows 15 per cent. on the saccharometer. The result is 80 per cent. of sugar to 100 parts of dry substance. The results obtained by Dr. Follenius vary from 75.2 to 81.4 and 86.5 per cent. In my table you will find I got as much as 94.4 per cent.

As to the quantity I got per acre in my experimental cultivation, I am able to inform you that at Dunbabin farm, Wavertree, near Liverpool, we got 42 tons 19 cwt. per statute acre; and further, that the steward of Mr. Gibbons informed me Nos. 1, 2, and 3 seed gave an average crop, while No. 4 gave a very heavy crop. From my Irish friends I have not yet been able to obtain particulars as to quantity of crop per acre. Some people imagine that beetroot is an exhausting crop. On this point Mr. George Martineau gave very conclusive evidence before the Select Committee already mentioned as having been held in 1879. He quoted Mr. John Algernon Clarke, secretary to the Central Chamber of Agriculture, and Dr. Voelcker, chemist to the Royal Agricultural Society, to the effect that it is not an exhausting crop, and that it is one very favourable to the growth of wheat, the deep cultivation necessary to grow beetroot increasing the production per acre.

As a proof that the soil is improved by the cultivation of beetroot, and the extra tilling and manuring which such cultivation entails, I may mention the case of an estate called Groena, in the Dukedom of Anhalt, which comprises about 700 or 800 acres of fertile land. They did not plant any beetroot before 1837, and then up to 1853 only a few acres for a trial. In 1853, however, they began to plant about 150 acres every year, and in 1856 the results proved conclusively that they had got as much corn as they had formerly done when the whole area of the estate was devoted to its cultivation. On another estate, that of Ossnarsleben, near Bernburg, which comprised about 1,000 acres of fertile soil, they generally had a little more than half of it planted with corn. They found, after introducing the cultivation of beetroot, that they had an increase of about 2 per cent. in the corn crop. In France, in the Arrondissement of Valenciennes, not only was the corn crop increased by the introduction of beetroot, but they were able to feed

11,500 head of cattle instead of 700. The conclusion to which these facts point is obvious. Beetroot, so far from being an exhausting crop, positively enriches the soil if it is planted with a due regard to rotation. That is to say, if some years are allowed to elapse before the planting is repeated, it is not only a paying crop in itself, apart from the question of the value of its fibre as food for cattle, but that it positively increases the fertility of the ground for wheat or other cereal production.

Stable manure is very efficient, but ought not to be used on the land within, say, nine months of planting the beetroot seed. Lime is often of great advantage to the soil, but an addition of potassium salts has not proved remunerative.

The question naturally suggests itself:—How often can beetroot be planted to secure the advantages just mentioned? I have taken particular care to obtain trustworthy information on this point, and find the general opinion of those best informed to be once in six to eight years. For my own part, and in this I only express a personal opinion, I think that with the help of the advanced chemical knowledge of the present day, and by use of artificial manures, the period between the beetroot crops might be considerably shortened. The cause of the so-called "beetroot idleness" of the soil is a too frequent planting of beetroot on the same soil; this, however, has been attributed, not so much to the disappearance of the ingredients of the soil necessary to the beetroot, as to the enormous increase of "nematoids," small insects which are like diminutive eels, which, when young, are very agile, and penetrate the young roots, where they gradually thicken to such an extent that their moving power is lost. It is not alone the beetroot that these insects attack, but all cereals, especially oats, also cabbage, and many other vegetables. Their destruction occupies the minds of the agriculturists to such an extent that they have actually founded a special experimental station for this sole purpose. It seems that the most successful way of getting rid of them is to plant catch-plants, that is, such as the nematoids prefer, and then destroy the plants with the insects. A fungus—*Arthrobotrys oligospora*—which was found by Professor Zopf to destroy the nematoids has, however, only a chance where a good deal of water is present to sustain its existence.

I have seen it stated that 120 nematoids have been found in a small beetroot, and also that five times as much beetroot is grown on soil free from nematoids as on that infested with that pest. What I take to be the proper rotation of crops may be found in a pamphlet by Dr. C. J. Eisbein, who is the Professor of Agriculture at Heddesdorf, near Neuwed. This is as follows:—First year, beetroot fertilised with stable manure; second year, oats or barley; third year, clover and grass; fourth year, wheat; fifth year, peas, beans, or other leguminous plants or potatoes, fertilised with stable manure; and sixth year, rye. An alternative succession would be: First year, beetroot fertilised with stable manure; second year, barley or summer wheat; third year, clover or grass; fourth year, wheat; fifth year, potatoes; sixth year, oats; seventh year, leguminous plants, such as peas, beans, &c., fertilised with stable manure; and eighth year, rye. Beetroot may be planted again in the fifth year, but as to the advisability of doing this I am not in a position to say anything positive. As to the cost of production in relation to the profit, I will not trouble you with figures, but will merely remark that the profit is a little more than one-third of the outlay.\* No other crop, in fact, brings such a profit to the farmers on the Continent, except where they can plant tobacco or linseed.

The points to be borne in mind in connexion with what has been already said may be thus summarised:—

1. That sugar beetroot grows in any soil if it is rich in soluble mineral ingredients, and has nothing of pipe-clay or sand in its composition.
2. That sugar beetroot requires a soil with a deep foundation, and one that is very equal in its layers.
3. That the ground ought to be well drained, or to drain itself well.

4. That water is a necessity, but that water in excess is injurious to beetroot cultivation; and

5. That it is impossible to plant beetroot profitably oftener than every fourth year.

So far I have only spoken of the cultivation of beetroot from an agricultural point of view; but I have no doubt it will be equally interesting to those who regard the question as a purely financial one. On this point, I may be permitted to quote from a publication, which is, no doubt, known to most of you, viz.: "The Sugar Cane." In the issue for the last month I find the following:—

"Since the end of October the following results of German and other Continental sugar factories for the 1888-89 campaign have been announced. These are obtained, as usual, mainly from the Deutsche Zucker-industrie:—

#### Dividends Declared.

"Immendorf, 45 per cent.; Heilbroun, 15 per cent.; Bennigsen, 11 per cent., besides dividend to beet contractors.

#### Net Profits Shown.

Name of Place.	Capital.	Profits.	
	Marks.	Marks.	Per Cent.
Brühl .....	1,050,000	254,750	24
Neuwerk .....	1,500,000	162,514	11
Königsutter .....	240,000	191,063	81
Niederhone .....	460,000	124,573	27
Olfstein .....	621,000	77,162	12½
Tuezo .....	591,000	46,371	8

I have nothing more to add except to point out the immense advantages that would accrue to agriculturists both in England and Ireland from the cultivation of beetroot as a rotation crop. The evidence given before the Select Parliamentary Committee in 1879, especially by Mr. George Martineau, points clearly to the conclusion that but for the existence of foreign bounties beetroot for the manufacture of sugar might be profitably grown both in England and Ireland; profitably not only in the certainty of producing a valuable crop, but also in enriching and fertilising the soil, and at the same time providing the cheapest and most useful form of food for cattle. These points, let me remind you, are not mere matters of individual opinion, either of mine or anybody else. They are facts, the truth of which, as I have endeavoured to show, I have proved by my own experiments and experience; facts which have also been demonstrated in an even more conclusive way by the continental examples already quoted. That the cultivation of beetroot would be a good thing for England, and an especially good thing for Ireland, I hope that what has been already said has done something at least to prove.

#### DISCUSSION.

Dr. HURTER said that to anyone acquainted with the agriculture of this country or with the manufacture of sugar this paper would be very interesting. Dr. Schack-Sommer had opened a future both to Ireland and England for an alteration in their agricultural system. It would be of course a question for farmers and landlords to consider how far the material benefits to be derived from the alteration were borne out by Dr. Schack-Sommer's facts, but for them the great thing was to discuss the results of his experiments, and he should be glad to hear the discussion opened.

Mr. J. GIBBONS wished to know if Dr. Schack-Sommer could give them any idea as to the cost of growing a crop of beetroot in this country, and the value which the agriculturist might expect to get from it?

\* See table.

Mr. RHODES said he would like to know what kind of manure was used for growing the beets? He was not quite certain whether it was superphosphate, or nitre, or stable manure. He was under the impression that artificial manures were largely used on the Continent, and in consequence large percentages of sugar were obtained.

Mr. JOHN HAVES said the reader had mentioned that his two fields were not specially prepared, also that the results on the German estate appeared to increase from 1853 in the amount of yield per acre. He should like to know what crops had been previously grown on the fields at Wavertree and in Ireland. He understood that nine months were allowed to elapse before the crop came up. He would like to know at what time of the year these fields were treated. Another point was the using of a special kind of catch-plant to catch injurious insects. Were these catch-plants grown along with the crops, and did they come up together; if so did not they take up some of the food or nutriment from the soil which otherwise would have benefited the beets, or, was there some special way and time for laying down and rooting the catch-plants up, or were they ploughed up with the crops and picked out afterwards?

Dr. J. CAMPBELL BROWN said that Dr. Schack-Sommer's contribution was encouraging to agriculturists in view of the necessity for introducing some change—they knew not what—into the agricultural system which had hitherto prevailed. The difficulty of finding a market for beetroots remained and would remain for some time, but they hoped would disappear with the abolition of bounties. A farmer could not grow beetroot for the manufacture of sugar to be consumed on the premises. He must be in easy reach of the manufacturer on account of the speed at which decomposition sets in, and the manufacturer was not likely to set up his boilers, &c. in the present state of the sugar industry. But it was worth trying to grow beetroot for the feeding of cattle; and he was glad to see from the details that a good deal of the land in this country could be used for the growth of beet without any change in the system of agriculture, with the exception of the change of seed. A good deal of the land in Scotland was already grown on the six-year rotation system. Another question was that of climate. Did the beetroot require a warm genial climate, or would it grow in the raw cold climate of the east coast?

Mr. J. W. MACDONALD said beetroot-growing ought to be successful in this country, if the fiscal conditions on the Continent were not working against them. On looking over some figures about sugar that day he found that during the last four years (or since the 1885-86 crop) the cultivation of the beetroot had increased 57 per cent., whereas the cultivation of cane sugar had remained stationary or decreased 2 or 3 per cent.

Mr. COGHILL asked whether it would be cheaper to make sugar from beetroot grown in this country than from sugar cane imported from abroad?

Mr. BECK asked if it could be ascertained what the real profits to an agriculturist would be. He remembered a crisis some years ago when some special inquiries were made as to the cost of producing beetroot sugar, and he understood that the actual expense of making beet sugar from the root came to about 15s. a ton. They knew the percentage of sugar but they did not know the actual cost of beet sugar. They must not forget that the beet sugar had been grown on virgin soil in England as far as the beetroot was concerned. It was generally understood that the sugar beet took a great deal of power out of the ground. These things should be taken into consideration when comparing results.

Mr. E. CAREY said it was extremely refreshing to get for once out of alkali making into another branch of chemical manufacture. Dr. Schack-Sommer had given them an estimate of the profit that would accrue from the cultivation of beetroot. He (Mr. Carey) did not quite understand whether the profit was on beet grown here or in Germany. He also understood that the Germans had the advantage of a bounty which amounted to about 2*l.* per ton on the sugar. Of course that would handicap home growers very much.

He would be glad if Dr. Schack-Sommer could give them any idea as to what he would estimate the actual profit to be to an English agriculturist, supposing he could get results similar to those obtained at Wavertree and in Ireland?

Dr. HURTER asked what prospects there would be for a farmer finding a market for his beetroot in this country. Would the sugar manufacturers in this neighbourhood or elsewhere be prepared to erect plant for manufacturing sugar from beetroot?

Dr. SCHACK-SOMMER in replying said he should begin by answering the remarks of the last speaker. Of course there could be no prospect of the farmer getting rid of his beetroot until works were erected for the purpose of preparing the beetroot for sugar manufacture.

On the Continent, generally, where beetroot was grown, a certain number of farmers in each district clubbed together and built a factory, each farmer undertaking to deliver to the factory a certain weight of beetroot during the season. The factories were run on this principle, and after the owner's contract quantity was worked up they bought further quantities in the open market if they deemed it advisable, but by their system each factory was secured a minimum quantity at a stipulated price each season. As a rule the several factories were connected each with separate estates, and built in such situations as would afford the best facilities to the growers for delivering their produce.

With respect to the probable profit of beet growing which Mr. Carey and three other gentlemen referred to, Dr. Schack-Sommer said he had prepared the following tabular statement of the cost of planting and working of sugar beetroot, including delivery at the works, viz.: taking English measures and weights, which have been checked and found approximately correct by English farmers.

AMOUNT REALISED.	
The result per acre:—	£ s. d.
1. 33,079 lb. beetroot at 1 <i>s.</i> 1 <i>d.</i> per cwt. . .	15 19 10
2. 33 per cent. residue for feeding cattle = 11,371 lb. at 3½ <i>d.</i> per cwt. . . . .	1 10 2
3. 8,250 lb. leaves and roots at 3 <i>d.</i> per cwt. . .	0 18 6
	18 8 4
COST OF GROWING, &c.	
Topping stubble. . . . .	0 6 0
Twice harrowing. . . . .	0 3 0
Deep ploughing with four horses or steam .	1 2 0
Twice harrowing in spring. . . . .	0 2 0
Artificial manure. . . . .	2 10 0
Drilling, harrowing, drilling and rolling .	0 12 0
Hoeing and weeding. . . . .	0 2 0
33 lb. of beetroot seed at 3½ <i>d.</i> per lb. . . .	0 10 4
Horse-hoed or drilled three times. . . . .	0 6 0
Hoed by hand twice. . . . .	0 9 0
Thinning. . . . .	0 5 0
Banking the roots. . . . .	1 0 0
Sending roots to works. . . . .	1 16 0
To make the silos. . . . .	0 1 6
Interest on ground capital. . . . .	2 10 0
Ground dues and taxes. . . . .	0 18 0
	12 13 0
	£ s. d.
Amount realised per acre. . . . .	18 8 6
Expense of working (or all and every expense incurred). . . . .	12 13 0
Net profit per acre. . . . .	5 15 6

From this table it would be seen that the cost of production per acre was about 12*l.* 13*s.*, and as the produce brought in the market about 18*l.* 8*s.* 6*d.* there would be a profit of 5*l.* 15*s.* 6*d.* per acre for the grower.

He thought he had already anticipated Mr. Rhodes' question with regard to the fertilisers generally used very fully in the paper just read; but with respect to the two experimental fields, he was not then in a position to state exactly how they were manured. He could state, however, that no artificial fertilisers had been used but only ordinary farmyard manure. The crop raised at Wavertree the year before the beet was sown was oats.

The question was asked if the present year had not been an exceptionally good one for beetroot growing, to which Dr. Schaack-Sommer replied that his comparisons were made of course with roots grown during the same season, and therefore under similar climatic conditions, but speaking generally the yield of saccharine in 1889 was by no means abnormal nor equal to the highest of recent years, since the yield of saccharine in 1888 was over 13 per cent., whilst in 1889 the average was under 12 per cent. The weight of roots per acre, however, in 1889 was much heavier than in 1888; a circumstance which might be attributed to the extremely favourable conditions under which the beet was sown in 1889. Beet should be sown as early as possible in the month of April, while the ground contained a large amount of moisture.

Dr. Schaack-Sommer stated that the experimental seeds were procured for him by Professor Scheibler, who had selected them from the best source in Germany. They were of four different kinds, and were of course distinguished by different German names. He remembered when he was a student experimenting with beetroot in the laboratory of the Polytechnic School at Aix-la-Chapelle, and at that time his experiments were made with globular roots similar to the one exhibited. This kind of root had been abandoned in Germany for many years past, and the long carrot-shaped root had superseded it. Quite recently France had become alive to the superiority of the long root over the old-fashioned globular one, and for the past two years had been largely drawing her seed from German sources, with the result that the production of sugar in France had increased to an enormous extent.

With regard to the question of climate: climate of course affected the roots materially, but since the experimental roots had been grown in Wavertree and Ireland under the most unfavourable conditions, when the soil was not specially prepared for their growth, and the latter part of the season was cold and wet, and yet the yield from the roots compared most favourably with the best roots grown in Germany, it must be admitted that the experiment showed most indubitably that the climate of England and Ireland was highly favourable to the growth of sugar beets.

In reply to Mr. Hayes, Dr. Schaack-Sommer said that a "catch-plant" meant a plant to which the insect nematode would be attracted for the purpose of destroying them. For instance, after a crop of beetroot had been grown, and the ground was found to contain a number of these pests, a crop of catch-plants would be raised, the insects attracted to them, and then all destroyed together; then a crop, say of flax or any similar quick-ripening plant, might be sown, and after that was gathered a second crop of "catch-plants" raised the same season and so effectually clear the ground of the nematode, without losing the use of the ground for that season. I may just add before closing that a small factory, to do 50 tons per diem, including boilers, machinery, in fact, a complete plant (not including buildings), has been offered to me at 8,000*l*.

After Dr. Schaack-Sommer had replied, Dr. HURTER said that it was an important paper. He himself had seen such estates on the Continent as had been described. Dr. Schaack-Sommer had given them a number of financial statements of various companies; did those results include growing and manufacturing combined or the manufacture only?

Dr. SCHAACK-SOMMER: The manufacture only.

Dr. HURTER then said that about two years ago he had seen a sad picture. He had seen the ruins of what had once been a large establishment near Magdeburg. He inquired what had been carried on, and was told "sugar manufacture," and further that the works were not rebuilt because sugar did not pay. He was glad to hear of the dividends that had been lately paid, and with those figures in front of them he thought that somebody might be induced to erect works to procure a small market for beetroots. There was no doubt it would be a great assistance to the English and Irish farmers if they could make a profit per acre of 5*l*. 15*s*. 6*d*.

## Manchester Section.

Chairman: Ivan Levinstein.

Vice-Chairman: Edw. Schunck.

Committee:

J. Angell.

G. H. Bailey.

R. F. Carpenter.

G. E. Davis.

H. Grimshaw.

Harold B. Dixon.

J. Grossmann.

P. Hart.

A. Liebmann.

Sir H. E. Roscoe, M.P.

C. Truby.

D. Watson.

Hon. Local Secretary:

J. Carter Bell, Bankfield, The Cliff, Higher Broughton Manchester.

SESSION 1890.

Notices of Papers and Communications for the Meetings to be sent to the Local Secretary.

Meeting held at Victoria Hotel, Manchester, on  
Tuesday, 4th February 1890.

MR. IVAN LEVINSTEIN IN THE CHAIR.

### ON THE ESTIMATION OF ARSENIC ACID.

BY W. YOUNGER.

WHEN hydrochloric acid and potassium iodide are added to a solution of arsenic acid, iodine is liberated.

This takes place more readily on boiling, and if the boiling be continued for some time, the liberated iodine is eliminated, leaving a clear solution.

On examining this clear solution it is found that the arsenic acid has been reduced to arsenious acid, which on cooling and neutralising with sodium bicarbonate can then be titrated with standard iodine and the arsenic estimated.

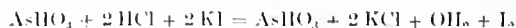
The following experiments were made in order to ascertain if the arsenic acid was completely reduced.

A measured quantity of a standard solution of arsenious acid was oxidised by the addition of hypochlorite of lime solution, acidified with hydrochloric acid and boiled to expel free chlorine, potassium iodide added, boiled until the solution became clear, cooled, neutralised and titrated, the iodine taken up being equal to the arsenious acid employed.

Again, a measured quantity of a standard solution of arsenious acid was oxidised by titrating with standard iodine, hydrochloric acid added, boiled until the solution became clear, cooled, neutralised and titrated, the iodine taken up being equal to the arsenious acid employed.

The iodine vapour driven off on boiling in the last experiment was absorbed by passing it through two bottles, each bottle containing the same measured quantity of the standard solution of arsenious acid neutralised with sodium bicarbonate.

The contents of the two bottles were then mixed and titrated for the excess of arsenious acid, when it was found that the arsenious acid which had been oxidised by the iodine vapour driven off on boiling was exactly equal to the iodine employed in titrating before boiling, showing that all the iodine had been eliminated. The reaction is as follows:



As an excess of potassium iodide is always employed, it is generally found on cooling that a further slight liberation of iodine takes place, but this is prevented by the addition of a few cc. of glycerine, which in no way interferes with the working of the method. Nitric acid and nitrates if present

must be eliminated. The more hydrochloric acid present the sooner the liberated iodine is driven off on boiling. Results obtained by this method agree closely with results obtained by reducing with sulphurous acid. This method has been employed for estimating the arsenic acid in various substances, and has been found to give results closely agreeing with those obtained by the other well-known methods for estimating arsenic.

#### DISCUSSION.

DR. BAILEY said that he did not gather from Mr. Younger's remarks whether he claimed any originality for the method. As a matter of fact the distillation of oxides with hydrochloric acid and estimation by passing the chlorine evolved into a solution of potassium iodide had been long ago published by Bunsen and by Fresenius, whose methods only varied in detail. The method was an admirable one and capable of a high degree of accuracy when proper precautions were taken. If its application to the determination of arsenic acid were new, then he thought Mr. Younger deserved the thanks of the Society for bringing it before them, though he had used it himself regularly in the laboratory as a general method, and took it that as issued by Bunsen it was intended to apply to all peroxidized compounds.

#### ANALYSIS OF DEACON GASES.

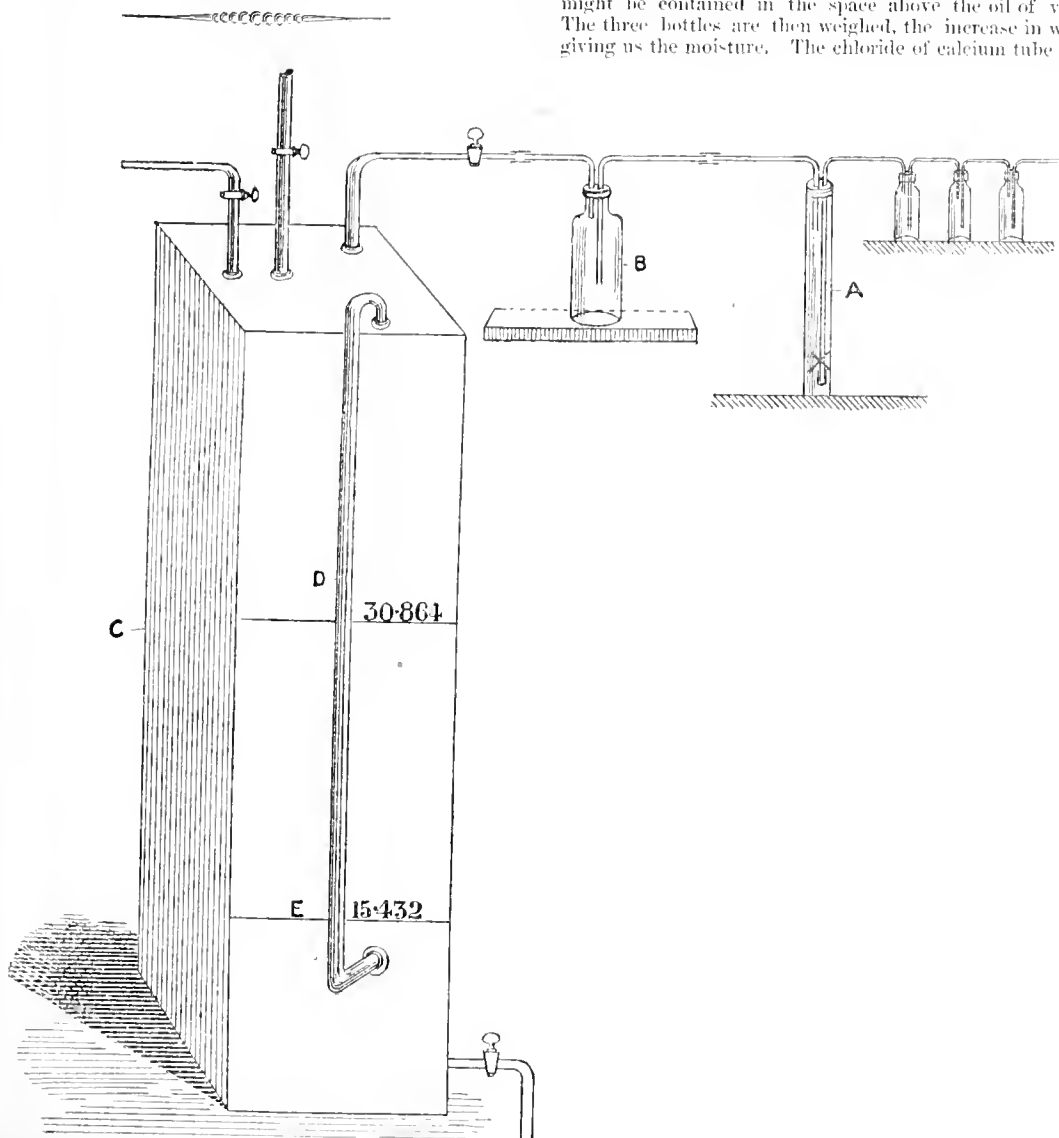
BY W. YOUNGER.

THE following is a method which I have employed to ascertain the composition of the Deacon gases.

The chlorine, hydrochloric acid, moisture, air and excess of nitrogen are obtained from one aspiration, which is taken by means of the apparatus described in my paper in the Journal of the Society of Chemical Industry, VIII., 88—89, to which is added three small bottles, about 30 cc. capacity each, containing oil of vitriol, each of which is weighed separately before taking an aspiration.

In the accompanying Figure the gases are first drawn through the bottles containing the oil of vitriol for the purpose of absorbing the moisture, then through the tube containing arsenious acid, from which we obtain the chlorine and hydrochloric acid, while by means of the oxygen apparatus we obtain a reading of the oxygen in the gases contained in the graduated box, which gives us by calculation the air, the remainder of the gases in the box being taken as nitrogen.

After the aspiration is taken a chloride of calcium tube is coupled up to the oil of vitriol bottles and air drawn through all three in order to remove any chlorine which might be contained in the space above the oil of vitriol. The three bottles are then weighed, the increase in weight giving us the moisture. The chloride of calcium tube is for





the purpose of drying the air before passing it through the oil of vitriol.

The method for obtaining the chlorine and hydrochloric acid is described in my paper referred to above, while the air and excess of nitrogen are obtained by calculation after having taken a reading of the oxygen. It was necessary, however, to ascertain if any of the chlorine or hydrochloric acid was taken up by the oil of vitriol, for which purpose the total chlorine was estimated, the contents of the three bottles being found to contain about 0.03 of a grain.

The volume of the chlorine, hydrochloric acid, moisture and gases contained in the box are taken as the volume of the gases which have been drawn through, and from which the per cent. by volume of each is calculated.

An aspiration of the Deacon gases was taken which gave the following results:—

	Per Cent.
Air .....	80.50 by volume.
Chlorine .....	4.41 "
Hydrochloric acid .....	0.03 "
Moisture .....	2.46 "
Excess of nitrogen (by difference) ..	12.57 "
	<hr/> 100.00 <hr/>

## Newcastle Section.

Chairman: T. W. Stuart.

Vice-Chairman: P. P. Bedson.

### Committee:

Alfred Allhusen.  
G. T. France.  
G. Gatheral.  
John Glover.  
T. W. Lovibond.  
John Pattinson.

H. R. Procter.  
B. S. Proctor.  
W. W. Proctor.  
W. L. Remondson.  
C. H. Ridsdale.  
J. E. Stend.

Hon. Local Secretary and Treasurer:  
Dr. J. T. Dunn, The School, Gateshead.

Notices of Papers and Communications for the Meetings to be sent to the Local Secretary.

Meeting held in the College of Science, Thursday,  
February 6th, 1890.

MR. JOHN PATTINSON IN THE CHAIR

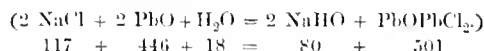
MR. SAVILLE SHAW exhibited a deposit of copper sulphate which had formed in the flue of a gas stove. The stove had a copper flue, and he explained the formation of the deposit, which, though greyish white, contained water nearly corresponding with that required by the formula  $\text{CuSO}_4 \cdot 5 \text{H}_2\text{O}$ , by supposing that the copper first oxidised, and that the copper oxide then brought about, in the way in which many metallic oxides are known to do, the combination of sulphur dioxide, from the combustion of sulphur in the gas, with oxygen; the trioxide so formed, or the acid formed from its reaction on the steam present, then forming copper sulphate with the copper oxide.

Mr. LOVIBOND instanced a similar case which had come under his knowledge, in which the bottom of a large brewing copper was destroyed and converted into copper sulphate through the use of a coal containing an unusually large amount of sulphur.

## PERSONAL RECOLLECTIONS OF THE "BACHET" PROCESS.

BY JOHN MORRISON, CHEMICAL ENGINEER, NEWCASTLE-ON-TYNE.

*Introductory.*—One of the earliest—if not the very earliest—of the various alkali processes carried out on the Tyne consisted in the decomposition of salt with litharge for the production of caustic soda—



This process was started at the Walker alkali works so far back as the year 1799, the salt being obtained from a brine shaft connected with the Walker colliery. The Walker works appears to have been the pioneer British alkali manufactory, and it is an interesting fact that the old hauling engine, with its massive oak beam and curious "sun and planet" motion—which had been erected in 1788 at the above pit by Messrs. Bolton and Watt—was not only a few years later saddled with the pumping of this brine, but was in active and useful work so recently as 10 or 12 years ago. Its steam was originally raised in (I believe cast iron) waggon boilers, and one or more of those of wrought iron which soon replaced the others, were, if I mistake not, in use as tanks up to the final dismantling of the Walker works.

How long the salt litharge process was continued at Walker's there seems to be no record to show; but it was probably abandoned prior to the adoption of the Leblanc process there in 1821. It is therefore not a little singular that at the very works, where half a century before the French process had probably ousted that of Lord Dundonald, a French gentleman was allowed to do his best to turn the tables on his own countryman, and to overthrow that process which the late Mr. William Gossage was wont to liken to a cat with nine lives, which none more than he had striven to destroy.

Up to the repeal of the salt tax in 1823 the Walker Alkali Company enjoyed the peculiar privilege of being allowed to make salt for soda manufacturing purposes free of duty. And with a litharge process so extremely imperfect it can only have been this fact, allied with what we should consider the then enormous price of soda, which at all enabled the manufacture to be carried to a remunerative issue.

In 1869 M. Bachet, a French banker and chemist, who had previously experimented at Channy, induced the Walker Alkali Company to erect experimental plant for the manufacture of caustic soda by his process, which differed from that of Lord Dundonald in the use of lime, and in the regeneration or revivifying of the lead for continuous use.

The plant was erected alongside what those who knew the Walker works 20 years ago will remember as the "hypo" house, where the Lo-sb hyposulphite soda process was carried on.

*Sodification.*—One part litharge, one part lime, half a part salt, and four parts water were ground together in an ordinary pan or mortar mill for about 20 minutes, by which time the creamy mass consisted of oxychloride and hydrated oxide of lead, undecomposed litharge, salt, and hydrate of lime, with caustic soda equal to 8 to 10 per cent. on the litharge employed instead of a theoretical 17.93 per cent. The use of lime, however, in this way was soon abandoned, for though it increased the solubility of the litharge, and caused a better decomposition, it possessed serious disadvantages. M. Bachet therefore subsequently treated two parts litharge and two parts salt with four parts water on the mill, which gave about the same total numerical result in soda as before, but with double quantities of lead and salt.

In practice the proportions were 2 cwt. litharge, 2 cwt. salt, and 4 cwt. water, these weights being adapted to the capacity of the plant in use. The mixture was ground on the mill from 20 to 30 minutes, by which time the whole had become white and thick. The large excess of water was requisite to bring the mass into a suitable consistency for pumping, and as soon as the mill operation was completed the charge was run off into a well, and thence forced into

filter-presses at a pressure of 100 lb. to 140 lb. per square inch. This probably was the earliest used of filter-presses for manufacturing purposes on the Tyne. The expressed liquors standing about 40° Tw. contained the soda together with a large quantity of salt, and PbO equal to about 1½ per cent. of the litharge used; and after precipitation with Na<sub>2</sub>S of the PbO as PbS, the clear portion was run into open pans and boiled down. The salt was rapidly as it collected was fished out and thrown on to perforated iron draining benches suspended over the pans, the steam from the boiling brine being intended to percolate these fishings, and to carry back to the pans their last traces of caustic soda. But it was an extremely imperfect—if not wholly useless—arrangement, and a hydro-extractor would probably have proved infinitely more efficient.

When as much salt as was possible had been fished from the liquors, they were run into a caustic pot to be finished in the usual manner. But it was found very difficult with the appliances and methods in use to completely separate the caustic soda from the salt. For whereas the first brine fishings contained only about 25 per cent. NaHO, the proportion steadily increased as the evaporation advanced, so that the later fishings would test as much as 15 per cent. to 16 per cent. NaHO.

Some of this excess was removed by lixiviation with fresh liquors, but the practical results were by no means as satisfactory as could have been desired.

**Regeneration.**—The magma from the filter-presses was returned to the mill, ground up with a little fresh water, and repressed to extract last traces of soda. The clear liquor standing about 18° Tw. was added to that from the first pressing; and the pressed cakes, which then weighed 25 per cent. to 30 per cent. more than the original litharge, were once more ground up with a little water and pumped into a conical iron regenerating vessel. Here a quantity of lime water equal to about 10 times the weight of the original litharge was added, the whole agitated for 30 to 40 minutes, and allowed to settle, the clear liquor being run off to vats.

Then the precipitate at the bottom of the regenerator was treated with a second 10 parts lime water, boiled for 15 minutes with constant agitation, and the clear liquor added to the first in the vats. During this operation the precipitate began to lose its white colour, and after another repetition with a third 10 parts water the clear liquor gradually became bright green. Finally a third boiling with only five parts lime water was given, and then, when after settling, the supernatant liquors were run off, the mass at the bottom of the vessel was found to be completely transformed into litharge again, and ready for a fresh operation on the mill.

The whole amount of water therefore used was equal to 35 times the weight of the original litharge employed. The first agitation with cold water extracted the last traces of salt, and (as calcium chloride) a portion of the chlorine combined with the lead. And the remaining washings with hot water removed the rest of the chlorine, besides dehydrating the hydrated oxide of lead. The decomposition of the PbCl formed of necessity the first reaction, for the presence of this PbCl prevented the last one taking place. And it was absolutely necessary to run off the clear liquors without delay, as a reverting reaction rapidly ensued.

The large volume of lime water employed necessarily carried away much lead in solution. An amount indeed equivalent to about one-tenth of the weight of litharge originally employed. It was necessary, therefore, to recover this by passing through the regeneration liquors in the vats a current of carbonic acid gas until a slip of blue litmus paper became red. The settled liquors having then been run off, the PbCO<sub>3</sub> precipitate was collected and boiled for half an hour in water to which the requisite quantity of lime was added for the formation of CaCO<sub>3</sub> and PbO. This reaction readily took place, but the difficulty then was to separate the lead from the chalk. And the only plan which seemed to offer was to maintain a gentle agitation just sufficient to keep the milky white CaCO<sub>3</sub> in suspension until siphoned gradually off. When this operation was carefully performed, about 50 per cent. of the PbO originally precipitated was found at the bottom of the vessel nearly free from CaCO<sub>3</sub>. The siphoned liquors containing the CaCO<sub>3</sub>

and the remainder of the PbO were then treated with sufficient hot water to dissolve the latter. And finally the CaCO<sub>3</sub> was settled out as worthless, and the clear liquor was removed for process use. It was, in fact, of the utmost importance to preserve this, for the CO<sub>2</sub> present in Tyne water would, if not got rid of in this way, have seriously affected the results in a process requiring such immense quantities of water for its completion.

I ought to mention that at first the PbCO<sub>3</sub> precipitates were regenerated in a specially constructed reverberatory furnace, but the method was so obviously objectionable that it was speedily supplanted.

The advantages of M. Bachet's newer method over the original "lime system" were as follows:—

1. Increased facility of regeneration. For whereas 100 parts water were necessitated by the old plan, 35 parts only of lime water sufficed in the amended one. In fact the excess of lime first used really retarded the regeneration, and the reduced quantity of water and lime in the new system considerably diminished the total quantity of lead dissolved even with the double quantity of litharge used. For the effect of the additional lime itself, apart from the increased water, was to augment the PbO solubility.

In the amended process the liquors from the first pressing contained about 25 per cent. PbO, and those from the second pressing about 10 per cent. PbO, which represented about one-third the quantity present in the lime process liquors.

2. The composition of the magma in the improved process was so comparatively constant that testings of each pressing were not absolutely necessary. This was decidedly not the case after three or four operations when working with lime.

3. An increased regenerative rapidity resulted in the new method from the more rapid settlement of the lead from each regenerating water.

I here subjoin the results of a series of operations which I find in my note-book, though in all probability they did not represent average work.

#### 1st operation:—

2 cwt. fresh litharge.  
2 " salt.  
4 " water.

Produce = 8·29 lb. NaHO = 3·7 per cent. on PbO.

Magma from presses = 288 lb.

Soda brine = 608 lb. containing 25 per cent.

PbO = 1½ per cent. on PbO.

Magma regenerated with 30 parts water to which 12 lb. CaO were added.

#### 2nd operation:—

Regenerated litharge.  
2 cwt. salt.  
4 " water.

Produce = 5·64 lb. NaHO and 214 lb. magma.

#### 3rd operation:—

Twice regenerated litharge with NaCl and H<sub>2</sub>O as before.

Produce = 5·83 lb. NaHO and 214 lb. magma.

#### 4th operation:—

Thrice regenerated litharge with NaCl and H<sub>2</sub>O as before.

Produce = 6·09 lb. NaHO and 215 lb. magma.

These four operations therefore required no less than about 13 tons water for regeneration, and 16 cwt. for sodification (less only the quantity returned to process from the PbCO<sub>3</sub> precipitating vats), against a production of 25·85 lb. NaHO.

**Objections.**—Hence, however promising the Bachet process might have been in theory, it must be evident that there were immense difficulties to overcome in practice. There were great volumes of water and large masses of solid materials to be moved about in order to secure a comparatively insignificant output, and where you had perforce to chuck in expensive litharge by the hundredweight, in order to extract with difficulty NaHO by (one might almost say) the pound, it becomes evident that stragulative commercial losses were almost inevitable. But M. Bachet himself was always hopeful and indomitably persevering. And yet while

I by no means think that the two years' experiments at Walker nearly exhausted the subject, they made it to my mind perfectly clear that the salt litharge decomposition would require to be much more complete, that the losses in irrecoverable, and in depreciated lead would have to be much less serious; that the enormous consumption of water would have to be greatly reduced; and that the separation of the  $\text{NaHO}$  from the brine and the fished salt would have to be rendered much less difficult before any practical man could reasonably anticipate an approaching dawn of success.

M. Bachel maintained that the new soda resulting from the reaction,  $\text{PbO} + \text{Na}_2\text{S} + \text{H}_2\text{O} = \text{PbS} + 2\text{NaHO}$ , would defray the cost of the  $\text{Na}_2\text{S}$  used to precipitate the lead from the pressed liquors. But the great depreciation in lead value involved by the sale of the  $\text{PbS}$  to the lead smelters, at one-third or one-fourth the cost of the original litharge, far more than counterbalanced this, and was indeed a rather discouraging drawback.

It was also hoped that the pressed soda liquors might be repeatedly employed on the mill with the regenerated litharge; but it was found that they caused no reaction without an addition of salt nearly equal to that employed at first.

In the same way it was also discovered that when fished salt was substituted for fresh salt, a considerably reduced chemical action ensued. That in fact whatever soda remained in the fished salt remained not simply dormant but in some occult way retarded the desired decomposition, and that hence, the same total amount only of  $\text{NaHO}$  was obtained from it as would have resulted from the use of a corresponding amount of fresh salt wholly free from  $\text{NaHO}$ . Then there was the great difficulty in separating the  $\text{NaHO}$  from the fished salt, to overcome which the conversion of the  $\text{NaHO}$  in the brine liquors into  $\text{NaHCO}_3$ , and recovery by precipitation from the more soluble salt was attempted. But so far as tried the plan was not a success.

Numbers of temperate and intemperate estimates as to manufacturing costs were from time to time during the course of the experiments painfully elaborated, but no one series of calculations, so far as practical accuracy was concerned, had probably any right to be regarded as more demonstrably reliable than another.

I have several among my stray memoranda, and may just give one as an example:—

Rough preliminary data for estimating manufacturing costs:—

Basis = batch of 5 cwt. litharge, 5 cwt. salt, and 10 cwt. water } With a production of 4 per cent.  $\text{NaHO}$  or litharge used.

Four per cent.  $\text{NaHO}$  or litharge = 22.4 lb.  $\text{NaHO}$  per batch.

Mixed liquors from 1st and 2nd pressing = 15 cwt. or	Lb.	Lb.
Less total water on magma	1,580	
	194	1,486

Add water in  $\text{Na}_2\text{S}$  precipitating solution = say 14

Total water to be evaporated from soda brine per batch = say 1,500  
per 22.4 lb. pure  $\text{NaHO}$  or 32 lb. 70 per cent. of caustic soda = 49 tons per 1 ton 70 per cent. caustic soda.

Salt per batch of soda liquor..... Lb. 569

Less salt retained in 70 per cent. caustic soda..... Lb. 3.25

Less  $\text{NaCl}$  per batch transformed into  $\text{NaHO}$  and  $\text{PbCl}$ ..... 42.2 45.5

Salt per batch to be fished out..... = say 514.5  
= say, 16 tons per 1 ton 70 per cent. caustic soda.  
Say, 1 part  $\text{CaO}$  soluble in 700 parts  $\text{H}_2\text{O}$ .

Line water for regenerating pressed magma = 35 times weight of original litharge, or 19,600 lb. per batch = 28 lb.  $\text{CaO}$  per batch or 17.5 cwt. per 1 ton 70 per cent. caustic soda.

Also 6.8 lb.  $\text{CaO}$  or 6,860 lb. (say, 7,000 lb.) lime water will be necessary for regenerating  $\text{PbCO}_3 = 4\frac{1}{2}$  cwt.  $\text{CaO}$  per 1 ton 70 per cent. caustic soda.

Total  $\text{CaO}$  per 1 ton 70 per cent. caustic soda = say, 22 cwt.

The calculation of this lime should nearly produce sufficient  $\text{CO}_2$  for precipitating the  $\text{PbO}$  in regenerating waters; or  $\text{CO}_2$  produced from coke would give, say, 7.7 lb.  $\text{CO}_2$  from 4.7 lb. coke (at 45 per cent.) to convert the 39.2 lb.  $\text{PbO}$  into 46.9 lb.  $\text{PbCO}_3$  or say 3 cwt. per 1 ton 70 per cent.  $\text{NaHO}$ .

Depreciation of lead in soda liquors. If latter contain 1 lb.  $\text{PbO}$  per 10 lb.  $\text{NaHO}$ , there will be 22.4 lb.  $\text{PbO}$  or 240 lb.  $\text{PbS}$  per ton pure  $\text{NaHO} = 165$  lb.  $\text{PbS}$  per 1 ton 70 per cent. caustic soda, which at, say, 15d. depreciatory loss per ton = 22s. 6d. per ton 70 per cent. caustic soda.

From this and a considerable quantity of minor data, I calculated on October 24th, 1871, the Bachel manufacturing costs per 1 ton 70 per cent. caustic soda to be as follows: It will be evident, however, that the estimate is extremely incomplete and speculative. For instance, I have presumed upon the cost of water to be wholly represented by the cost of pumping from the river, to say nothing of its storage, &c. Also the cost of finishing or fusing the caustic soda, and some other items, are probably very wide of the mark. Nothing also is down for oxidising sulphides or caustic liquors. Still, it formed an honest attempt at reasonable accuracy at the time named, the presumption being (somewhat unfairly, perhaps) that certain difficulties then unresolved were really practically overcome, or would be so with larger and more perfectly equipped plant. And the figures given having been reduced from those yielded by an assumed weekly output of 50 tons 70 per cent. caustic soda, were thought to offer a much better chance towards arriving at practical accuracy than any which might be directly based upon a single ton.

#### COSTS OF 1 TON 70 PER CENT. "BACHEL" CAUSTIC SODA REDUCED FROM FIGURES OBTAINED FROM A 50-TON WEEKLY ESTIMATE.

	£ s. d.	£ s. d.
Coals .....	3 11 8½	
Coke .....	0 1 2½	
Burnt lime .....	0 18 2½	4 11 1½
Depreciation on lead as sulphide.....	1 2 6	
Further loss of lead throughout process, say.....	1 0 0	
Loss of salt .....	1 3 1½	3 5 7½
Labour:		
Engines and boilers.....	0 3 10½	
Mills and presses .....	2 14 8½	
Oxychloride regenerators .....	0 9 7	
$\text{PbCO}_3$ regenerators .....	0 2 10½	
Soda liquors (precipitation of $\text{PbS}$ , &c. &c.).....	0 0 11½	
Soda liquors (evaporation, firing, fishing, &c. &c.) .....	1 13 1	
Preparing lime water .....	0 0 11½	
Finishing and packing caustic soda.	0 1 5	
Repairs and sundries .....	0 2 1½	
Caustic drums .....	1 0 0	6 9 10
Management, &c. ....	0 3 7	
Interest on capital and depreciation..	1 18 5½	2 2 0½
Total manufacturing costs .....		16 8 7½

I do not recollect at this distance of time the market value of 70 per cent. caustic soda in 1871, but in all probability it did not exceed 12l. to 15l. per ton.

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SESSION 1890.

March 26th: Mr. John R. Ashwell. "Some Physical Properties of Cotton."

Notices of Papers and Communications for the Meetings to be sent to the Local Secretary.

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### SESSION 1890.

March 4th (Glasgow):—

Discussion on Mr. Fawsitt's paper, "The Fixation of Nitrogen."

Mr. G. McRoberts. "The Manufacture of Blasting Gelatin."

Mr. J. S. MacArthur. "The MacArthur-Forrest Process of Gold Extraction."

March 5th (Glasgow): The "Graham" Lecture (subject: "On the Basicity of Acids and the Acidity of Bases") will be delivered by Professor Crum Brown, D.Sc., F.R.S., under the auspices of the Philosophical Society of Glasgow.

April 1st (Glasgow):—

Annual General Meeting of Section.

Mr. Jas. Hope. "The Estimation of Nickel and Cobalt."

May 6th (Edinburgh):—

Dr. J. B. Readman. "The Manufacture of Phosphorus, Part II."

Notices of Papers and Communications for the Meetings to be sent to the Local Secretary.

Meeting held 4th February 1890, in the Rooms of the Philosophical Society, Queen Street, Edinburgh.

MR. G. BEILBY IN THE CHAIR.

## AN ACCOUNT OF THE MANUFACTURE OF PHOSPHORUS.—PART I.

BY J. B. READMAN, D.Sc., F.R.S.E., F.C.S.

THERE are few more interesting elementary substances than phosphorus. Its history is associated with the later alchemists, and the uses to which it is now applied are numerous and important.

It appears to be nearly certain that phosphorus was first discovered by Brand or Brandt, an alchemist belonging to Hamburg, more than two centuries ago. He prepared the element from evaporated urine, which contains but a small amount of phosphoric acid.\*

The process was at first kept secret, but was afterwards sold, and then the method of preparing phosphorus began to be found out, for it appears that the Hon. Robert Boyle contributed a paper on phosphorus to the Royal Society in 1680. He obtained it, as Brand did, by heating a mixture of evaporated urine and sand in an earthenware retort, condensing the distillate.

About a century later Gahn discovered the existence of phosphate of lime in bones, and in a few years after we learn that the Swedish chemist Scheele described in detail a process by which he prepared phosphorus from bone-ash.

He warmed the bone ash with dilute nitric acid and then precipitated the soluble lime with sulphuric acid. He then

evaporated the liquid from which the gypsum had been separated to a thick syrup and distilled the dried residue with charcoal.

This roundabout process was first simplified by Nicolas and Pelletier, and afterwards improved upon by Fourcroy and Vauquelin. Those chemists treated the ground bone-ash direct with sulphuric acid, first determining the exact amount of acid required for its complete decomposition. About this time phosphorus was proved by Lavoisier to be an elementary body.

Bone-ash has been used for the preparation of phosphorus from the days of Scheele till quite within recent years. Now, however, native mineral phosphates of lime containing quite as much phosphorus as bone-ash contains have taken the place of the latter in the estimation of the phosphorus manufacturer, owing no doubt to the lower price at which it can be obtained.

When selecting a phosphate of lime for the production of phosphorus by the ordinary method care is taken to obtain one which contains a high percentage of phosphorus, and at the same time a minimum amount of iron and alumina. We shall see the reason of this when the details of the process are further described.

Pure bone-ash conforms to this description to a marked degree, as we may see by the following analysis:—

	Per Cent.
Phosphoric acid .....	39.55
Lime .....	52.46
Magnesia .....	1.02
Ferrie oxide .....	0.17
Silica .....	0.51
Carbonic acid, alkalis, fluorine, &c. .	4.43
Water .....	1.86
	100.00

Among the mineral phosphates that are met with in the market suitable for the manufacture of phosphorus those known as Canadian, Spanish, Somme, Sombbrero, and Norwegian apatite may be taken as typical examples. The following are analyses of these:—

	Canadian.	Spanish.	Somme.	Sombbrero.
Phosphoric acid ( $P_2O_5$ ) .	37.51	33.38	35.29	34.78
Lime .....	51.18	47.16	50.22	37.97
Carbonic acid' .....	..	4.10	2.97	1.46
Oxide of iron .....	..	2.59	2.46	..
Alumina .....	8.38	0.89		
Fluorine .....	..	..	..	..
Magnesia .....	..	..	5.20	0.46
Sulphide of iron .....	..	..	..	..
Sulphuric acid .....	..	0.57	..	0.45
Alkaline salts .....	..	..	..	..
Chlorine .....	..	..	..	..
Insoluble, &c. ....	2.85	..	5.61	2.84
Total water and organic matter .....	0.08	7.30	1.40	12.36
Undetermined .....	..	4.01	..	10.08
	100.00	100.00	99.15	100.00

Another mineral phosphate found native in one of the Leeward Islands in the West Indies and known by the name of "Redonda" phosphate—from the small island from whence it is derived—has been proposed as a source of phosphorus.\*

\* The island of Redonda is situated about 25 miles to the south-west of Antigua, 26° 6' lat., 61° 35' long., and is under British protection. Its length is estimated at one mile, its breadth at a third of a mile, and its height at 1,000 ft. The coast is rocky and only affords a single landing place, situated at the south-west, not far from the foot of a ravine in a rock. (See this Journal, 1889, 63.)

\* The usual amount of phosphoric acid in urine is .21 per cent. = .691 per cent. of phosphorus.

This mineral is not a phosphate of lime—like those just mentioned—it consists of phosphate of alumina and iron without any lime, and has the following percentage composition:—

	Per Cent.
Phosphoric acid ( $P_2O_5$ ) .....	39.23
Alumina .....	23.48
Peroxide of iron .....	10.60
Silica and insoluble .....	5.85
Combined water and organic matter ..	17.20
Moisture .....	3.66
	<hr/> 100.02

Equal to phosphorus 17.12 per cent.

Large quantities of Redonda have been shipped to this country since the deposits were discovered about 25 years ago, amounting, it is said, to 20,000 tons. The mineral was guaranteed to contain 35 per cent. of anhydrous phosphoric acid, and has been the subject of numerous investigations and patents, some of which are of an exceedingly ingenious nature.

For example, Spence\* dissolved the ground mineral in sulphuric acid, and to the solution he added sulphate of ammonia or sulphate of potash, and crystallised out the alum which resulted, leaving phosphoric acid in the mother-liquors. Townsend roasted the powdered phosphate after intimately mixing it with soda ash. The residue he dissolved in water, and on evaporation and crystallisation obtained tribasic phosphate of soda ( $Na_3PO_4 + 12 H_2O$ ), leaving aluminate of soda in the mother-liquors. The phosphate of soda was then dissolved in water and the phosphoric acid precipitated by milk of lime.

This process yielded a remarkably pure precipitated phosphate of lime.

Carbonic acid was next passed through the mother-liquors (of aluminate of soda), by which gelatinous alumina was precipitated and carbonate of soda left in solution ready on evaporation to be used over again.

The specimen of Redonda which lies on the table is taken at random from a large cargo imported some years ago into this country. It contains 35 per cent. of anhydrous phosphoric acid, and loses about 25 per cent. in weight when heated to dull redness, the loss being organic matter, combined water and moisture. The roasted sample contains a higher percentage of phosphorus than is contained in the purest phosphate of lime obtainable in commerce. Notwithstanding this, and the low price at which this mineral can be obtained, none of the processes proposed for the removal of the phosphoric acid appears to have been adopted.

In describing the manufacture of phosphorus as at present carried on, I purpose dividing it into several stages under the following heads:—

1. The preparation of phosphoric acid from phosphate of lime and sulphuric acid, with description of the apparatus employed.

\* Spence patented two processes, the first in 1870, which was incomplete, and was perfected by a second patent in 1873. The first idea was to dissolve the phosphate of alumina in sulphuric acid, and to the solution add ammonia or potash to form an alum; then crystallise. To the mother-liquid add ammoniacal gas water, iron, and any residue of alumina were precipitated, and the clear liquor was boiled down and dried to a state fit for manure; the amendment of this patent was that after the iron, &c. had been thrown down by the ammoniacal gas water and phosphate of ammonia obtained, pure ammonia was added to the monophosphate, and a tribasic phosphate of ammonia was formed.

The second process consisted in mixing the mineral phosphate with sulphate of soda (salt cake), coal, and a little oxide of iron, and heating these together until decomposition took place; then lixiviating out the phosphate of soda that is formed; to this solution caustic lime is added, phosphate of lime is precipitated, and caustic soda remains in the liquor; this is boiled down in the usual way; the precipitated phosphate is either used as it is, or it is converted into superphosphate.

Spence was so confident of the success of this process that he decided to erect plant on a large scale, and to alter the arrangement of his works. He was too sanguine, and his conclusions were at least premature, for after incurring heavy expenses the process had to be abandoned, and large quantities of the mineral were thrown on his hands.—"Life of Peter Spence, of Manchester, from Chem. Trade Jour., January 14th, 1890."

2. The concentration of the phosphoric acid, together with an account of the mixture of the acid with carbonaceous matter.
3. Phosphorus retorts and furnaces, with description of the distillation and condensation.
4. Refining and packing.

1. *The Preparation of Phosphoric Acid, &c.*—This is prepared from one or other of the mineral phosphates of lime, which is ground to a fine powder and then decomposed with chamber sulphuric acid of 1.55 sp. gr. The decomposition is effected in large wooden tuns made of pitch pine or other suitable wood, and provided with agitators also made of wood. Open steam is blown in during the operation. Sufficient sulphuric acid is added to convert all the lime present in the phosphate of lime into sulphate of lime.

After the expiry of some hours, during which the contents of the tuns are continually agitated, the emulsion is run off to large wooden filters lined with lead and provided with false bottoms or having ashes in layers of various sizes spread over the floor of the filters.

Here the hydrated sulphate of lime is separated from the solution of phosphoric acid, which passes through the filter as a bright and clear sherry-coloured fluid.

At first the phosphoric acid runs from the filters at 1.17 sp. gr., but gradually gets reduced by careful washing to 1.01 sp. gr.

Special precautions are observed to avoid cracks on the surface of the gypsum contained in the filters, the formation of which would afford too ready an outlet for the washing water.

Whenever the effluent from the filters reaches sp. gr. 1.01 the washing is stopped, and the hydrated sulphate of lime is piled up in the centre of the filters to drain, and finally it is thrown out.

This waste product usually contains about 62 per cent. of water, and when this is expelled by heat what is known as phosphatic gypsum is obtained having the following composition:—

#### Phosphatic Gypsum.

	Per Cent.
Sulphate of lime (anhydrous) .....	71.14
Phosphate of lime .....	6.56
Silicious matter .....	12.10
Oxide of iron, alumina, organic matter ..	5.55
Combined water and moisture .....	4.65
	<hr/> 100.06

2. *The Concentration of the Phosphoric Acid, and Apparatus employed.*—The strong phosphoric acid liquor flowing from the gypsum filters is blown by one of the acid-resisting steam elevators, "blow jacks," to stock tanks, and from there it is run by gravitation to the evaporators, which are circular tanks made of iron or wood, lined with thick lead, and heated by means of lead coils and high-pressure steam. Each evaporator is provided with an agitator, which is kept in motion during the evaporation.

Sulphate of lime, which is slightly soluble in the acid liquors, is deposited as the evaporation advances, and so great does this deposit become that filtration or decantation is necessary before the acid attains the degree of concentration required. If no agitation is employed during the concentration the gypsum firmly attaches itself to the coils, forming around them a thick coating, which must be removed by chipping.

The precipitated gypsum is thrown on a filter and washed in the manner previously described. When the clear acid liquor becomes concentrated to sp. gr. 1.4 or 1.5, the evaporation is stopped, and any deposited gypsum again separated.

The strong solution of phosphoric acid should now contain a mere trace of lime. Any iron, alumina, or magnesia present in the original phosphate of lime is dissolved to a considerable extent by the sulphuric acid treatment, and appears in this acid liquor.

A specimen of the concentrated acid is lying on the table containing 62.19 per cent. of  $P_2O_5$ , and no lime.

The strong syrupy acid is now mixed with carbonaceous matter, such as wood charcoal or coke, in coarse powder,

to the extent of about 25 per cent.; it is then carefully desiccated either in iron pots or in a muffle furnace so as to expel all the moisture, sulphurous acid, and hydrocarbons, and afterwards it is carefully stored away in air-tight iron boxes ready to be used in the distillation.

**3. Phosphorus Retorts and Furnaces with description of the Distillation and Condensation.**—The distillation is carried out in small bottle-shaped retorts made of the best weathered and prepared Stourbridge fire-clay; these retorts or pipes are about 3 ft. long by 11 in. external diameter—the clay being about 1 in. thick.

The retorts are placed in a furnace something like a Belgian zinc furnace in construction, but having only two tiers of retorts, each row being placed back to back with their narrow mouths projecting an inch or so through the brickwork. There are 28 retorts in one furnace, that is to say—taking one side of the furnace and considering it for a moment—there are in the first tier seven retorts and directly above these there are another seven, making 14 retorts which, as explained, project slightly through the side of the furnace. The other side of the furnace is similarly equipped.

The mouths of the retorts are connected to the condensers by 2-in. malleable iron or copper pipes which are luted with clay joints both to the retort and to the condenser.

Hot water is used in the condensing trough, so that whenever the phosphorus is condensed it fuses and runs down by gravitation to the lower end and may then be lifted out conveniently by a ladle.

Each retort holds between 20 and 30 pounds weight of phosphorus mixture and yields up the phosphorus existing in the free uncombined phosphoric acid in about 15 hours.

Whenever the distillation is completed the 2-in. pipes are disconnected from the retorts and the residue is rapidly withdrawn and the bench of retorts again recharged, the heat being steadily maintained.

Should a retort be cracked or fused either during the distillation or at charging, any phosphorus mixture it may contain is withdrawn, the dampers are closed and the retort removed from the furnace; a new retort, heated in an adjoining kiln to the required temperature, is substituted and all proceeds as before.

The crude phosphorus thus obtained is dark mahogany-brown in colour, as may be observed in the large specimen of this substance placed before you. It contains a good deal of impurity.

**4. Refining and Packing.**—The crude phosphorus is fused under water in a lead-lined circular tank heated by lead coils for high-pressure steam or steam jacketed. As much water as can safely be decanted off the surface of the crude phosphorus is removed and about 4 per cent. of bichromate of potash (on the weight of the crude phosphorus), is added in solution. An agitator with which the vessel is provided is set in motion and is continued in motion during the refining. After stirring for half an hour with the solution of bichrome about the same weight of oil of vitriol is added; in this way chromic acid is formed which oxidises the lower oxides of phosphorus and leaves the element pure and nearly colourless.

Another method of refining, and one more suitable under certain conditions, is to redistil the crude phosphorus in an iron retort. The phosphorus distils over rapidly and at a comparatively low temperature. A combination of these two methods is in some ways desirable.

The refined phosphorus is next moulded into "wedges" or "sticks." The "wedges" are made by melting the phosphorus and transferring it into circular tin dishes, 10 in. diameter by 4 in. deep, and when filled a star-shaped mould is introduced into each, which divides the circle into 10 wedge-shaped pieces, suitable for packing into tin cases. After this cold water is introduced, which soon solidifies the phosphorus and admits of its easy removal.

#### DISCUSSION.

Dr. DOBBIN asked how the inaccuracy of the text-book statement regarding the manufacture of phosphorus (which was so very explicit) was to be accounted for. Had the

statement no real foundation in fact, and who was the author of it? Was the process which Dr. Readman had described the one carried out by the very few phosphorus manufacturers in the country?

The CHAIRMAN asked if it was an essential feature of the process that the retorts should be as small as had been stated? He thought it would have added further to the interest of the paper if some idea of the cost had been given.

Dr. READMAN, in reply, said that it was not to the interest of manufacturers to correct mis-statements in text-books. With reference to the smallness of the retorts, in his experiments he commenced with a gas retort, but found it to be an entire failure, and he was of opinion that it was impossible to make a retort to stand the necessary heat more than 4 ft. long. As to the cost, he did not think it would be of interest to give it, but perhaps in his concluding paper he would say something in that connexion.

#### MOISTURE IN PAPER PULP.

BY G. H. GEMMELL, F.C.S., F.I.C.

STRAW and wood pulp as imported into this country from Germany and Norway—in bales of from 2 to 4 cwt. each—contains a high percentage of water, varying from 40 to 60 per cent., but the price of the material is usually based on the amount of air-dried substance present. The amount of moisture, however, in an air-dried pulp varies within considerable limits, according as the fibre is derived from wood or straw, and also according as it is prepared by mechanical means only, or by the soda, bisulphite, or other chemical process. It is also known that variations of temperature and humidity of the atmosphere causes a considerable change in this figure. In the paper trade there is no fixed standard adopted, some pulp manufacturers selling their material on the basis that it contains 12 per cent. of moisture when air-dry, others only 10 per cent., while many paper-makers affirm that 8 to 9 per cent. should be the utmost limit allowable; nor is there a uniform method adopted for calculating the weight of air-dried substance from the absolutely dry pulp, hence discrepancies and disputes often arise between buyer and seller. The paper-maker who tests his own pulp usually adds 10 or 12 per cent., as the case may be, to the percentage of absolutely dry pulp found, and this method of calculation is apparently acquiesced in by some of the manufacturers of pulp, but it is obviously incorrect. A sample of pulp containing, say, 60 per cent. of moisture and 40 per cent. of absolutely dry pulp, would by this method be said to contain 44 per cent. of air-dry material, but the correct method of stating is that if 90 of absolutely dry pulp becomes 100 air-dried, what will 40 absolutely dry become, this gives 44·44 per cent. as the correct figure. I have made a few experiments with a view of ascertaining what should be the average amount of moisture allowed for in an air-dried pulp, and though incomplete, think the results may be of interest to members of this Society. A sample of straw pulp containing 60 per cent. of water was allowed to remain exposed to the atmosphere, at the ordinary temperature of the laboratory, for 14 days, being turned over daily to expose fresh surfaces. A weighed quantity was then dried in a water oven, till constant in weight. 421 grms. of this air-dried pulp became 377 grms. of absolutely dry pulp, or a loss of 44 grms., which is equivalent to 11·67 per cent. of moisture, calculated from the dry pulp. Another sample of this same pulp, first made absolutely dry, was also exposed during the same 14 days, and was again re-dried. 1,102 grms. of the air-dried pulp became 1,017 grms. of absolutely dry, a loss of 85 grms., equivalent to 8·35 per cent. of moisture

Here we find that two samples of the same pulp, one wet and the other dry, do not become equal in the percentage of moisture after 14 days' exposure, but differ by 3.32 per cent., though at the end of the first seven days there was no perceptible difference to the touch in the wetness of the samples.

A similar trial was made with a sample of wood pulp containing 55 per cent. of moisture, and side by side with some of the same pulp previously made absolutely dry. At the end of five days there was no perceptible difference in the wetness of each sample, but they were kept exposed for five days more and a portion of each was then dried. The previously wet pulp by that time contained 13.6 per cent. of water, the previously dry pulp 10.6 per cent., a difference of 3 per cent. The remaining portions of these samples were exposed for a further seven days and then dried; the wet pulp then contained 12.2 per cent. moisture, and the dry pulp 10.4 per cent., a difference of 1.8 per cent. The moisture in the previously dry pulp thus remained practically constant while that of the wet sample more nearly approached that of the dried sample, and it was apparently only a question of being exposed for a sufficient length of time for the percentage of water to be equal in each. The weather during these trials was generally damp and wet, hence the results are probably above rather than below the average, and therefore 10 per cent. of moisture does not appear to be an unreasonable allowance to make for air dried pulp. In connection with this matter it may be of interest to note that the "Scotsman" and "Dispatch" newspapers contained when air dried 9.4 and 9.1 per cent. of moisture respectively, and two different makes of note paper 7.7 and 7.4 per cent. To obtain an average sample of the pulp for analysis is of course of the utmost importance, but owing to the nature of the material and the method of packing in bales is one of no little difficulty. 100 tons of pulp represent 1,000 bales, and to open 10 per cent. of these would be out of the question, and I have generally found that the paper-maker and seller agree that 10 to 15 bales in all are sufficient to open. The sample should also be taken as early as possible after the arrival of the consignment, and it might be advisable in some cases to weigh each bale before opening to remove the sample, though it is scarcely within the province of the chemist to do so, and from the figure thus obtained the paper-maker can calculate the amount of moisture originally in the bale. Thus suppose 10 bales invoiced at 3 cwt. each were weighed before taking the sample and were found to average 2 cwt. 3 qrs. or a total of 1 ton 7 cwt. 2 qrs., this is a total loss in weight of 2 cwt. 2 qrs. If on testing the sample drawn from these bales 55 per cent. of moisture is found, then the actual weight of dry pulp in the 10 bales would be  $\frac{45}{100}$  of 1 ton 7 cwt. 2 qrs. = 12 cwt. 1 qr. 14 lb., as the bales originally weighed 30 cwt. the amount of dry pulp then present was—

$$\frac{12 \text{ cwt. } 1 \text{ qr. } 14 \text{ lb.} \times 100}{30 \text{ cwt.}} = 41.08 \text{ per cent.}$$

It has been suggested that to obtain a sample a hollow steel punch should be driven through the bale, and the portion thus obtained transferred at once to a weighing bottle. Another method is to take a piece of the pulp from each sheet in the bale, but I think both these methods open to objection. The most satisfactory method is to open the bale to the middle, a sharp chisel being the best tool to use. Three strips 2 or 3 inches wide are then taken in every case right across the bale, from end to end. The first strip is taken from the external portion of the bale three or four layers deep, the second strip midway between the external and middle portions, and the third strip from the middle. 30 to 40 strips in all are thus taken. These are rapidly torn to pieces of about 1 in. square and thoroughly mixed, and from this a sample weighing 2 to 3 lb. removed to the laboratory for the analysis. The sample should be securely tied in oiled paper—this being more satisfactory than bottles—the loss of moisture not being more than a fraction of a per cent. In testing for moisture, weigh out 4,000 to 5,000 grains in a large beaker, dry for 22 hours in a water bath at 100° C. and weigh, and then again for a further two hours or till the weight is constant.

## DISCUSSION.

MR. IRVINE asked if the wood or straw pulp was sold on an absolutely dry basis, or as containing such a percentage of moisture as these fibrous materials attract to themselves? The reason he asked that question was, because he once knew a very serious lawsuit that arose over a somewhat similar case.

THE CHAIRMAN asked how far in a matter of that kind atmospheric moisture made a difference? He noticed that certain kinds of writing paper took the ink differently on a dry day from a wet day.

MR. GEMMELL said, in reply to Mr. Irvine, that the paper was always sold as containing a certain percentage of moisture. That basis varied. The Germans said that the paper absorbed 12 per cent., others said only 10 per cent., while paper-makers generally allowed 7, 8, or 9 per cent., at the most. As to the Chairman's question, the kind of weather made little difference in the amount of moisture absorbed, perhaps not more than 1 per cent.

## Journal and Patent\* Literature.

### I.—GENERAL PLANT, APPARATUS, AND MACHINERY.

#### PATENTS.

#### Improvements in Refrigerating and Freezing Apparatus.

H. Puplett and J. L. Rigg, London. Eng. Pat. 507, January 11, 1889. 8d.

This invention relates to improvements in that class of refrigerating apparatus in which a liquid is heated in one vessel, the gas or vapour being driven off into a second vessel, in which it liquefies by accumulated pressure and subsequent cooling. The first-named vessel being then cooled, and the pressure thereby reduced, the liquefied gas returns in the gaseous form, to assume which, it abstracts heat from the liquid surrounding the second vessel, being itself absorbed again by the mother-liquor remaining from the previous operation in the first vessel.

According to the inventors, an apparatus of this kind constructed in the ordinary way is liable to the danger of explosion from over-heating, to avoid which and to raise the general efficiency, they, instead of employing a liquid direct, use gas or vapour from an anhydrous liquid which readily gasifies, such as carbonic anhydride, ammonia, or ether. They next introduce into the first vessel, as an absorbent, any substance which does not evaporate, but possesses the property of readily absorbing the gas or vapour, such as charcoal, calcined clay or calcium chloride. They also prefer to make the first vessel in duplicate for working alternately,

\* Any of these specifications may be obtained by post, by remitting the cost price, plus postage, to Mr. H. Reader Lack, Comptroller of the Patent Office, Southampton Buildings, Chancery Lane, London, W.C. The amount of postage may be calculated as follows:—

If the price does not exceed 8d. ....	3d.
Above 8d., and not exceeding 1s. 6d. ....	1d.
" 1s. 6d., " " 2s. 4d. ....	1½d.
" 2s. 4d., " " 3s. 4d. ....	2d.



one being heated whilst the other is cooled. The specification is accompanied with one sheet of drawings on which two varieties of the improved apparatus are illustrated.—B.

*Improved Means for Preventing Ignition, and Retarding Combustion.* A. V. Newton, London. From A. Nobel, Paris, France. Eng. Pat. 568, January 11, 1889. 8d.

THE main object of this invention is to prevent the flame arising from the ignition of blasting charges in mines from communicating itself to the surrounding atmosphere and thereby igniting any fire-damp or combustible matters that may be suspended therein. It can also be applied to the protection of buildings from fire. To effect this, the air of the mine or building is saturated with water by means of a fine spray, thus rendering it incapable of supporting combustion, though mixed with volatile combustible liquids such as benzine (petroleum spirit), ether or bisulphide of carbon.

"Provided the dispersion is fine enough, no fire can be fed by atmospheric air holding its own weight of water in suspension."—E. S.

*Improvements in the Method of and Means for Concentrating or Evaporating and Distilling Solutions for the Production of Dyes and other Bodies.* J. Frost, Moldgreen. Eng. Pat. 1803, February 1, 1889. 8d.

THE improvements consist in the methods of heating the solutions and chemicals, and of promoting rapid concentration and evaporation, in order to economise time and labour, to prevent waste and to lessen the amount of heat required. A vertical coil formed of pipes of any suitable material has thin inclined plates fixed along each side of the pipes. The coil being heated by the passage of steam through it, the solution to be concentrated is allowed to flow on to the top of the first pipe, from thence on to those below in succession, and becoming gradually heated is "thrown off the pipes on to the thin plates, in the form of spray." The solution is thereby rapidly concentrated and ultimately falls into a receiver. Some modifications of the apparatus are shown on the two sheets of drawings attached to the specification.—E. S.

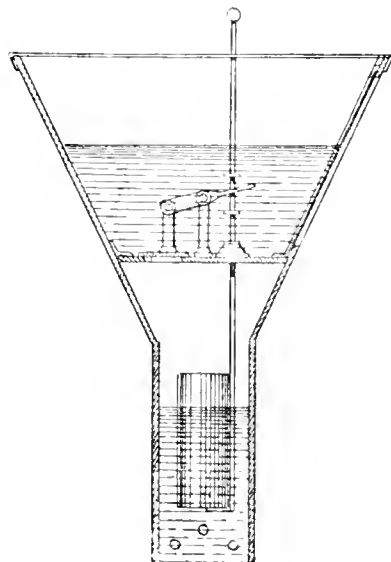
*Improved Means and Apparatus for Softening and Purifying Water, and for the Removal and Prevention of Incrustation in Steam and other Boilers.* E. Harrison, London. Eng. Pat. 1847, February 1, 1889. 8d.

A NUMBER of perforated central plates of copper and zinc of any suitable shape are arranged alternately, and placed between larger outer plates of perforated zinc or meshed wire, which are kept apart by studs, the whole apparatus being threaded on a central spindle provided with a nut and thumbscrew at the lower end, and a hooked head at the upper end, for readier transportation. The outer plates are cut radially, and are turned up in a fan-shape, the lower tips of the arms "forming feet upon which the apparatus stands upon an insulated base in the boiler," the whole, when in use, "inducing and establishing galvanic attractive action under the influence, and by the agency of the heat of the boiling water; and the incrustation of fur, instead of being deposited on the boiler plates, as usual, is deposited upon the meshed wire, &c., from which it is readily removed."—E. S.

*Improvements in or Pertaining to Funnels or Reservoirs for Liquids.* H. G. Payne, London. Eng. Pat. 1891, February 2, 1889. 6d.

THE lower part of the conical head of the funnel is closed with a diaphragm, in which a hole is left, closed by a valve.

The opening and closing of the valve is governed by a float, which is suspended in the narrow neck of the funnel, and



is acted upon by the liquid rising in the lower vessel as it is being filled, thereby preventing an overflow.—B.

*Separating Liquids of two different Densities.* Sir B. Samuelson, Bart., Banbury. Eng. Pat. 3016, February 20, 1889. 6d.

THIS invention relates to certain improvements in apparatus for separating liquids of different densities, such as milk and cream, and has reference more particularly to the position of the inlet and the outlet openings for the liquids, and to their adjustment. For details, the specification and drawing must be consulted.—B.

*Improvements relating to Distillation, and Apparatus therefor.* B. Hoff, Jaroslau, Austria. Eng. Pat. 3468, February 26, 1889. 8d.

THIS invention relates to the distillation or refining of liquids which are composed of fluids of various boiling points, the object being to obtain the purified product or products as a result of one distillation only, by means of inexpensive apparatus and without the use of cooling water.

The improvements consist in exposing to a succession of condensing surfaces of regulated areas the mixed vapours rising from a liquid under treatment, whereby, according to the principle upon which the improvements are based, the gases are condensed in the order of the boiling points of the resulting liquids, those possessing higher boiling points being condensed first, and those with the lowest last. The apparatus may consist of a number of upright cylindrical vessels conveniently grouped together, each vessel provided with a division or false bottom, on which the condensing material is placed, such as pebbles, shingle, or other silicious matter. Pipes connect the vessels to convey the vapours from the lower part of one to the upper part of the succeeding vessel, suitable valves being provided for drawing off the condensed liquor from each. The following examples are given of the extent of condensing surface required in the treatment of various liquors:—

To separate ethyl ether in a pure state (boiling point, 35.5° C. or 96° F.) from other vapours formed during its manufacture, which vapours generally consist of alcohol (boiling point, 78.4° C. or 173° F.) and steam (100° C.), 900 square metres (9,690 sq. ft.) of condensing surface are required when dealing with a volume of 100 cub. m. (3,532 cub. ft.) of the mixed vapours.

To separate pure wood spirit from water and the empyreumatic oils, 1,200 sq. m. (12,916 sq. ft.) are needed. The same for chloroform.

To separate benzene from toluene and its other attendant gases, 3,000 sq. m. (32,292 sq. ft.).

For aqueous sulphuric acid only 400 sq. m. (4,305 sq. ft.).

To separate petroleum vapour from steam, 14 sq. m. (150 sq. ft.) are requisite when obtaining a distillate having a boiling point of 150° to 170° C. (302° to 338° F.) and 0.82 sp. gr.—B.

*A "Boiler Composition," for the Cleansing and Prevention of Incrustation of Steam Boilers.* J. Parsons, Newnham. Eng. Pat. 8583, May 23, 1889. 4d.

This is a mixture of coal-tar, various oils, soda-ash, soap and tallow, which requires to be painted over the inside of the boiler.

It is claimed that the composition will in time fill up the pores of the plates, and so render the inside of the boiler "smooth and glass-like," and prevent oxidation and incrustation.—D. A. S.

*Improvements in Apparatus for Determining the Quantity of Moisture in Steam.* G. H. Barnes, Reading, Mass., U.S.A. Eng. Pat. 10,521, June 28, 1889. 8d.

In this invention the determination of the total quantity of moisture contained in steam is carried out in two stages. In the first, the steam drawn from the main is made to deposit part of its moisture by gravitation in a vessel which is provided with water-gauge and draw-off cock, whence the resulting deposit is run off and weighed. In the second stage the partially dried steam passes a thermometer previous to reaching a small orifice through which it issues into the atmosphere, whilst a second thermometer records the temperature of the issuing steam. As all the moisture remaining in the steam after the first deposition is evaporated during expansion, the issuing steam becomes superheated or attains a higher temperature than that due only to its density under atmospheric pressure. The difference of temperature recorded in this manner gives a measure of the amount of moisture left in the steam before expansion, and this added to the weight of water withdrawn from the depositing chamber determines the percentage of moisture as compared with the whole weight of steam passed through the apparatus. Numerical examples are given in the specification.—B.

*Improvements in Furnaces or Kilns for Burning Bone-Black.* A. Weber, New York, U.S.A. Eng. Pat. 12,070, July 30, 1889. 1s. 3d.

The furnace or kiln is constructed on the regenerative system and heated by means of gas generated in a separate furnace. The raw bones are fed automatically by gravity from stationary hoppers to the vertical retorts of the kiln. The bone-black retorts are oval-shaped, made of refractory material, and provided at their upper end with cast-iron mouthpieces which are closed by covers communicating by means of cast-iron sockets with feed pipes which are also connected with bottom openings of hoppers placed above the kiln. To the kiln placed vertically below the retorts are attached air-tight coolers fitted with dampers at their lower end, which are operated automatically by suitable mechanism so as to discharge the bone-black in small quantities at suitable intervals of time, a corresponding quantity of raw bones being fed through the feed pipes into the upper ends of the retorts. The gases of distillation in the retorts are drawn off by means of an exhaustor, and after the liquid products of distillation have been separated therefrom, the gas is reconnected to the furnace and utilised in heating the retorts. The tar obtained is also burned in the furnace and adds to the economic results obtained in working the kiln.

—F. S.

*Improvements in Apparatus for Condensing Vapours and Cooling Gases or Liquids.* J. Popper, Vienna, Austria. Eng. Pat. 16,763, October 23, 1889. 6d.

The improvements "relate to air condensers and cooling apparatus, in which air is used as the cooling medium, and the liquid to be cooled does not come into direct contact with the cooling air itself, but is contained in a closed vessel." For the mode in which this is effected the drawings accompanying the specification must be consulted.—E. S.

*An Improved Instrument for Sampling or Gauging the Depth of Liquids in Tanks, Vats, or other Vessels.* E. E. Horley and F. Sedgley, London. Eng. Pat. 17,275, October 31, 1889. 8d.

The instrument consists of a glass tube open at both ends, and having a valve adapted to close against the lower end. This valve hangs below the tube whilst it is descending through the liquid, but on reaching the bottom of the tank the progress of the valve is stopped; the glass tube, suitably weighted, continues its descent until it comes in contact with the valve, when a spring-catch retains the latter against the lower end of the tube. The instrument can then be withdrawn, when the nature and depth of the lower stratum of fluid, however small, being visible through the glass tube, can be measured by a scale.—E. S.

## II.—FUEL, GAS, AND LIGHT.

*Wery's Apparatus for Heating Retort Furnaces.* L. Brémond. *Journal des Usines à Gaz*, 1889, 153; *Proc. Inst. Civil Eng.* 99, 49.

THE cost of fuel for heating retort furnaces is a most important expense in the manufacture of gas. Various recuperative furnaces for utilising the waste heat are known, likewise the non-conducting coverings of Le Treust and others, besides other means that have been proposed for economising the fuel. It costs the Paris Gas Company 14½d. for fuel for producing 35,300 cub. ft. of gas, while the cost to many other companies is double that amount, and, in small works, the cost rises to as much as 9s. 7d. per 35,300 cub. ft. These differences may be partly due to variations in the price of the fuel; the coke being, in some places, valued at the selling price, while others calculate it at a uniform and conventional rate. However this may be, considerable variations do exist in the quantity of fuel used for heating the furnaces, even in the best managed works of similar sizes, and the differences become enormous if small works are compared with large ones. It is therefore desirable to find a simple and inexpensive means for economising fuel, applicable to all gasworks, and Wery's apparatus fulfils these conditions, effecting a saving of from 15 to 20 per cent. in the fuel.

On behalf of the Mechanical Arts Committee of the "Société d'Encouragement," Mr. Pillet has reported on Wery's smoke-consuming chimney, the principles of which are simply to mix a certain quantity of relatively cold air with the furnace gases in the chimney; this is effected by the application of the principle of Giffard's injector; the air mixes with the hot gases in the chimney, passing through a circular orifice, reduced in section as much as possible, and proportional to the chimney. The current of cold air surrounds the furnace gases in motion, mixes with them, lowers their temperature and diminishes the velocity of the draught. Without considering the theory of the principle, or determining whether similar results could be obtained by carefully regulating the dimensions of the exit orifice, Mr. Pillet found, from two carefully-made experiments, that the Wery process was efficacious both for smoke-consumption and economy of fuel. He experimented with a 12-h.p. portable engine and a tubular boiler without return flues or artificial draught. The consumption of smoke was perfect,

and the economy of fuel, with Wery's apparatus, for an equal amount of work, and for the same quantity of water evaporated, amounted to 23 per cent.

Other trials were made by Mr. De Bange, General Director of the old Cail manufactory at Paris, with 20-h.p. and 80-h.p. engines, and with puddling furnace and generator furnaces. Decided economy of fuel was found and also a sensible reduction in the time required for getting up steam. With the steam boilers the saving was about 20 per cent., and with the puddling furnace, in addition to the economy of fuel, the quality of the iron was superior to that obtained before using the apparatus. Mr. De Bange also made a trial to determine the quantity of water evaporated per pound of coal, with and without the apparatus, and found that the steam produced per pound of coal was increased by 28 per cent. by the apparatus.

Trials were also made at Denain by Mr. Jubeau with a puddling furnace on two steel ingots, each weighing about 4 tons, in two similar furnaces, the one with, and the other without the apparatus; these showed an economy of fuel of 16.79 per cent. in favour of the apparatus.

Other trials have also been made by Mr. J. Gaudry in the shops of the Compagnie Transatlantique, at Penhoet, which gave 10.2 to 10.7 lb. of water evaporated per pound of coal with the apparatus as compared with 7.8 lb. of water per pound of coal without it.

As regards gasworks, the Wery apparatus produces a similar effect to that of Le Treust, which creates an intermittent draught in the chimney and drives the products of combustion back into the furnace when the register is closed. With the Wery chimney, vacuum and pressure are alternately seen, according to the condition of the fuel. No machinery or steam is required. It can be fixed to existing chimneys, but the most convenient plan is to construct a brick foundation and place the apparatus on it, surrounded by a sheet-iron chimney. An apparatus and chimney of a total height of 30 ft., and the chimney  $11\frac{1}{4}$  in. diameter, was substituted for a brick chimney 62 ft. high and  $35\frac{1}{2}$  in. diameter; so that such large chimneys are not required, and the flames are not seen escaping at the top. At the small gasworks where it is in use, and where the dampers have to be closed each evening until the following morning, the heat is maintained and the furnace regains its ordinary temperature in half the usual time; there was also an economy of 16 per cent. in the fuel in the month of March, with intermittent working; but it will be easily understood that in winter, when the furnace is working regularly day and night, the economy would be considerably greater, the more so because the apparatus gives better results when the outer temperature is low. This was proved at the same works during a 12 days' trial in November last, when the saving in fuel was 28 per cent., as compared with the corresponding period of the preceding year. The Wery apparatus is of very simple construction, and requires only occasional cleaning when coal or tar are used for fuel. It is destined to become generally used in gasworks, as it will effect considerable economies which have not hitherto been realised by any other system.

*Storage of Petroleum used as an Illuminant for Prussian Lighthouses.* Zeits. für Bauwesen, 1889, 397, and Proc. Inst. Civil Eng. 99, 51.

PETROLEUM is almost exclusively used as the illuminant for lighthouses on the Prussian seaboard, and a description of the various special store-tanks is given, and their efficiency is considered; the principal ends kept in view being the avoidance of waste by leakage, and in drawing supplies, and maintaining a low temperature.

The tanks are of wrought iron, and generally of a size sufficient for storing a year's supply. Their shape is cylindrical, the larger ones of 3 ft. 3 in. diameter, 5 ft. high, constructed of  $\frac{5}{16}$ -in. to  $\frac{3}{16}$ -in. plates for the sides,  $\frac{1}{2}$ -in. to  $\frac{3}{4}$ -in. for the cover, and  $\frac{3}{8}$ -in. to  $\frac{1}{2}$ -in. for the base, that which is of the main importance being an efficient closing of the joints.

They are made in four different manners, viz.: 1st, of single plates with double-riveted joints; 2nd, of the same,

but with caulked joints; 3rd, of the same, but with brazed joints; and, 4th, with a double skin, forming a water-jacket, with single riveting and caulked or brazed joints. The third-mentioned method is that found to be the most efficient, although expensive. One objection to the water-jacketed tanks is that in frosty weather the water has to be let off.

Diagrams are given showing the various forms of tanks, and of the special brickwork cellars in which they are placed, these being generally isolated from the other buildings. The tanks are fitted with glass gauges, and are filled by a hose passed through a trap in the roof of the cellar.

For comparison with the present improved method of storage, a return is given relating to various lighthouse stations under the old system, when the oil was drawn direct from the casks, and varied from 5.4 to 13.7 per cent.

A tabular statement is given of the number of tanks and their form of construction at each of the 17 lighthouse stations, the names of the makers, the cost, cubical content, cost per litre, and percentage of loss per annum now and formerly, the greatest loss quoted being reduced from 13.7 to 1.01 per cent.

Particulars are also given of the cost of construction of the cellars at six stations.

#### PATENTS.

*Improved Methods of producing Combustible Gases and in Apparatus therefor.* B. H. Thwaite, Liverpool. Eng. Pat. 16,970, November 22, 1888. 8d.

THE gas distilled from coal in the ordinary way is caused to pass through a second retort containing incandescent coke, the residue from an earlier distillation. When gas has nearly ceased to be evolved, valves are reversed, and superheated steam is driven through both retorts, entering the second one first. Any heavy hydrocarbons that may have deposited in this second retort "are broken up, and re-arranged" by the superheated steam, which, both here and in the first retort, is itself decomposed, yielding a considerable quantity of water-gas before the temperature falls below the point required for this operation. The gas produced after the admission of the superheated steam may either be mixed with the ordinary coal-gas first obtained, or employed separately. In place of coke broken brick may be used in the second retort.—A. R. D.

*Improvements in Apparatus for the Manufacture of Gas for Illuminating and other Purposes, which Improvements also render said Apparatus suitable for other Uses.* F. J. Jones, Leek, and L. Alexander, London. Eng. Pat. 17,613, December 3, 1888. 8d.

THE process consists essentially in effecting the distillation of the coal or other material at a comparatively low temperature (so that the benzene and other hydrocarbons of high illuminating power may not be decomposed) by causing the gaseous products of combustion from a furnace to pass through a stratum of coal, so that the said products will be mixed with the gas and tarry vapours and other hydrocarbons which distil off, and in then superheating this mixture of gases and vapours, whereby the volatile constituents are converted into a rich permanent gas whilst the excess of carbon is deposited.

The gas, instead of being generated under the pressure necessary to force it through the hydraulic main and being allowed to cool down immediately after leaving the retort as in the ordinary retort system, passes freely or under exhaustion from the distilling chamber (no hydraulic main being used), and is then immediately superheated, "the gas passing from the superheater directly to the purifiers" (unless it is required to be cooled down, which may advantageously be done by passing the gas through a regenerator) after which it is ready for consumption.

The improved apparatus wherein this process is carried into practical effect is described, and drawings forming part of the specification are given, which it is necessary to consult in order to clearly understand.—F. S.

*Improvements in Apparatus for Utilising Liquid Hydrocarbon for Lighting and Heating Purposes.* F. B. Hill, London. Eng. Pat., 17,869, December 6, 1888. 11d.

Liquid hydrocarbon is forced by compressed air from a reservoir into an annular vaporising chamber which surrounds, or is surrounded by, an annular passage for the vapour from the chamber to the burner. This passage surrounds, or is surrounded by, another passage for the supply of atmospheric air to the burner. The chimney either extends upwards through the centre of vaporising chamber, or surrounds the air passage, thus enclosing the whole arrangement. The vapour and air currents travelling downwards meet under the vaporising chamber to form an annular flame, having a downward direction, and curling either outwards or inwards, according to the disposition of the chimney. It will be seen that the heated products of combustion, passing away up the chimney, may be made to heat the air required to support combustion, and to assist in the vaporising of the hydrocarbon, though this is chiefly effected by the action of the flame on the lower surface of the vaporising chamber.

—A. R. D.

*Improvements in Regenerative Kilns.* E. Brook and J. J. Brook, Huddersfield. Eng. Pat. 18,451, December 18, 1888. 11d.

The kilns are preferably built in sets of not less than four. Overhead run a main gas passage and a hot air flue. The upper part of each kiln constitutes a combustion chamber, and below this the goods to be fired are placed upon a floor perforated to allow connexion either with the chimney draught or with the outer air. The connexions and damper arrangements are such that while, say No. 1 kiln is being heated by the internal combustion of gas with hot air supplied from the overhead passages, the products of combustion escape through the perforated floor, and may be made to heat other kilns on their passage to the chimney. When the firing of No. 1 is finished, air is drawn in through the perforated bottom, and, passing through the kiln, reaches the hot air flue above in a highly heated state. It is now available either for supporting combustion in a kiln in course of firing, or for drying and heating goods in another kiln.

—A. R. D.

*Improvements in the Production of Water-Gas and in obtaining Hydrogen, Carbonic Oxide, or Carbonic Acid, each separately therefrom, also obtaining certain By-products.* C. F. Claus, London. Eng. Pat. 50, January 1, 1889. 8d.

Though "water-gas" produced according to any method may be treated by this process for the separate recovery of its constituent gases, the patentee prefers to prepare it by passing superheated steam through a layer of fuel, the relative proportions of carbonic oxide, carbonic acid, hydrogen and ammonia produced depending upon the depth of the layer of fuel and upon the temperature of the steam. By this means a continuous current of "water-gas" can be obtained without the use of an air-blast and the previous ignition of the fuel.

The gases, after cooling, are scrubbed or washed with ammonia in order to recover the carbonic acid (ammonia being also retained) as sequeicarbonate of ammonium, and then passed over oxide of iron after previously heating the gases to a temperature (100—500 C.) at which oxide of iron is reduced by hydrogen but not by carbonic oxide. The carbonic oxide passes on to a gasholder, while the hydrogen unites with the oxygen and leaves spongy metallic iron from which hydrogen is regenerated by means of superheated steam. Suitable apparatus is described for the continuous working of the process; for instance, the carbonic acid is driven off in a heating apparatus from its solution in ammonia, the ammonia being returned to the scrubbers for repeated use, while the carbonic acid, after passing through a tower containing sulphuric acid to retain any ammonia, and through an oxide of iron purifier, is led to the gasholder. The process may be used in conjunction with the ammonia-soda process, the free ammonia produced in this

latter process being used for the removal of the carbonic acid from "water-gas," while the sequeicarbonate of ammonium can be utilised in the ammonia-soda process.—O. H.

*Improvements in Apparatus for Carburetting Gas.* J. D. Hall, Liverpool. Eng. Pat. 387, January 9, 1889. 8d.

A COPPER box is divided into compartments by a number of perforated copper slides, or partitions. Between the copper slides are also arranged a series of flannel curtains or diaphragms, the lower portions of which dip in any suitable hydrocarbon contained in the box. The gas to be carburetted traverses the apparatus from end to end, and in passing through the flannel becomes charged with the vapour of the hydrocarbon, this latter being constantly replenished from below by capillary attraction. At both inlet and outlet are situated catch-boxes to obviate any difficulties that might be caused by condensation.—A. R. D.

*An Improved Composition of Artificial or Block Fuel.* C. H. Mowll, Dover. Eng. Pat. 1806, February 1, 1889. 4d.

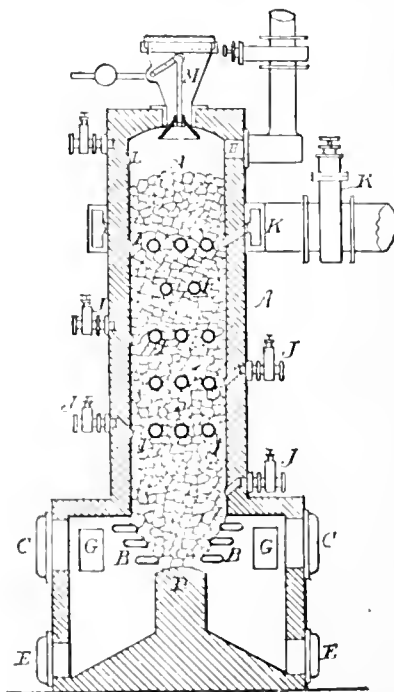
BRUQUETTES, which are stated to be less friable and of more value as a combustible than ordinary briquettes, are made by compressing a mixture of slack or small coal with pulverised peat. Cinders, ashes or calcined coal and an adhesive or agglomerative substance, such as pitch, may be added if desired, the mixture being moistened or slightly heated before compression.—O. H.

*Improvements in or relating to the Generation of Gases known as Water-Gas, Siemens' Gas, and Producer-Gas, and in Apparatus connected therewith.* J. von Langer and L. Cooper, Leeds. Eng. Pat. 7252, May 1, 1889. 11d.

This invention relates to improved forms of producers or gas generators, to the admission of air to the producer, and to improved valves and their arrangements, and mode of actuating valves in groups.

A is the producer, B the fire-bars, C the fire-cleaning doors, D the hearth, E the ash doors, F is an outlet for

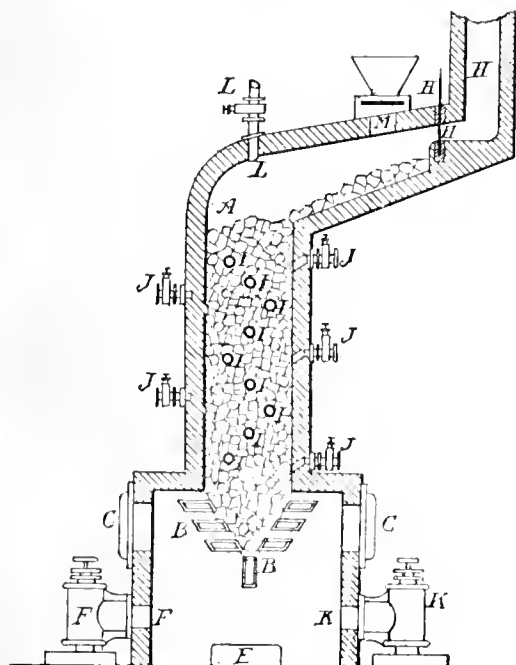
Fig. 1.



water-gas, and G a corresponding outlet for Siemens or producer-gas. At the top of the producer (in some cases) an outlet H is provided to carry off the products of combustion when lighting up or cleaning the fire, and is closed by a suitable valve. Air is admitted to the fuel in the producer through a series of openings I I, at different planes in the circumference, controlled by suitable valves J J. A special or main supply of the air blast is indicated at K, entering beneath the green fuel and controlled by a suitable valve K<sup>1</sup> placed in the blast pipe, the producer-gas generated finding an outlet through the openings G G at the base. When the air is shut off and the steam is turned on at L, water-gas is likewise generated in passing downwards and finds an outlet at openings F F (not shown) at opposite points to G G.

In this figure the main blast enters the bottom of the producer at K and passes upward through H, controlled by valve H<sup>1</sup>, while air is also admitted at points I I as in Fig. 1.

Fig. 2.



Steam is admitted at L, and the water-gas outlet is at F. The fuel is fed through hopper M, dropping upon an inclined shelf, from which it falls gradually into the body of the producer.

The specification further contains drawings of a producer adapted to turn in a circular direction upon an axial bearing, and in doing so to open or close the inlets for steam and air, and outlets for the gases. Also a producer mounted upon the ram of a hydraulic press, and adapted to move up and down in a vertical line.

A suitable controlling valve and box for admitting air at several points to the producer, and a water-gas valve, are shown in the drawings by detailed sections.—F. S.

*Improvements in Heating Boilers by Gaseous Fuel.* O. Inray, London. From E. R. Ware, Chicago, U.S.A. Eng. Pat. 15,043, September 24, 1889. 8d.

THIS consists in a novel arrangement whereby the exit for the waste products of combustion is fixed in about the same horizontal plane with that part of the heating chamber at which the fuel is ignited, whereby the consumption of fuel is carried on without a direct draught through the heating chamber; the waste products of combustion escape from the combustion chamber only by passing downward below

the plane of ignition, and thence out below the lower edge of the "water leg." The specification is accompanied by drawings.—F. S.

*Improved Gases or Gaseous Mixtures for Use in the Production of Light, Heat, and Power.* E. Tatham, Balmain, New South Wales. Eng. Pat. 16,138, October 14, 1889. 4d.

THE improved gases or gaseous mixtures are called "Carburetted Hydro-oxy Gas" or "Bi-carburetted Hydro-oxy Gas." In practice they are produced in one way by charging the hydrocarbon gas or hydrocarbon vapour with "carburetted oxygen" as the coal-gas issues from its place of manufacture, and while still hot, but not above the temperature of, say, 300° F., and the proportions of the mixture are regulated according to the illuminating power required, that is, for a gas or gaseous mixture suitable for ordinary illuminating purposes, and for use in engines as a motive power, 2½ per cent. of oxygen is added to ordinary coal-gas of 16 candle power. It is preferable to add to gases of a greater specific gravity or containing a larger quantity of carbon than coal-gas an increased amount of the carburetted oxygen. The mixtures so produced are said to give a most brilliant light, and are more economical for heating and engine purposes than ordinary coal-gas.—F. S.

*Improvements in Coal-Gas and like Gases, and in the Manufacture thereof.* E. Tatham, Balmain, New South Wales. Eng. Pat. 16,142, October 14, 1889. 4d.

THE improvement consists in the admixture with coal-gas or similar gases of a reasonable quantity of oxygen during manufacture, preferably as it issues from the retorts, and in the conversion of the tar produced or other similar hydrocarbons into gas or vapour, which is admixed preferably while still hot with oxygen. (Compare Eng. Pat. 16,138 of 1889, preceding abstract).—F. S.

*Improvements in the Manufacture of Gas.* D. H. Knapp, Norwich, New York, U.S.A. Eng. Pat. 16,646, October 22, 1889. 6d.

A CYLINDRICAL retort is set upright in a furnace, and a hole in the cover allows a similar but smaller retort to hang suspended by a flange. The smaller retort has an upright pipe fixed to the bottom, and open at both ends. Oil being fed into the smaller retort becomes vaporised, and the vapours being conducted downwards by the upright pipe into the larger retort, are there converted into permanent gas for illuminating purposes.—G. H. B.

### III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

#### PATENTS.

*Improvements in Apparatus for the Production of Pyro-ligneous Acid.* J. H. du Vivier, Paris, France. Eng. Pat. 2569, February 13, 1889. 8d.

See under VII., page 189.

*Improvements relating to Distillation and Apparatus therefor.* B. Hoff, Jaroslau, Austria. Eng. Pat. 3468, February 26, 1889. 8d.

See under I., page 167.

## IV.—COLOURING MATTERS AND DYES.

## PATENTS.

*Improvements in the Method of and Means for Concentrating or Evaporating and Distilling Solutions for the Production of Dyes and other Bodies.* J. Frost, Moldgreen. Eng. Pat. 1803, February 1, 1889. 8d.

See under I., page 167.

*The Production of New Azo-Dyes, applicable for Dyeing and Printing.* O. N. Witt, Charlottenburg, Germany. Eng. Pat. 2499, February 12, 1889. 6d.

THE patentee has discovered that  $\alpha$ - $\beta$ - $\alpha$ -dihydroxynaphthalene ( $\beta$ -naphthohydroquinone) will combine with diazo-compounds to form colouring matters capable of dyeing mordanted fabrics. In these combinations the diazo-compounds obtained from non-sulphonated amines are most suitable for preparing cotton dyestuffs, whilst the sulphonated compounds are used preferably in dyeing wool and silk. Ninety-three kilos. of aniline, 200 kilos. of hydrochloric acid (containing 37 per cent. of HCl), and 2,000 litres of water are diazotised at a temperature not exceeding 5° C. by the addition of an aqueous solution of 69 kilos. of sodium nitrite. This liquor is then run into a cold aqueous solution of 160 kilos. of dihydroxynaphthalene and 150 kilos. of crystallised sodium acetate in 2,000 litres of water. A dark orange precipitate is formed, which is filtered, washed and made into a paste with water. It may then be used for dyeing or printing on cotton in connexion with a suitable mordant. Alumina gives a bluish-pink, iron a greyish-violet, and chromium a purple. The following is an example of the method employed for preparing wool or silk dyestuffs:—184 kilos. of diazobenzene sulphonic acid (from sulphanilic acid) are suspended in water and added to the solution of dihydroxynaphthalene prepared as above. A dark orange solution results, and the dyestuff is salted out, forming an orange precipitate, which is filtered and dried. Its solution dyes wool mordanted with alum a dark bluish-red, whilst with chromium a bluish-purple. Other amines or their sulphonic acids may be employed in place of those already mentioned, such as toluidine, xyldine,  $\alpha$ - and  $\beta$ -naphthylamines, or the nitro- and hydroxy-derivatives of aniline, Brönner's  $\beta$ -naphthylamine sulphonic acid or Piria's naphthionic acid.—T. A. L.

*A New Colouring Matter for Dyeing and Printing Purposes, and its Method of Manufacture.* R. Guehn, Basle, Switzerland. Eng. Pat. 2635, February 14, 1889. 4d.

By heating dimethyl- or diethyl-*m*-amidophenol with succinic acid and zinc chloride, colouring matters are obtained which dye wool, silk and mordanted cotton red with a yellow fluorescence. 30 grms. of diethyl-*m*-amidophenol, 50 grms. of succinic acid, and 20 grms. of zinc chloride are heated for 15 minutes to 205°, or for 1—2 hours at 195°, allowed to cool, dissolved in 1 litre of water on the water-bath, and precipitated with 150 grms. of salt. The mother-liquor is decanted when cold, the precipitate again heated with 1 litre of water and 20 grms. of ammonium chloride, and reprecipitated with 150 grms. of salt. The mother-liquor is again decanted, and the semi-fluid residue dissolved in 10 cc. of hydrochloric acid and 1 litre of water, and evaporated to dryness. This colouring matter forms a very hygroscopic brownish-red powder soluble in water and spirit. The hydrochloride of the colouring matter obtained by heating equal weights of dimethyl-*m*-amidophenol, zinc chloride, and succinic acid anhydride to 190°—210°, forms green crystalline needles, also soluble in water and alcohol.

—T. A. L.

*Production of Triphenylmethane Colours Dyeing with the Aid of Mordants.* T. R. Shillito, London. From J. R. Geigy and Co., Basle, Switzerland. Eng. Pat. 3333, February 25, 1889. 4d.

A PROCESS for the synthesis of tricarboxylic acids of aurin and its homologues by oxidising salicylic acid or  $\beta$ -cresotinic acid (from *o*-cresol) together with bodies like methyl alcohol, formaldehyde, "methylal," dihydroxydiphenylmethanedicarboxylic acid, dihydroxyditolylmethanedicarboxylic acid in sulphuric acid solution with the aid of nitrous acid. Two kilos. of salicylic acid are dissolved in 15 kilos. of concentrated sulphuric acid and 4 kilos. of methyl alcohol. The solution is heated to about 70°, when 1.5 kilos. of sodium nitrite are added. After a short time the melt is poured into water, and the precipitate washed, neutralised with an alkali, and dried.—T. A. L.

*Manufacture of Azo Colouring Matters.* J. Imray, London. From "La Société Anonyme des Matières Colorantes et Produits Chimiques de St. Denis," A. F. Poirrier and D. A. Rosenstiehl, Paris, France. Eng. Pat. 4825, March 19, 1889. 4d.

COLOURING matters giving very deep colours extending to black are obtained by diazotising the mono- and disulphonic acids of aniline, of the toluidines, xyldines and naphthylamines, combining them with  $\alpha$ -naphthylamine, rediazotising the compound obtained and combining it with the phenyl, tolyl, benzyl, xylyl, or naphthyl derivatives of *m*-phenylenediamine or with the corresponding secondary and tertiary derivatives of cresylene or "xenylenediamine." 30 kilos. of sodium  $\alpha$ -naphthylamine disulphonate are diazotised in an aqueous solution with 7 kilos. of sodium nitrite and 45 kilos. of hydrochloric acid and combined with 900 litres of a solution of  $\alpha$ -naphthylamine hydrochloride containing 14.3 kilos. of  $\alpha$ -naphthylamine. The compound thus obtained is again further diazotised by adding 20 kilos. of hydrochloric acid and subsequently 7 kilos. of sodium nitrite in 15 litres of water. The diazo compound which precipitates is filtered off and added as a paste to 26 kilos. of diphenyl-*m*-phenylene diamine in alcohol. After standing some hours the liquid is acidified and the colouring matter which precipitates is filtered off and converted into the sodium salt.—T. A. L.

*Manufacture of Colouring Matters giving Grey, Brown, and Analogous Tints.* J. Imray, London. From "La Société Anonyme des Matières Colorantes et Produits Chimiques de St. Denis." Eng. Pat. 5032, March 22, 1889. 4d.

THE nitroso-derivatives of secondary or tertiary aromatic amines, when heated, form basic colouring matters varying in shade from brown to grey. 10 kilos. of nitrosodimethylaniline hydrochloride are dissolved in 50 litres of water and the solution boiled for three hours. After cooling the product is run into 100 litres of water and the colouring matter precipitated by the addition of zinc chloride. Alcohol or benzene, toluene, or other hydrocarbons may be substituted for the water, and in place of dimethylaniline the nitroso-derivatives of other tertiary and secondary amines may be employed. The colouring matters form brown powders soluble in water, alcohol and acids. Their solutions are precipitated by alkalis and alkaline carbonates giving brown flakes soluble in hot and insoluble in cold water.—T. A. L.

*Improvements in Colouring Matters.* H. H. Leigh, London. From R. G. Williams, Albany, U.S.A. Eng. Pat. 11,795, July 24, 1889. 6d.

A PROCESS for obtaining cotton dyestuffs by combining one molecule of a tetrazo compound with two molecules of the alkylised acids of the orchella lichens or compounds of the same, and also by combining one molecule of a tetrazo compound with one molecule of a phenol or amine and reacting with this intermediate compound on one molecule of the alkylised acids of the orchella lichens. These acids

can be obtained by the direct action of alcohols on the lichens or else by digesting the orchella lichens for a short time with water in presence of lime, filtering and precipitating the orsellinic and other acids by the addition of hydrochloric acid. The acids thus obtained are filtered off, dried at a low temperature and boiled with an alcohol in order to produce the alkylised derivative. A colouring matter which dyes unmordanted cotton a scarlet shade from an alkaline or soap bath is obtained by diazotising 28 lb. of benzdine sulphate in the usual manner and running the solution into 36.4 lb. of the methylated acids of the lichens, 100 lb. of caustic soda and 100 gallons of water. The dye-stuff forms immediately and is salted out, pressed, and dried. The following is an example of the method for obtaining a mixed azo colour. 30.2 lb. of toluidine sulphate are diazotised and run into 28 lb. of the sodium salt of  $\alpha$ -naphthylamine mono-sulphonic acid, 30 lb. of sodium acetate and 100 gallons of water. The mixture is well stirred, and when the intermediate product is formed it is run into a solution of 18.2 lb. of the methylated lichen acids and 100 lb. of caustic soda. The colouring matter thus formed is similar to Benzopurpurin 4 B, but is faster to light.—T. A. L.

*Improvements in Dye Colours.* H. H. Leigh, London, From R. G. Williams, Albany, U.S.A. Eng. Pat. 14,294, September 10, 1889. 6d.

This is an extension of Eng. Pat. 12,767, 1889 (this Journal, 1889, 981), and describes the preparation of isomeric intermediate compounds which will combine with amines and phenols. The following example illustrates the method employed: 32 lb. of toluidine sulphate are diazotised with 14 lb. of sodium nitrite and combined with 25 lb. of sodium naphthionate in presence of sodium acetate. This compound is then combined with the diazo compound from 25 lb. of naphthionic acid, and when the combination is complete 25 lb. more of sodium naphthionate are added, and the mixture stirred until the colour is completely formed. The chief claim is substantially as follows: The production of colouring matters by combining one molecule of a tetrazo compound with one molecule of an amine, then combining one molecule of a diazotised amine with the primary intermediate product thus produced, and afterwards reacting on the secondary intermediate product thus obtained with one molecule of an amine or phenol.

—T. A. L.

*Production of New Blue Colouring Matters.* S. Pitt, Sutton. From L. Casella & Co., Frankfort-on-the-Maine, Germany. Eng. Pat. 16,698, October 22, 1889. 4d.

BLUE colouring matters easily soluble in water are produced by the action of *p*-phenylene or *p*-toluylene diamine on certain basic substances which on reduction only yield polyamines. Those which have given the best results are the triamido-azobenzene (Ger. Pat. 32,502), and diamido-azoxybenzene (Ger. Pat. 44,045). The triamido-azobenzene has the formula  $(p\text{-NH}_2\text{C}_6\text{H}_4\text{N}_2\text{C}_6\text{H}_4\text{NH}_2)_3(o \text{ and } p)$ , and is obtained by the action of *p*-amidodiazobenzene on *m*-phenylenediamine. 10 kilos. of the hydrochloride of this base and an equal weight of *p*-phenylenediamine are heated for three hours to 130°–140°, and then for one hour to 160°–170° with agitation. The melt is dissolved in dilute hydrochloric acid, and the colouring matter precipitated with salt as a dark blue powder soluble in 5 times its weight of boiling water, and in 11 times its weight of water at 20°. It dyes mordanted cotton a deep blue fast to light and washing. The second method is to convert diamido-azobenzene ( $m\text{-NH}_2\text{C}_6\text{H}_4\text{N}_2\text{O}$ ) into its hydrochloride and mix it with *p*-phenylenediamine in the proportion of 1 to 3 equivalents, heating the mixture to 160°. The operation is finished in 3–6 hours, water being given off, and the melt is sufficiently pure to be used directly. It may be purified by dissolving it in dilute hydrochloric acid and precipitating with salt.—T. A. L.

## V.—TEXTILES: COTTON, WOOL, SILK, Etc.

### PATENT.

*Improvements in and relating to a New and Useful Fibre Produced from the Leaves or Needles of the Pines Australis and other Coniferous Trees.* W. Latimer, Wilmington, North Carolina, U.S.A. Eng. Pat. 13,127, August 20, 1889. 6d.

THE acicular leaves of the pine are first treated with an alkaline solution, washed and then passed through a decorticating machine, preferably that designed for the purpose (U.S. Patent 315,666, April 11, 1885). The fibre so prepared is carded or combed in any suitable machine, but preferably in that already mentioned, and is then dried, when it is ready to be spun and woven.—E. J. B.

## VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

### PATENTS.

*Improved Apparatus for Dyeing and Washing Hats and other Articles, applicable also to Veneering Hats.* T. Wareham, Toronto, Canada. Eng. Pat. 14,606, October 11, 1888. 8d.

It is claimed that by means of this invention, which cannot be understood without reference to the drawings accompanying the specification, the operations of mordanting, rinsing, dyeing, exposing to the atmosphere, and veneering can be successively and uninterruptedly effected.—E. B.

*Improvements in the Manufacture of Paperhangings, Curtains, Carpets, and other Articles or Coverings from Jute.* H. Sauvage, Paris, France. Eng. Pat. 17,574, December 3, 1888. 4d.

JUTE textiles are rendered suitable for wall decorations, carpets, &c., by dyeing or printing the same after they have undergone the following preparation: scouring with alkalis, bleaching with "chlorozone," shearing, and stiffening with Caragheen moss and dextrin.—E. B.

*An Improved Process for Dyeing Blue.* E. Edwards, London. From Standaert Brothers, Hamme, Belgium. Eng. Pat. 18,709, December 21, 1888. 4d.

A SHADE of blue resembling indigo is obtained on cotton by boiling the cotton material with a solution of extract of logwood and "soda salt," then working in a cold bath of ferrous sulphate, rinsing, passing again into the first bath, and tinting in a bath containing soap, salt, or sulphate of soda, and a colouring matter such as aniline blue. After being immersed in this bath for about an hour the material is rinsed and dried.—E. B.

*Improvements in and connected with Indigo Dyeing.* J. Cowan, Glasgow. Eng. Pat. 18,904, December 27, 1888. 6d.

INDIGO in a dry state is ground with zinc oxide or zinc powder, crystallised sugar and potassium carbonate. The mixture is suitable for making into parcels for sale. To set a vat the mixture is placed in a tub, water is added and then bisulphite of soda. The reduced indigo so prepared may, in a concentrated state, be supplied to dyers ready for their use. Instead of potassium carbonate other alkalis and alkaline salts may be used, and various other sugars are mentioned besides crystallised sugar. Sulphuric acid may, according to option, be added before or along with the bisulphite of soda.—E. B.



*Processes for the Manufacture of New Chrome Mordanting Substances, and for the Application thereof for Dyeing and Printing Purposes.* W. L. Wise, London. From M. von Gallois, Hoechst a/Main, Germany. Eng. Pat. 376, January 8, 1889. 6d.

NORMAL chromium chromate,  $\text{Cr}_2(\text{CrO}_4)_3$ , is prepared by dissolving chromic hydrate (1 mol.) in chromic acid (3 mols.); a basic chromium sulphate-chromate, of the composition  $\text{Cr}_2(\text{SO}_4)(\text{CrO}_4)(\text{OH})_2$ , is obtained by dissolving excess of chromic hydrate in a mixture of sulphuric and chromic acids or by double decomposition between solutions of basic chromium sulphate and potassium or sodium bichromate. An addition of magnesium or sodium acetate is made to these solutions before they are used. In mordanting for yarn or piece-dyeing the material is impregnated with the solution, squeezed, dried and aged or steamed. A printing colour is made by boiling and thickening a solution of basic chromium sulphate, cooling partially and adding potassium chromate and magnesium acetate, or the sulphate-chromate of chromium, made from the hydrate, may be used. The printed goods are dried and aged or steamed. Resists or discharges may be effected by printing thickened mixtures of organic acids, and when these are applied it is recommended to pass the pieces through a weak bath of carbonate of soda before dyeing.—E. B.

*Improvements in Machinery for Printing Floorcloths or similar Materials.* E. Ostlere, Kirkealdy, and A. M. Strathern, Springburn. Eng. Pat. 1283, January 24, 1889. 11d.

THIS invention cannot be properly explained without reference to the drawings which accompany the specification.—E. B.

*Improvements relating to the Washing or Scouring of Wool, Cotton Waste, and other Dirty Materials or Fabrics.* J. Portella, Hornsey. Eng. Pat. 1453, January 26, 1889. 6d.

THE use is claimed of a scouring mixture composed of soap, ammonia and turpentine. An advantage attending the use of such a mixture is that the more valuable impurities, such as lanolin in wool and the oily matters in cotton waste, can be easily recovered. When the mixture is applied for the purpose of cleaning cotton waste a further addition of potash or soda is made.—E. B.

*Improvements in Machinery for Printing Canvas, Floorcloth, Linoleum, and other Textile Fabrics specially prepared therefor.* L. Heyde, Dundee. Eng. Pat. 1759, January 31, 1889. 6d.

THE improvements in question consist in the combination of both block and roller printing in the same machine. The printing rollers are mounted in suitable framework on the same level. The material to be printed passes under the printing rollers and is pressed against the same by a series of pressure rollers covered with an endless travelling band. It then passes on to the block printing machine where the remainder of the pattern is printed. A claim is made for a method of supplying the colour to the blocks. For details of the mechanical arrangements the original specification must be consulted.—E. B.

*A Cold Process of Dyeing Hair and Feathers by means of Paraphenylenediamine or similar Bases.* H. Erdmann, Halle, Germany. Eng. Pat. 2525, February 13, 1889. 4d.

AFTER imbuing the feathers or hair with the solution of a para-amidophenol or a paradiamine, they are treated with a solution of either benzo- or tolu-quinone or an oxidising agent, such as hydrogen peroxide, ferric chloride or a permanganate. The following phenols and amines are specified; para-amidophenol, symmetrical triamidophenol, paraphenylenediamine, toluenyleneparadiamine, dimethylparaphenylenediamine and naphthylenediamine (1:5). In this way fast shades of brown to black are produced.—E. B.

*Improvements in Dyeing Mixed Woven Fabrics.* T. Ingham, Manchester. Eng. Pat. 2984, February 20, 1889. 6d.

By means of this invention animal and vegetable fibres in mixed fabrics can be simultaneously dyed black and other colours. The scoured goods are passed full width through a bath of one or more of the following colouring matters:—logwood, catechu, fustic, bark, berries, peachwood, with or without coal-tar dyes such as Benzo-azurine. After squeezing and drying, the colouring matter is oxidised and fixed by passing the cloth in the open state through a solution of any or mixture of some of the following:—bichromate of potash, bichromate of soda, bichromate of ammonia, chromic acid, nitrate of iron, sulphate of iron, sulphate of copper, carbonate of soda, or sulphate of soda. The dyed cloth is then rinsed or cleansed first in cold water and then in hot, or passed through a bath of carbonate of ammonia, carbonate of soda, or soap, finally being rinsed again in cold water before drying. Examples are given of the application of this method for dyeing black, blue-black, brown, and bronze, on fabrics composed of cotton and wool.—E. B.

*Improved Cleansing, Scouring, and Bleaching Compound for Wool and other Fabrics, also applicable to Cleansing Purposes generally.* A. H. Parker, London. Eng. Pat. 9410, June 6, 1889. 4d.

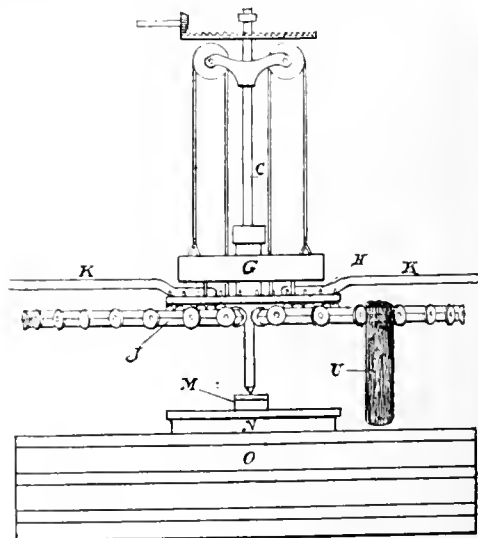
THE compound consists of a mixture of "pearlash, borax, ammonia, soft soap, Scotch soda, and Jeyes' disinfectant."—E. B.

*Improvements in Dye-extracting Apparatus.* F. Rhodes, Westfield. Eng. Pat. 15,558, October 3, 1889. 6d.

THIS invention relates to an improved form of apparatus used for extracting dye-woods. The improvements in question consist in employing, in combination with an ordinary dye-vat and appliances for heating and raising water, an extracting vessel of rectangular or cylindrical shape, one end of which is permanently closed, the other end being provided with a moveable steam-tight door. Water or liquor from the dye-vat is forced or pumped to the top of this vessel, and after passing through the dye-wood contained in it, flows back into the dye-vat. Several modifications of the arrangements are described.—E. B.

*Improvements in Apparatus for Dyeing Skeins.* J. C. Newburn, London. From La Société Grandire et Fils, Bolbec, France. Eng. Pat. 15,881, October 9, 1889. 6d.

THE skeins U are hung on the triangular reels J carried by the platform H, which slides on the square shaft C and is



counterbalanced by G. On the axis of each reel is fixed a pinion which, when H is lowered, gears with a crown wheel on the fixed platform N, so that the revolution of the platform H causes the rotation of the reels on their own axes. K K are bars for lowering the platform, O the annular dye vat, and M the footstep bearing for the vertical shaft. Instead of counter-balancing the platform, the shaft and the boss of the platform may be screwed, and friction rollers working on the edge of the vat may be substituted for the pinions on the reels and crown wheel on the fixed platform. In the Figure the platform is shown raised, in working the platform being lowered and fixed to ensure the contact of the pinions and crown wheel.—O. H.

*Improvements in Machines for Dyeing Cotton and Analogous Materials.* L. Weldon, Amsterdam, U.S.A. Eng. Pat. 18,973, November 26, 1889. 6d.

This invention relates to machines for dyeing raw or unmanufactured cotton. The improved machine consists of a perforated rotary cylinder or cage, which is placed in the dye-vat, so as to be partially submerged. The cylinder is provided with compartments, and at the centre of each compartment there is extended a rod armed with laterally-projecting pins or wings, termed a "breaker-bar." During the working of the machine the cotton contained in the compartments falls on the "breaker-bars," and is well distributed, so that even dyeing results.—E. B.

## VII.—ACIDS, ALKALIS, AND SALTS.

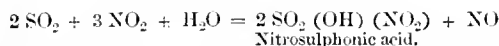
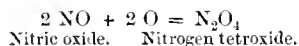
*Report by G. Wyss to the "Société Industrielle de Mulhouse" upon a Paper by E. Sorel, entitled "Etudes sur la fabrication de l'acide sulfurique."* Bull. Soc. Ind. Mulhouse, 1889, 237—239.

In his historical résumé of the work already done by other workers in this field, Sorel has failed to mention some of Lunge's researches, and others of Hurter and Eschellmann.

Sorel has set himself the task of demonstrating that temperature plays a preponderating part in the formation of sulphuric acid, and he has brought to light the important fact that it is the fall of temperature at the walls of the chambers that restarts the process of manufacture. This function he has thoroughly studied, and his determination of the tension of aqueous vapour in the presence of sulphuric acid, and of the tension of the nitrous compounds in solution in sulphuric acid, are researches of fundamental importance, which must be taken into account in all future investigation in this direction.

Nevertheless, his theory of the sulphuric acid process is not new. Lunge published an identical view in 1885 (Ber. 18, 1391), and again, in a more complete form in 1887 (Ber. 21, 67; this Journal, 1888, 208), at about the same time that Sorel's memoir was presented at Cherbourg.

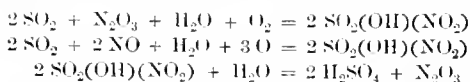
Among the reactions instanced as primary ones the following two—



should rather be looked upon as secondary. The author of this report points out also that Davy's theory is based upon the existence of hyponitric acid (nitrogen tetroxide) in the chamber gases, and that for this reason it is untenable, and not because, as stated by Sorel, it admits the formation of chamber crystals. In fact, the chamber crystals are nothing else but nitrosulphonic acid.

Lunge and Sorel are, therefore, the first propounders of the very probable theory that the sulphuric acid process does not depend essentially upon the reduction and oxidation

of the oxides of nitrogen, but upon the formation of nitrosulphonic acid and its decomposition by water, according to the equations—



The explanation of the reactions in the Glover tower are most interesting, and it can no longer be admitted that the affinity of NO for oxygen can produce of itself the formation of sulphuric acid in the tower, when the extremely short time, as calculated by Sorel, that the gases remain in the active zone of the tower is taken into consideration. The interpretation of the reactions in the first chamber is highly plausible.

It is evident that the cooling effect of the pipes connecting the chambers has a remarkable influence in facilitating the restarting of the process at the entrance of the second chamber. In this view Sorel agrees with Schertel (Chem. Ind. 12, 80; this Journal, 1889, 283). Lunge has ascribed this restarting of the process to the more complete mixing of the gases which would be brought about by passing through the pipes.

The application of his theory that Sorel proposes is certainly a possible one, and an economy in the first cost of plant will be realised by carrying out his ideas or in modifying their details. At any rate, it should be taken into consideration by every manufacturer desirous of keeping his methods of working up to date.—H. S. P.

*The Manufacture of Sulphuric Acid.* E. Sorel. Bull. Soc. Ind. Mulhouse, 1889, 240—293.

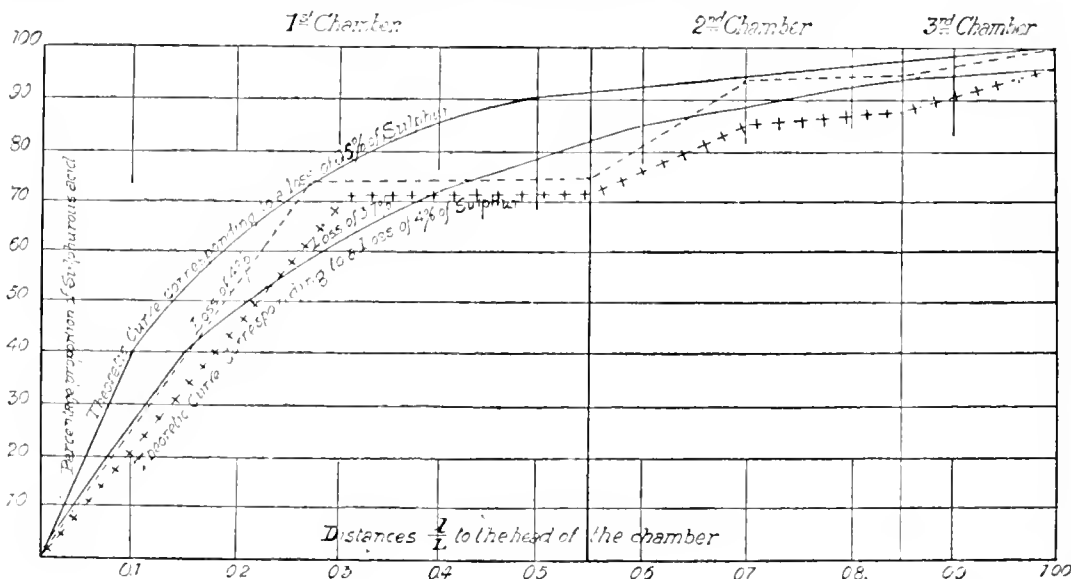
AFTER a short summary of the history of the sulphuric acid manufacture, and pointing out the progress that manufacturers have made up to the present time in the direction of economical working, showing that before the introduction of the Gay-Lussac and Glover towers only 1.75 kilos. of monohydrated acid ( $\text{H}_2\text{SO}_4$ ) per cubic metre of chamber space were made as compared with 4.25 kilos. per cubic metre to-day, the author draws attention to the accompanying diagram, which is taken from a publication by Lunge and Naef (Chem. Ind. 1884, 5; this Journal, 1884, 633), and which shows the curves representing the progress of the oxidation of the  $\text{SO}_2$  in the chambers of the Uetikon works. The ordinates of these curves represent the successive amounts of  $\text{SO}_2$  that have been oxidised per 100 parts of  $\text{SO}_2$  introduced into the chamber, and the abscissae the distances from the front wall of the first chamber. It is seen that the back parts of the apparatus do very little work, and that there are zones where the process is almost at a standstill. According to the author's own experience these curves fairly represent the general course of the process in the lead chamber. There is, therefore, room here for a further economy. But to know how to effect this, it is necessary to have a precise knowledge of the reactions that go on in the chamber, and of the causes which can modify these reactions. Upon this point the author maintains that the manufacturer has made scarcely any advance since the time of Clément and Desormes (1793), and he quotes from Scheurer-Kestner's article in Wurtz's dictionary to show this: "L'acide nitrique n'est que l'instrument de l'oxygénation complète du soufre: c'est sa base, le gaz nitreux, qui prend l'oxygène à l'air atmosphérique pour l'offrir dans un état qui lui convienne." After such an avowal of impotence, one can understand why manufacturers have had to renounce all originality of method, and to content themselves with perfecting themselves in the use of the apparatus bequeathed to them by their predecessors.

In considering the problem of the sulphuric acid process, the first thing to be noted is, that because the oxides of nitrogen act as a vehicle of oxygen, now removing it from the air and now giving it up to the  $\text{SO}_2$ , they must exist in a condition allowing of their easy dissociation. There is, therefore, no question of sharply defined reactions taking

place, no matter what physico-chemical conditions may exist in the surrounding medium, but rather of a series of transformations absolutely dependent upon the medium that surrounds the reacting materials.

If it were possible to unite all the most favourable conditions in every part of the chamber, the speed of oxidation of the  $\text{SO}_2$  at any part would be proportional to the  $\text{SO}_2$  remaining

in the gases as they traversed that part. The two plain-line curves in Lunge and Naef's diagram show the theoretical curves corresponding to a final escape of  $\text{SO}_2$  equal to 0.5 per cent. of sulphur burnt in one case and to 4 per cent. in the other; the broken line curves show the experimental data actually obtained for losses equal to 0.4 per cent. and 3.7 per cent. of sulphur burnt.



The theoretical and practical curves show a general concordance, but the differences that they do show are so sharp that it is not possible to look upon them as the result of chance. It is clear that we have to deal with conditions in certain zones of the chamber which hinder the progress of the reactions.

The conditions to be investigated are dependent upon variations in:—

- Temperature.
- Tension of aqueous vapour.
- Capacity of the nitrous products to combine with sulphuric acid.
- The relative proportions of oxygen and sulphurous acid in relation to the nitrous compounds.
- The reducing energy of sulphurous acid in relation to the nitrous compounds.

It is quite clear that oxidation and reduction of the nitrous compounds cannot take place simultaneously and in the same spot, nor can they occur in succession in the same spot unless the state of the surrounding medium at that spot varies.

A. *Temperature*.—There are three causes tending to produce a rise of temperature:—

- The continuous introduction of hot gases and of steam.
- The heat generated by the chemical reactions.
- The condensation of a part of the gaseous products.

The cause tending to produce a *fall* of temperature is the continuous radiation from the chamber walls. For each section of the chamber, therefore, a certain state of equilibrium will become established, and since of the three causes of rise of temperature that caused by the chemical reactions is the chief, the temperature inside the chamber will vary in degree above that of the air according as the chemical reactions are more or less intense in one part than the other. The temperature at the front of the chamber is therefore the highest.

But the walls do not transmit the whole of the heat produced in any one section, and consequently the temperature of the inner part of the chamber does not follow the same law, dependent upon the energy of the chemical reactions, and, as a matter of fact, at a height of 1.5 metres

from the floor of a No. 1 chamber there is only a difference of about 2° C. between the front and back of the chamber. At the surfaces of the walls, however, the gases are necessarily cooled. Moreover, within the chamber certain strata seem to be the chief seats of chemical activity, and sharp rises of temperature are indicated at such parts. Consequently, in a transverse section of a chamber we find different temperatures according to where the thermometer is plunged.

In a set producing about two kilos. of  $\text{H}_2\text{SO}_4$  per cubic metre the following temperatures were indicated in the first chamber, at points 1.5, 11.5, and 21.5 metres respectively from the end-wall at the front of the chamber. At each distance two holes were made in the top of the chamber, one in the axis of the chamber (designated by *a*) and the other .5 metre from the side (designated *b*). The experiments were made in the summer, so that the temperatures are relatively very high. The temperature at the top of the chamber 0.1 metre from the wall was 87°.

	1.5 m. from fore-end.		11.5 m. from fore-end.		21.5 m. from fore-end.	
	<i>a.</i>	<i>b.</i>	<i>a.</i>	<i>b.</i>	<i>a.</i>	<i>b.</i>
0.50 m. from top of chamber.....	86	76	79.5	90	74	85
3.50 m. from top of chamber.....	86	81	88.5	88.5	86	85
6.00 m. from top of chamber.....	85.5	83	82	83	84	82
6.15 m. from top of chamber, i.e., at 0.1 m. above the acid on the floor.....	84	79	85.5	85.5	84.5	79.5

Thus in spite of the radiations from the side we find the temperature at certain parts near it notably higher than the interior temperature at the axis.

In a set making 3.25 kilos.  $\text{H}_2\text{SO}_4$  per cubic metre, in which, of course, the temperatures were consequently very high, the author found in the first chamber, at 16 metres from the fore-end (0.4 of its total length), the following temperatures:—

(TEMPERATURE OF THE GASES IN THE INLET  
PIPE, 82.5°)

	At 0.15 m. from side.	At 0.50 m. from side.	In the Axis.
0.4 m. from top .....	94°	87.7°	88.4°
1.5 m. " .....	..	87°	88.9°
1.6 m. from floor.....	91°	..	..
0.5 m. " .....	..	88°	90.2°

At this distance from the fore-end, the highest temperature is seen to be close to the side.

In the same chamber the following temperatures were also noted:—

On the side wall.....	78.5 to 79.7
At 0.02 m. from side.....	84
0.04 m. " .....	91
0.06 m. " .....	95.25
0.08 m. " .....	95.25
0.10 m. " .....	98.5
0.12 m. " .....	97.3

The cooling influence of the side wall is therefore only felt close to the wall.

Such variations in temperature as are here indicated must cause movements in the gases which thoroughly mix them and give them a great uniformity of composition in each section. The author would agree with Abraham (Dingl. Polyt. J. 245, 414), that each molecule travels in a spiral round a horizontal axis from one extremity of the chamber to the other.

In these movements of the gases, the particles of liquid in suspension pass into zones of different temperatures, and consequently change their state of concentration and richness in nitrous products at each instant, so as to bring themselves into equilibrium with that portion of the gaseous atmosphere which surrounds them. Therein lies the secret of the rationale of the sulphuric acid process.

*B. Tension of Aqueous Vapour in presence of Dilute Sulphuric Acid.*—Heretofore we have only Regnault's experiments on this subject. But he only carried them up to a temperature of 35° C., which is much too low for the purpose under discussion. The author has extended his investigations to a temperature of 95°. Briefly stated, his method, which is more fully described in the original, consisted in aspirating an accurately measured volume of air very slowly over pumice-stone saturated with sulphuric acid of known strength, maintained at a constant and known temperature, and trapping the water-vapour carried over by the air, in a weighed Liebig's tube, filled with concentrated  $\text{H}_2\text{SO}_4$ .

The experiments were made at 45°, 60°, 77°, and 95°, with acids containing 50.25 per cent., 56.75 per cent., 65.75 per cent., 71.73 per cent., and 83.52 per cent. of pure monohydrated  $\text{H}_2\text{SO}_4$ . The results obtained and those obtained by Regnault for lower temperatures were expressed in curves, and the numbers contained in the accompanying table were then deduced by graphic interpolation.

TENSION OF AQUEOUS VAPOUR EMITTED BY DILUTE SULPHURIC ACID.

Percentage of H <sub>2</sub> SO <sub>4</sub> in Acid.	Temperatures.																		
	10°	15°	20°	25°	30°	35°	40°	45°	50°	55°	60°	65°	70°	75°	80°	85°	90°	95°	
Per Cent.	mm.	mm.	mm.	mm.	mm.	mm.	mm.	mm.	mm.	mm.	mm.	mm.	mm.	mm.	mm.	mm.	mm.	mm.	
44	4.4	6.1	8.5	11.5	15.5	20.9	28.1	37.4	48.3	..	..	..	..	..	..	..	..	..	
46	4.0	5.5	7.7	10.5	14.5	19.7	26.3	33.6	44.1	59.6	76.5	96.4	..	..	..	..	..	..	
48	3.7	5.0	7.1	9.6	13.4	18.1	23.9	30.5	40.1	53.5	69.0	86.8	107.2	132.1	..	..	..	..	
50	3.3	4.5	6.5	8.8	12.0	16.4	21.4	27.4	35.9	47.4	61.3	77.0	95.6	118.1	152.0	192.6	236.7	..	
52	3.0	4.0	5.8	7.9	10.9	14.5	18.9	24.1	31.5	41.5	54.0	67.9	84.5	104.5	131.2	166.5	207.9	251.5	
54	2.6	3.6	5.0	7.0	9.5	12.5	16.5	21.3	27.8	36.2	47.2	59.9	74.8	92.6	116.1	146.8	183.5	222.0	
56	2.2	3.1	4.3	6.0	8.1	11.0	14.2	18.5	24.1	31.0	41.6	51.6	65.0	80.6	100.9	128.2	160.0	195.0	
58	1.9	2.6	3.5	5.1	7.2	9.1	12.0	15.8	20.4	26.1	34.5	44.0	55.4	68.4	86.2	110.6	138.5	169.5	
60	1.6	2.1	3.0	4.3	6.1	7.5	10.0	13.0	16.9	21.6	28.7	36.7	46.1	56.7	72.3	94.0	118.7	146.0	
62	1.4	1.8	2.6	3.6	5.0	6.5	8.1	10.5	13.9	17.7	23.9	30.0	37.7	46.2	59.7	78.2	100.7	125.0	
64	1.2	1.6	2.2	3.0	4.0	5.5	6.5	8.2	10.9	14.0	18.7	23.9	30.3	37.4	48.0	63.8	83.7	105.0	
66	1.1	1.4	1.8	2.5	3.5	4.5	5.4	6.5	8.9	11.5	15.2	19.1	24.2	30.3	39.0	52.5	70.0	88.0	
68	0.9	1.2	1.5	2.1	3.0	3.8	4.5	5.4	7.2	9.5	12.3	15.4	19.4	24.1	31.4	42.5	56.0	72.0	
70	0.8	1.0	1.3	1.8	2.5	3.3	3.8	4.4	5.9	7.5	9.5	12.1	15.5	19.8	25.5	33.9	44.4	57.0	
72	0.7	0.8	1.0	1.4	2.0	2.8	3.2	3.6	4.8	6.0	7.5	9.5	12.0	15.4	20.0	26.2	33.7	43.4	
74	0.5	0.6	0.6	1.2	1.7	2.1	2.6	3.1	3.9	4.9	6.0	7.5	9.5	12.1	15.4	19.5	24.5	31.5	
76	0.4	0.4	0.5	1.0	1.4	1.8	2.1	2.5	3.0	4.0	4.8	5.9	7.5	9.5	11.8	15.0	18.5	22.0	
78	0.3	0.3	0.4	0.8	1.1	1.4	1.7	2.1	2.4	3.0	3.5	4.0	5.7	7.0	8.5	10.5	13.0	15.8	
80	0.2	0.2	0.3	0.6	0.8	1.1	1.3	1.6	1.9	2.4	2.9	3.3	4.1	5.0	6.2	7.5	9.3	11.0	
82	0.1	0.1	0.2	0.4	0.5	0.5	0.5	1.1	1.4	1.7	2.0	2.3	2.7	3.2	3.9	4.7	5.6	6.8	

It can be now seen how the differences of temperature shown to exist in the chamber will affect the concentration of the acid that is either held in suspension in the gases or collected in a thin film upon the walls.

Thus, referring to the temperatures given above at 16 metres from the fore-end of the chamber; the acid which was collected from the wall at this point showed an average of 52.5° B. = 66 per cent.  $\text{H}_2\text{SO}_4$ , and the temperature on the chamber side was 78.5° C., which corresponds to a vapour-tension of 39 mm. At the interior temperature of 95°, acid having the same vapour-tension as this would show 56.6° B., and contain 72.33 per cent.  $\text{H}_2\text{SO}_4$ . This agrees with practical experience, so that it may be concluded that the acids collected at different parts of the same transverse section are in equilibrium as regards the tension of the water-vapour they emit.

*C. Tension of the Nitrous Products in presence of Dilute Sulphuric Acid at Various Temperatures.*—By nitrous products the author designates all the oxides of nitrogen from nitric oxide (NO) to nitric acid, inclusive. He summarises the work done by previous chemists, chiefly by Lunge and Weber, and points out that they have shown that, in the presence of sulphuric acid in a suitable state of concentration, the oxides of nitrogen in contact with oxygen form nitrous acid, even when the oxygen is in excess, which then forms with the sulphuric acid a relatively stable compound. Further, Lunge has shown that nitrous acid which had previously been considered as quite unstable is nevertheless capable of existing at a temperature of 150°, and forms, with suitably concentrated sulphuric acid, a very stable compound under certain conditions, not reducible by sulphurous acid.

The author has attempted to show the conditions of equilibrium of this compound at various temperatures; and in order to avoid the influence of oxidising or reducing agents, he has employed an atmosphere of nitrogen.

He allowed a mixture of dilute sulphuric acid and of pure nitrous acid dissolved in concentrated sulphuric acid to flow, drop by drop, through a spiral tube 5 metres long immersed in a water-bath at a constant temperature, and passed a current of nitrogen in a contrary direction through the spiral; the quantity of nitrous acid in the mixture of acids before and after passing through the tube, and the volume of the nitrogen drawn through the tube being severally accurately determined, he was able to calculate the weight of nitrous acid given up by the mixture of acids used.

The following table shows the results he has obtained:—

Sp. Gr. of Sulphuric Acid.	Grammes $\text{N}_2\text{O}_3$ per Litre.	1 Litre of Nitrogen measured at 0° and 760 mm. Abstracts <i>n</i> mgrs. of $\text{N}_2\text{O}_3$ at a Temperature of $t^\circ$	
		<i>t</i>	<i>n</i>
1.824	142.0	37.0	mg. 0.079
1.791	28.4	41.4	0.009
.. ..	.. ..	61.2	0.049
1.774	28.4	40.1	0.009
.. ..	.. ..	62.1	0.005
.. ..	.. ..	80.7	0.274
1.745	25.876	29.5	0.044
.. ..	.. ..	61.4	0.196
.. ..	.. ..	75.0	0.402
.. ..	.. ..	88.9	0.980
1.672	4.734	29.5	0.29
.. ..	.. ..	46.0	0.37
.. ..	.. ..	75.0	1.29
.. ..	1.590	58.0	0.54

Sp. Gr. of Sulphuric Acid.	Grammes $\text{N}_2\text{O}_3$ per Litre.	1 Litre of Nitrogen measured at 0° and 760 mm. Abstracts <i>n</i> mgrs. of $\text{N}_2\text{O}_3$ at a Temperature of $t^\circ$	
		<i>t</i>	<i>n</i>
.. ..	1.640	90.0	mg. 2.72
1.666	9.403	45.0	2.69
.. ..	7.730	65.0	9.59
1.642	14.652	28.1	2.00
.. ..	.. ..	42.0	4.58
.. ..	.. ..	63.4	21.73
.. ..	.. ..	75.8	56.58
1.624	9.905	90.1	36.44
.. ..	10.795	89.9	40.39
1.606	11.558	91.0	64.38
1.603	1.259	70.0	4.90
1.601	2.500	40.0	0.94
.. ..	2.216	65.2	7.29
1.597	1.792	90.0	11.77
.. ..	12.500	89.9	109.15

He intends to continue the research to see if a law connecting these results can be found. At present the conclusion can only be drawn that the acid mixture behaves like a solution of nitrous acid in sulphuric acid, rather than as a compound. The dissolution of the nitrous acid is very rapid with rise of temperature, and the richness of the sulphuric acid in nitrous acid is probably a function of the tension of the nitrous compounds in the surrounding atmosphere.

When the degree of dilution is moderately large, the tension of the nitrous acid at high temperatures is sensibly proportional to the richness of the sulphuric acid solution.

*D. Influence of relative Proportions of Oxygen and Sulphurous Acid upon the Nitrous Compounds.*—The tension of the nitrous products in the gases being related to the degree of concentration of the sulphuric acid and to the richness of the solution of nitrous acid in sulphuric acid, it is clear that the latter solution will become richer or poorer according to whether the oxides of nitrogen in the gases are able to pass into the form of nitrous acid or not.

Lunge has shown that, in the presence of concentrated sulphuric acid, NO and O, even when the O is in excess, only produce nitrous acid, which then enters into solution.

On the other hand, pure sulphurous acid, acting upon warm nitrous sulphuric acid, reduces the dissolved nitrous acid just as it reduces the gaseous acid.

If, then, we have a mixture of sulphurous acid, oxygen, and nitric oxide in the presence of nitrous sulphuric acid, there will either be a reduction of the dissolved  $\text{N}_2\text{O}_3$ , or an oxidation of the NO, dependent upon the temperature, the degree of concentration of the sulphuric acid, and the relative proportions of sulphurous acid, oxygen, and of NO in the mixture.

Practice has shown that in extreme cases, with a high temperature, absence of oxygen, and excess of water, a reduction takes place, whilst when the oxygen is in excess, the acid concentrated, and the temperature low, there is oxidation.

The author has investigated, to a certain extent, the intermediate cases.

He caused measured quantities of a mixture of sulphurous acid, oxygen, nitric oxide, and water-vapour in known proportions to come in contact with nitrous sulphuric acid of various specific gravities, and containing various known quantities of nitrous acid at different temperatures, and

then determined the resulting amount of nitrous acid in the sulphuric acid.

The experiments showed that the reactions were complicated. The following points were, however, made clear. When the sulphuric acid has a higher specific gravity than 1.630, then sulphurous acid does not reduce nitrous acid, but forms with it in the presence of sulphuric acid a solution of nitrosulphonic acid, provided that there is an excess of oxygen and a greater quantity of nitrous acid present in the gases than corresponds to the tension of the nitrous acid held in solution by the sulphuric acid. In all other cases a reduction takes place. And that this statement also holds good when the specific gravity of the sulphuric acid is below 1.600, but only to a certain low limit of temperature (about 70° C.), beyond which a reduction takes place, even in the presence of an excess of oxygen and of nitrous acid.

*E. The Reducing Energy of Sulphurous Acid in Relation to the Oxides of Nitrogen.*—The author merely draws attention to the results already obtained, chiefly by Winekler, Weber, and Lunge.

Mixtures of NO, N<sub>2</sub>O<sub>3</sub>, and N<sub>2</sub>O<sub>4</sub> are not reduced by SO<sub>2</sub> if the gases are dry. A small quantity of water causes the formation of crystals of nitrosulphonic acid at the expense of the higher oxides; the NO is not altered. A large quantity of water produces the formation of H<sub>2</sub>SO<sub>4</sub>, but in this case NO is reduced even in presence of excess of O. With dilute sulphuric acid instead of water, there is no reduction of NO at from 40°–50° C. if the acid be not below sp. gr. 1.32. A reduction takes place if the acid be weaker than this. When the acid is concentrated the reduction becomes more difficult as the concentration is greater.

Nitric acid much diluted with water or with sulphuric acid below sp. gr. 1.36 is not reduced at the ordinary temperature, but heat causes its reduction. When the density of the sulphuric acid is above 1.4, reduction takes place in the cold accompanied by the formation of N<sub>2</sub>O<sub>3</sub>; reduction then becomes more difficult as the concentration of the solvent increases. In an atmosphere rich in oxides of nitrogen, a little nitric acid is found to be dissolved along with N<sub>2</sub>O<sub>3</sub>.

*Theory of the Sulphuric Acid Process.*—It has been shown in the preceding that a solution of nitrous acid in sulphuric acid liberates a certain quantity of nitrous acid in an inert atmosphere. The quantity liberated depends upon the concentration of the sulphuric acid, the richness of the solution (below a certain degree of concentration of the sulphuric acid), and upon the temperature. Let us eliminate a certain quantity of the nitrous acid, either by actual removal of it or by reducing some of it to nitric oxide. The solution will then liberate a fresh quantity of nitrous acid and will go on impoverishing itself if this be again removed.

On the other hand, if we reoxidise the NO the solution may take up again the N<sub>2</sub>O<sub>3</sub> formed.

If we raise the temperature of the solution the tension of the nitrous acid dissolved rapidly increases. If we dilute the solution the same result is observed, but the tension increases more rapidly.

It follows then that if, in an atmosphere containing a definite amount of nitrous acid, we bring together two solutions of nitrous acid in sulphuric acid at the same temperature, the one a solution in concentrated acid, and the other in dilute acid, the more concentrated acid solution will be enriched at the expense of the other. This might also take place if the temperature of the stronger acid were higher (to a certain limit) than that of the weaker acid.

Sulphurous acid does not hinder the dissolving of nitrous acid in sulphuric acid when the latter is of a suitable concentration, and if there is an excess of oxygen present, so long as the tension of the nitrous acid existing in the gaseous mixture, or capable of being formed in it by the oxidation of NO, is greater than the tension of the nitrous acid dissolved already in the sulphuric acid. But if the tension of the nitrous acid in the gases be less than that of the solution, then sulphurous acid facilitates the decomposition of the solution, whether we dilute the acid or raise the temperature.

Now let us suppose that we have a certain quantity of sulphuric acid of, say, 57° B., containing such an amount of nitrous acid as to be in equilibrium with the surrounding atmosphere at the temperature there existing, and let us suppose that this sulphuric acid is held in the state of mist in the atmosphere. Let us isolate a certain volume of the gaseous mass and cool it under constant pressure. A portion of the aqueous vapour in the atmosphere will thus be condensed both by the fall of temperature and by the contraction of the volume, and will consequently dilute the sulphuric acid suspended in the atmosphere. This dilution will at once destroy the equilibrium existing between the nitrous acid dissolved and that in the atmosphere, the sulphuric acid will liberate nitrous acid which will then be reduced by the sulphurous acid present, with formation of nitric oxide and sulphuric acid. The sulphuric acid formed will tend to again concentrate the acid solution. The reaction will therefore tend to stop when the quantity of acid formed and that submitted to the experiment approach each other.

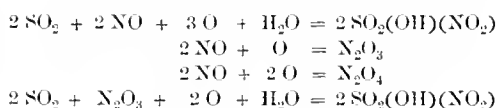
Now let us assume that this same gaseous mass is brought back to the initial temperature. The acid will then liberate water-vapour so as to bring itself into equilibrium with the conditions of the higher temperature, and will become again concentrated to 57° B. It will then be able to fix more nitrous acid, and will thus determine the re-oxidation of the nitric oxide, provided there is still a sufficient quantity of oxygen present.

In these variations of concentration and of temperature we find the immediate cause that brings about the successive reduction and oxidation of the oxides of nitrogen and the consequent rapid oxidation of sulphurous acid.

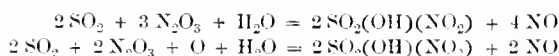
The following equations represent the reactions:—

#### 1ST SERIES.—FORMATION OF NITROSULPHONIC ACID.

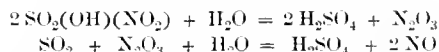
##### a. By Direct and Simultaneous Oxidation.



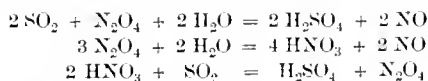
##### b. By Indirect Oxidation of SO<sub>2</sub>.



#### 2ND SERIES.—LIBERATION OF N<sub>2</sub>O<sub>3</sub>.



To these may be added the less important or accidental reactions:—



The author remarks that his theory resembles those of Davy and Winekler, but differs from them in not assuming the intervention of any body whose existence in the chambers is hypothetical. Davy's theory rests upon the formation of chamber crystals, which only are found in very abnormal working; that of Winkler depends upon the formation of hyponitric acid (N<sub>2</sub>O<sub>4</sub>) which is only found under particular and well-known conditions. (Compare preceding abstract.)

The author then applies his theory to explain in detail the whole sulphuric acid process from the burners to the Gay-Lussac tower.

*The Glover Tower.*—Not only is the acid introduced into the Glover tower, denitrated and concentrated, but there takes place there a formation of sulphuric acid equal to 16 or 18 per cent. of the total production in small towers, and to as much as 24 per cent. in large towers (having a capacity of 8 or 10 cubic metres per 100 kilos of

sulphur burnt), provided there is a plentiful supply of nitrous products.

According to Scheurer-Kestner (Compt. Rend. 1884, November 24, and 1885, March 2; this Journal, 1885, 457) from 1 to 9 per cent. of this sulphuric acid is contained as  $\text{SO}_3$  in the burner gases, which becomes hydrated in the Glover tower. Taking the higher figure, 9 per cent., this leaves an actual production of sulphuric acid in the Glover tower of from 9 to 16 per cent.

These amounts correspond to an introduction of a quantity of recovered nitrous acid equal to 8 or 10 parts of commercial nitrate of soda in the one case, and to 22 or 24 parts of nitrate of soda per 100 parts of sulphur burnt, in the second case. In both cases it may be assumed that nitrous acid from 2.5 parts of nitrate of soda per 100 parts of sulphur burnt are introduced to make up for loss. We have, therefore, the following quantities of available oxygen:—

Oxygen from recovered nitrous acid.....	0.74 or 0.90	1.60	2.21	
Oxygen from 2.5 parts NaNO <sub>3</sub> .....	0.22	0.22	0.22	
Total oxygen per 100 of sulphur burnt.....	0.96	1.12	1.82	2.43

Since 16 parts of oxygen are required to oxidise 32 parts of sulphur from  $\text{SO}_2$  to  $\text{SO}_3$ , it is evident that if a simple reduction of nitrous acid took place in the Glover tower there could only be produced there 0.48 or 0.56 to 0.91 or 1.22 per cent. of sulphuric acid. It is obvious, therefore, that the nitrous acid entering the tower must be successively reduced and oxidised, in round numbers, 20 times.

Take into account the fact that in the active zone of the Glover tower there is a maximum of 0.55 of empty space, and further that the denitration all takes place in the upper half of this zone; in the smaller towers, therefore, having from 4 to 6 cubic metres of working capacity, these successive oxidations and reductions must take place in a space of from 1 to  $1\frac{1}{2}$  cubic metres, while in the larger towers, having a working capacity of from 8 to 10 cubic metres, they take place in a space of from 2 to  $2\frac{1}{2}$  cubic metres (per 1,000 kilos. of sulphur burnt in 24 hours).

Assuming that there is admitted into the burners the minimum quantity of air compatible with good working, for every kilogramme of sulphur burnt there enters the tower 7,140 litres of gas measured at  $0^\circ$  and 760 mm. and in the dry state.

The composition of this gas will be—

	Litres.
$\text{SO}_2$ .....	699.1
O .....	593.2
N .....	5,847.8
	<u>7,140.1</u>

In a tower producing 18 per cent. of the total yield, this volume of gas will be reduced to 6,951.3 litres, and in a tower producing 25 per cent. of the total yield will be reduced to 6,877.9 litres, or there will pass through the working zone of the smaller tower an average volume of 7,045 litres, and through that of the larger tower an average volume of 7,009 litres. In two towers working at these rates, it was found that the mean temperature of the gases issuing from the smaller one was  $80^\circ$  and from the other one  $95^\circ$ ; by referring to a table of the tensions of aqueous vapour, it is easy to calculate the actual volumes of the gases at these temperatures.

In the first case it is—

$$7,405 \times \frac{760}{760 - 35.5} \times (1 + 0.293) = 9,542 \text{ litres;}$$

and in the second case—

$$7,009 \times \frac{760}{760 - 43.4} \times (1 + 0.348) = 10,006 \text{ litres.}$$

The time that this volume of gas is in the working space of the Glover tower varies, therefore, from 9 to  $21\frac{1}{2}$  seconds, and consequently each period of oxidation and reduction lasts from half a second to one second.

Such a phenomenon cannot be explained solely on the ground of the affinity of nitric oxide for the oxygen of the air. The author proceeds to show that it can be explained according to his theory.

He takes the actual case of an apparatus producing in winter 19,500 kilos. of  $\text{H}_2\text{SO}_4$  per 24 hours. The Glover tower had a working space of 55 cubic metres (by working space is meant the total space occupied by the packing above the grate), with a height of 5.6 metres. This tower was fed by 62,000 litres of  $60^\circ$  B. acid from the Gay-Lussac tower, and by 10,000 litres of chamber acid of  $55^\circ$  B. It produced 4,900 kilos. of  $\text{H}_2\text{SO}_4$  in the form of  $60^\circ$  B. acid per 24 hours.

Acid entering the tower:—

62,000 litres of $60^\circ$ B. =	106,082 kilos. of $60^\circ$ B.
10,000 litres of $55^\circ$ B. =	14,426 kilos. of $60^\circ$ B.

120,508 kilos. of  $60^\circ$  B.

corresponding to—

82,850 kilos. $\text{H}_2\text{SO}_4$ .....	+ 23,232 $\text{H}_2\text{O}$
11,301 kilos. $\text{H}_2\text{SO}_4$ .....	+ 4,849 $\text{H}_2\text{O}$
<u>94,151 kilos. <math>\text{H}_2\text{SO}_4</math> .....</u>	<u>+ 28,081 <math>\text{H}_2\text{O}</math></u>

Acid leaving the tower:—

$$120,508 + \left(4,900 \times \frac{160}{78.1}\right) = 126,782 \text{ kilos. of } 60^\circ \text{ B.}$$

corresponding to—

$$100,017 \text{ kilos. } \text{H}_2\text{SO}_4 + 26,765 \text{ } \text{H}_2\text{O}.$$

There left the tower, therefore—

$$28,081 - 26,765 = 1,316 \text{ kilos. } \text{H}_2\text{O}$$

in the form of vapour. To this must be added the water introduced with the nitric acid, which was equal to 118 kilos., making a total of 1,434 kilos. of water-vapour.

This weight of vapour was held in solution by the gases. The temperature of these at the exit of the Glover tower was  $95^\circ$ . In the dry state, and measured at  $0^\circ$  and 760 mm., their volume was shown above to be 6,878 litres per kilo. of sulphur burnt. Assuming that 1 kilo. of sulphur produces 3 kilos. of  $\text{H}_2\text{SO}_4$ , the total volume of gas on a total yield of 19,500 kilos. of  $\text{H}_2\text{SO}_4$  is—

$$6,878 \times \frac{19,500}{3} = 44,708 \text{ cubic metres.}$$

The nitrous acid consumed in the tower was equivalent to 24 parts  $\text{NaNO}_3$  per 100 of sulphur. Assuming that this left the tower entirely in the state of NO, there would leave

$$0.24 \times \frac{19,500}{3} \times \frac{30}{85} = 550.6 \text{ kilos. NO.}$$

If  $V$  = the actual volume of gas leaving the tower,  $f$  = the tension of the aqueous vapour,  $h$  = the tension of NO in the mixture, these values may be found from the equations—

$$V \times \frac{f}{760} \times \frac{0.805}{1.3477} = 1,434.$$

$$V \times \frac{h}{760} \times \frac{1.3434}{1.3477} = 550.6.$$

$$V \times \frac{760 - f - h}{760} \times \frac{1}{1.3477} = 44,708.$$

whence—

$$f = 28.8 \text{ mm., } h = 6.6 \text{ mm., } V = 62,355 \text{ cubic metres.}$$

Referring to the author's table of the tension of aqueous vapour emitted by sulphuric acid, it will be seen that if the acid introduced into the tower had the same temperature as the gases, viz.,  $95^\circ$ , an aqueous vapour tension of 28.8 mm. would correspond to a strength of  $74\frac{1}{2}$  per cent.  $\text{H}_2\text{SO}_4$  = sp. gr. 1.671. If the temperature were  $85^\circ$  it would contain 71.2 per cent.  $\text{H}_2\text{SO}_4$  = sp. gr. 1.64, if the temperature were  $75^\circ$  it would contain only 66.5 per cent.  $\text{H}_2\text{SO}_4$  = sp. gr. 1.575. Such differences would produce very different working in the tower.

In the case under consideration we have—

$$550.6 \times \frac{38}{30} = 697.4 \text{ kilos.}$$

nitrous acid issuing from the tower in a volume of 44,708 cb. metres of gas measured at  $0^\circ$  and 760 mm. = 15.6 mgrm.



nitrous acid per litre of gas. Acid of sp. gr. 1·671, even at a temperature of 90° in an atmosphere containing this quantity of nitrous acid, would dissolve a very considerable quantity of the latter and carry it down to a lower stratum. There, owing to the higher temperature, the acid would be denitrated little by little, the sulphurous acid would be oxidised, and the NO produced would re-ascend to the higher stratum, become re-oxidised, fixed by the sulphuric acid, and carried down again. It is this continuous circulation of nitrous acid which explains the action of the Glover tower as a producer of sulphuric acid.

It is obvious that the more concentrated the acid introduced into the tower the larger is the zone of denitrification and the better chance the nitric oxide has of being re-oxidised, so as to be carried down again; and consequently the more acid would actually be manufactured in the Glover tower.

The author then proceeds to show that if the acid used in the tower is too dilute a too rapid reduction takes place, and the Glover tower ceases to be an important factor of production. But even if the acid used be concentrated, the result is not satisfactory if the top part of the packing is cold, as the acid will there become diluted. The cooling of the packing is produced by three causes:—

1. Too small a sectional area, in which case the acid falls in large quantities upon the first layers and cools them.
2. A too great height of packing, so that the gases get too cold before they reach the top.
3. Exposure of the tower to the wind.

With regard to the third cause, the author has found great improvement in the working of a Glover tower by shielding it from the wind.

**Reactions in the First Chamber.**—In order to prevent the lead of the chamber being too rapidly attacked, manufacturers keep the acid on the sides at between 51° and 53° B. (sp. gr. 1·540 to 1·580 = 64 per cent. to 67 per cent.  $\text{H}_2\text{SO}_4$ ). Reference to the experiments of the author on the temperatures of the chamber show that the temperature of the wall is about 75° when that of the axis is 90°. For acid of 67 per cent. at the walls with a temperature of 75°, the tension of aqueous vapour is 27 mm.; the acid in suspension in the atmosphere having necessarily the same tension as this, but being at a temperature of 90°, must consequently contain 73 per cent.  $\text{H}_2\text{SO}_4$  (57° B.). If the acid on the wall only contain 64 per cent.  $\text{H}_2\text{SO}_4$ , the tension of its aqueous vapour will be 37·4 mm., and therefore the acid in the interior will contain 71 per cent.  $\text{H}_2\text{SO}_4$  (55·7° B.). These theoretical figures agree with the result of practical tests, several instances of which the author quotes, and justify the assumption that throughout each transverse section there is equilibrium of aqueous vapour tension.

He explains the inutility of the second half of a chamber as shown in Lunge and Naef's diagram, as follows. As the gases pass from one end of the chamber to the other they become poorer in  $\text{SO}_2$ . We have seen that in the interior parts of the chamber the heat given out by the chemical reactions is not compensated for by the radiation from the walls, and in fact we find a marked elevation of temperature towards the middle of the chamber; but from this point to the end of the chamber there is no material change at the axis.

This is clearly shown by the following observations of temperature at the axis of a chamber at different distances from the fore-end:—

	0·5 m. from Top.	3·5 m. from Top.
1·5 m. from fore-end .....	° C. 77	° C. 80
11·5 " " " .....	81	86
21·5 " " " (steam jet) .....	86	91
31·5 " " " .....	83	90
42·5 " " " (steam jet) .....	84	91

In the neighbourhood of the walls the temperature is found to fall barely 2°. Owing, however, to the decrease of sulphurous acid in the gases, the production of sulphuric acid tends to decrease, and the acid which condenses on the walls is always a little weaker at the end than in the middle (about 1° B.). The tension of the aqueous vapour is consequently greater, and the acid in suspension in the interior must be weaker. It is therefore less capable of determining the oxidation of the nitric oxide, and consequently of sulphurous acid, and the fact of this taking place just when the oxidation of sulphurous acid becomes more difficult, owing to the smaller quantity present in the gases, is the explanation of Lunge and Naef's diagram.

**Influence of the Connecting Pipes.**—The author ascribes the renewed activity of sulphuric acid formation at the entrance of the second chamber not to the better mixing of the gases in the pipe, but to the cooling influence of the latter. The gasses being cooled there 20° or 30°, condensation of water takes place which dilutes the acid in suspension and suddenly liberates from it a large quantity of nitrous acid, which thereupon produces the rapid oxidation of the sulphurous acid.

**Application of the preceding Views.**—The author suggests the following apparatus:—A Glover tower capable of producing 25 per cent. of the total yield, a chamber capable of oxidising 75 per cent. of the sulphurous acid that enters it (Lunge and Naef's diagram shows that this chamber would be about a quarter the capacity of ordinary chambers); following upon this chamber, a series of cooling pipes and small towers, the latter traversed by acid of 60° B. from top to bottom, whilst the gas and steam are introduced at the bottom. Assuming that these towers have each a cubical content equal to half that of the Glover tower, he reckons that with three towers there would be produced 93·27 per cent. of the total theoretical yield with a saving of about 60 per cent. in the capacity of the apparatus used.

**Conclusions.**—The author draws the following conclusions from his investigations:—

1. The successive oxidations and reductions of the oxides of nitrogen which determine the transformation of sulphurous acid to sulphuric acid are brought about by the variations in degree of hydration of the sulphuric acid suspended in the gases.
2. In the apparatus ordinarily used these variations of hydration are brought about by changes of temperature and are in direct relation to these.
3. The working of a sulphuric acid apparatus can be advantageously modified by increasing artificially and at certain points the concentration of the acid in contact with the gases and denitrating this acid at neighbouring points so as to bring about an active circulation of the oxides of nitrogen between the neighbouring zones.
4. The application of this process on an industrial scale should permit of a decrease in the first cost of plant and assure greater regularity of working.—H. S. P.

#### *Suggestions for the Improvement of the Sulphuric Acid Process.* F. Bode. Zeits. f. angew. Chem. 1890, 11—13.

It was first pointed out by Lunge and Naef (Chem. Ind. 1884, 5; this Journal, 1884, 633), and afterwards by Schertel (Chem. Ind. 12, 80—81; this Journal, 1889, 283), that only the first half of the sulphuric acid chamber was efficient. Schertel says, "only in the first half of the first chamber does the sulphurous acid become oxidised, whilst in the second half the quantity of sulphurous acid in the gases undergoes no decrease. After passing into the second chamber a revivification of the process takes place, but dies out again as soon as the gases have passed through the first half of this chamber," and so on in the other chambers. Schertel then proposes to substitute a number of small chambers for the large systems now in use. Lunge (Zeits. f. angew. Chem. 1889, 385; this Journal, 1889, 774—775) thinks Schertel's proposal too crude, and suggests the adoption of a preliminary mixing apparatus for the gases, together with suitable means of cooling within the apparatus. Further, he proposes the use of towers or columns of perforated porcelain plates (plattenthürme) between the

chambers, in which the enfeebled reactions will again be set going.

Sorel (this Journal, 1890, 181) recommends a system of cooling tubes and towers down which 60° B. acid is allowed to percolate.

The author points out that Schertel's suggestion merely means the same thing as the subdivision of the existing length of chamber into two parts, so that each part then becomes a chamber—in other words, substituting two short chambers for one long one. The term "short" is, however, a relative one, and instead of decreasing the length we may make a short chamber by increasing the breadth. An extreme case would be that in which what is now called the length of a chamber would be considered its breadth and *vice versa*. In such a case the gases would enter at one of the long sides of a chamber and pass out at an opening in the opposite long side.

Schertel's plan involves an increased expenditure of sheet-lead, the author's a decreased expenditure. Supposing it is desired to convert a set of a total capacity of 3,240 cm., consisting, say, of three chambers each 6 × 6 × 30 m. Following Schertel's plan, and making it into six chambers, this would mean an increase of wall-surface in the ratio of 100:109. Adopting the author's plan we have: (a.) The number of chambers (three) remains unaltered, but the breadth of each is doubled; this means a decrease of wall-surface in the ratio of 100:86.4 (b.) Six chambers are made, each of 12 m. width; this means a slight increase of surface—about 4.5 per cent. If only five chambers be formed, however, the surface is the same as in the case of the original three chambers.—H. S. P.

#### *The Formation of some Fluorine Salts and their Industrial Value.* M. Netto. Zeits. f. angew. Chem. 1890, 45—46.

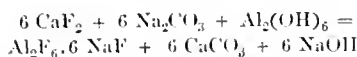
Sodium fluoride melted with aluminium chloride forms cryolite and sodium chloride (G. Forster, this Journal, 1888, 851); melted with sulphate of alumina, cryolite and sodium sulphate result (Winkler); with aluminium hydrate, sodium fluoride forms cryolite and sodium aluminate,  $3\text{Na}_2\text{O} \cdot \text{Al}_2\text{O}_3$  (Schuch); this, when extracted with water and treated with carbon dioxide, yields the hydrate of aluminium and sodium carbonate. Aqueous solutions of sodium fluoride and aluminium sulphate when mixed together form a fluorine aluminium hydrate, and this, when fused with sodium fluoride, also yields cryolite.

Magnesium salts behave in a similar manner to aluminium salts towards sodium fluoride. Aqueous solutions of sodium fluoride and magnesium sulphate give an amorphous precipitate of fluorine magnesium hydrate; when boiled with water and magnesite the analogue of cryolite ( $3\text{NaF} \cdot \text{AlF}_6$ ) is formed— $3\text{NaF} \cdot \text{Mg}_2\text{F}_6$  (Tissier). Magnesium sulphate melted with sodium fluoride forms magnesium cryolite,  $6\text{NaF} \cdot \text{Mg}_3\text{F}_6$ —a crystalline powder—the crystalloids being regular cubes. This body behaves like cryolite towards acids; treated with caustic soda magnesium hydrate is separated whilst cryolite dissolves completely.

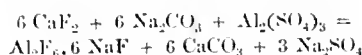
In commenting on the possibility of replacing natural cryolite by artificial cryolite or by this magnesium cryolite, the author points out the advantage of the last when compared with the artificial cryolite in point of price, owing to the magnesium salts being cheaper than those of aluminium; further, that a cheap method of preparation of sodium fluoride is what is really required to make either process a success.

The direct preparation of cryolite according to either of the following equations is possible, but the reaction is incomplete and the mass gets thicker and thicker as the quantity of calcium carbonate formed increases:—

I.



II.



—C. A. K.

#### *The Soda Industry in the Year 1889.* A. Hasenclever. Chem. Ind. 12, 431—436.

BITTER competition among German manufacturers engaged in the soda industry has made business profitless to all. Repeated attempts to form a combination have failed through the pretensions claims of individual producers. The fact that in 1888 the exports, reckoned on 100 per cent. carbonate of soda, exceeded the imports by 18,000 tons is no criterion of the general sound development of the industry, and profitable working has occurred in only exceptional cases. A sound basis for a lasting export trade in soda is lacking in Germany. A superficial comparison of the more favourable conditions that obtain in England, with her extended water highways and over-sea communications, shows the great disadvantages under which Germans have to compete in all industries which depend upon the bringing of various raw materials of small value to one point. The low railway tariff in England, arising from the competition of private railway companies, gives a great advantage to English makers, whilst the German State railways maintain a high tariff. Were there cheap carriage of fuel alone in Germany, great benefit would follow, as industries would then be carried on more generally throughout the land instead of being concentrated in the vicinity of the coal-fields.

The soda industry has grown in England, as it has in Germany, from year to year, during the last decade. In England there were used in this manufacture 568,512 tons of salt in 1878, and 781,348 tons in 1888. The increased production in England is entirely due to the manufacture of ammonia-soda. Many of the smaller Leblanc-soda makers have stopped their works, whilst the larger ones have gone on as before, and have reaped a moderate profit by the aid of a combination to limit the production of bleaching powder. The production of ammonia-soda in England has been limited to about one-fifth of the total make of soda, and the price obtained has enabled the firm of Brunner, Mond, and Co., to divide a dividend of 25 per cent.

In France, where, owing to trade combination and the high import duties, the price of soda has been the highest for many years, there seems to be a falling off in demand. The Leblanc-soda makers have suffered most, as the ammonia-soda makers are guaranteed the sale of a fixed amount.

In Austria, a combination among soda manufacturers has reduced the yearly make of all the works, and there would seem to be in that country a prospect of future stability were it not that German competition appears to preclude any material rise in price.

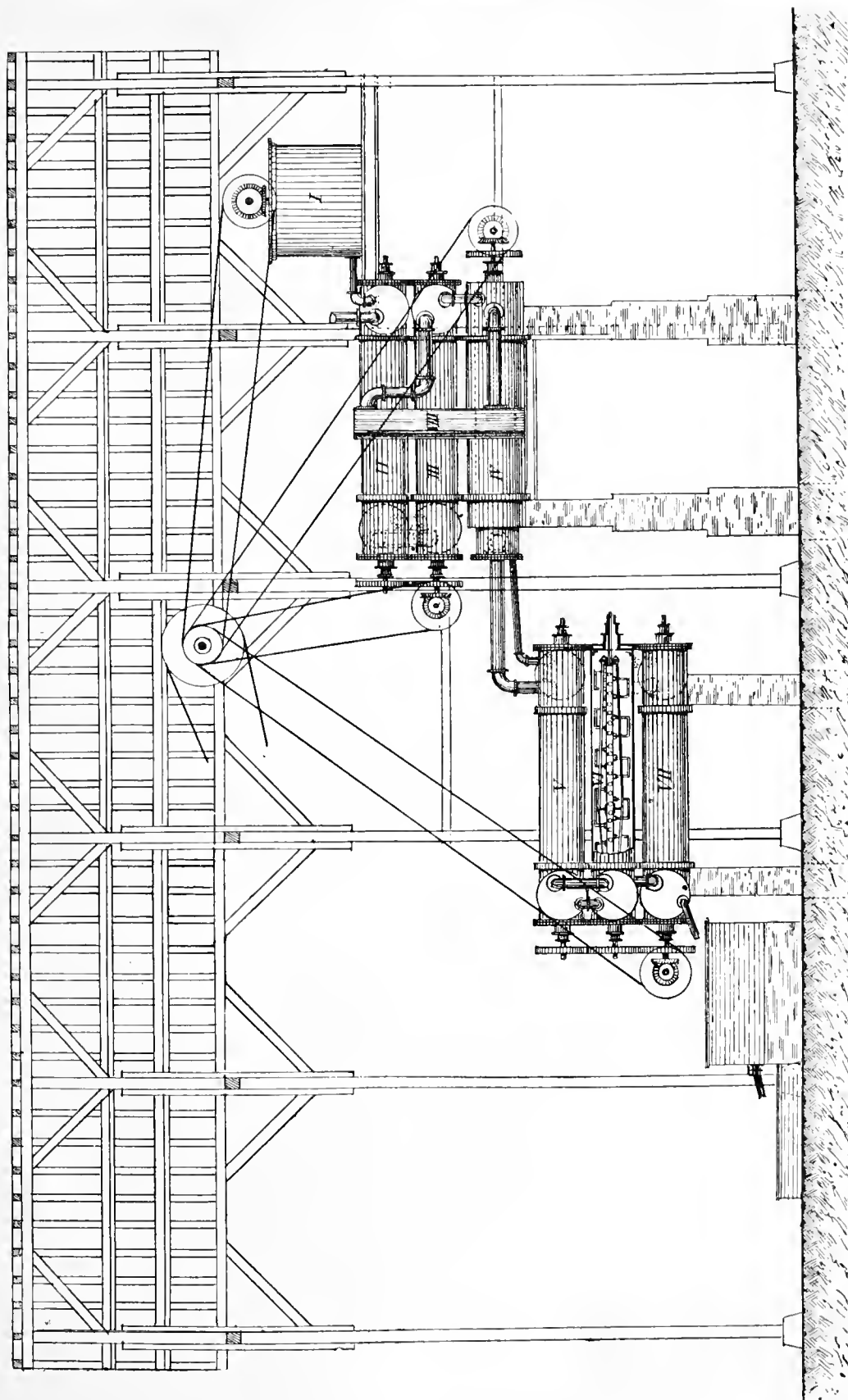
If, under such unfavourable conditions, some makers have carried on their works at a profit, this has been due either to an increase in the value of by-products, or to the working of other branches of manufacture which permit of being carried on in connexion with the soda process.

After drawing attention to Mond's method of recovering ammonia and tar from fuel as carried out at Northwich (this Journal, 1889, 505), the author goes on to describe other processes that have been adopted in connexion with soda works.

The "Chemische Fabrik Rhebania" has been making sulphuric acid from zinc blende, in connexion with spelter works at Stolberg, Oberhausen, Hamborn, Bensberg, and Dortmund. Ten years ago 12,000 tons of sulphuric acid, 60° B., were made, and in 1888 the quantity reached 51,010 tons.

W. Grillo has worked out a process for making liquid sulphurous acid from blende, and at Lipine, in 1888, 1,141 tons of liquid sulphurous acid were thus made and found a ready sale enclosed in iron vessels; but in Silesia the process has not been profitable.

Since the consumption of sulphuric acid increases year by year, the author thinks that both in Silesia and Belgium, in the course of time, all zinc works will utilise the gases from blende roasters. One cause for an increased demand for sulphuric acid will be the increased manufacture of sulphate of ammonia from the ammonia recovered from fuel and from coke-ovens. In Silesia 120 new coke-ovens adapted to this purpose were erected in 1889.



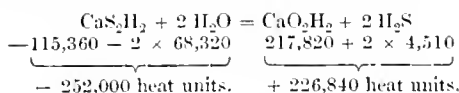
Efforts on the part of ammonia-soda makers to combine the manufacture of chlorine with the ammonia-soda process have so far led to no practical results. In view of the present value of hydrochloric acid, the author has been of opinion that up to the present day in Germany, the Leblanc-soda manufacturer has had an advantage over the ammonia-soda maker, but owing to the enormous rise in the price of fuel and the fact that twice as much fuel is used in the Leblanc process as in the ammonia-soda process for a given weight of soda, he thinks that in 1890 the Leblanc-soda maker will be at a disadvantage. The increased price of salt will affect the old process more unfavourably than the new, since the ammonia-soda makers usually have their own brine. The value of bleaching powder and hydrochloric acid is also likely to fall, in view of the manufacture of hydrochloric acid and chlorine from magnesium chloride, the introduction of electrolytic methods of bleaching and the making of chlorine by Campbell and Boyd's process now being worked out at the North British Alkali Works in Glasgow (Eng. Pats. 10,187, July 13, 1888, and 18,056, December 11, 1888; this Journal 1889, 983).

Reference is next made to Chance's sulphur-recovery process (this Journal 1888, 162) and to the patents of Gossage, Rawes (Eng. Pat. 1393, March 22, 1882; this Journal, 1882, 454), and Opl (Ger. Pat. 23,142, October 8, 1882; this Journal, 1882, 452), and the author then proceeds to describe experiments that he has carried out on a manufacturing scale with the v. Miller and Opl process for recovering sulphur or sulphuretted hydrogen from soda-waste.

According to the method of von Miller and Opl (Eng. Pat. 2334, January 29, 1884; this Journal, 1884, 373, 393, 551) calcium sulphhydrate is decomposed by steam instead of by carbonic acid, as in Chance's patent.

The following considerations among others induced the author to attempt to carry out in a large scale the v. Miller and Opl process in preference to that of Chance:—1. By the v. Miller and Opl process, pure sulphuretted hydrogen is formed instead of a gas containing 33 per cent.  $H_2S$  as in Chance's process. 2. Calcium hydrate is formed in the former process, for which a use can be more easily found than for the calcium carbonate formed in the latter process. 3. The employment of lime kilns and blowing engines is avoided, and the employment of boilers for generating steam is a cheaper plan of working.

The heat required to carry out the reaction was estimated theoretically as follows:—



That is to say, for the recovery of every 64 kilos. of sulphur from  $\text{CaS}_2\text{H}_2$ , 25,160 heat units must be supplied to complete the reaction, or for every 100 kilos. of S, 40,000 heat units are needed. Supposing that 1 kilo. of coal yields 5,000 heat units, then 8 kilos. of coal would be required to recover 100 kilos. of S. This amount left a wide margin for practical working. The apparatus consisted of horizontal wrought-iron cylinders, fitted with suitable appliances for agitating their contents. The accompanying Figure shows the arrangement.

The soda-waste is mixed with water in the mixing apparatus I., and passes thence into the cylinders II. and III., where it is subjected to the action of sulphuretted hydrogen, whereby calcium sulphhydrate is formed. The cylinder IV. is surrounded by a steam-jacket. In this cylinder the sulphhydrate is warmed up and then passes into the three lower cylinders V., VI., VII., where it is decomposed by steam. The steam enters the lowest cylinder VII. first, and then passes through into the two upper ones. The evolved  $H_2S$ , together with the excess of steam, then leave V. by a pipe and pass into the jacket round cylinder IV., where they warm up the calcium sulphhydrate contained in the cylinder. From the jacket the  $H_2S$  and uncondensed steam next pass into a condenser VIII. cooled with water, where the rest of the steam condenses, and the  $H_2S$  is then passed into the cylinders III. and II. Here half the  $H_2S$  is used to form

calcium sulphhydrate,  $\text{CaS} + \text{H}_2\text{S} = \text{CaS}_2\text{H}_2$ ; the other half passes from the apparatus II. to be made further use of:—



The experiments that have been carried out show that the conversion of all the  $\text{CaS}$  into  $\text{CaS}_2\text{H}_2$  is easily effected in the absorption cylinders, and that heat is evolved in the reaction, so that it is necessary to cool these cylinders with water. A liquor of  $28^\circ \text{B.}$  is easily obtained containing 212 grms. of sulphur per litre. The decomposition of the sulphhydrate by steam only takes place rapidly enough, however, when the solution is concentrated. The further the decomposition has been carried the slower it goes on. The author intends to publish a more detailed account of the experiments later. They have indeed shown that it is possible to recover nearly all the sulphur as  $H_2S$  by this method, but the quantity of coal used in generating the necessary quantity of steam was 90 times the theoretical quantity. A modified apparatus reduced this amount considerably, but nevertheless the reaction was too slow. He does not yet consider the experiments as completed, but does not think the future prospect is promising for the process.

The fact that by this process pure  $H_2S$  is obtained is on closer consideration not a great advantage, except that gasometers are not needed for collecting it. This is obvious from the fact that the gas from Chance's process, which contains 33 per cent.  $H_2S$ , is quite suitable for burning to make sulphuric acid, and contains only about the same quantity of air as would be consumed in burning pure  $H_2S$ .

The question of using the  $H_2S$  for making S is one that must be considered, not on the basis of present prices, but in view of a considerable fall in price, due to the competition which would ensue were all Leblanc-soda makers to recover the sulphur from their waste. England alone would produce 142,000 tons, whereas her import of sulphur is now only 32,000 tons.

The cost of plant for working up the waste in a works using 1,000 tons of salt per week is reckoned in England at 1,000,000 marks. In that country the calcium carbonate produced (which is too fine for use in making soda) can be disposed of for cement-making more easily than in Germany, owing to the higher cost of carriage in the latter country. Moreover, English works are generally very limited in space, and have to pay a good deal to get the waste removed. Finally, taking into account the fact that, whilst in England to make 100 kilos. of S, 0.38 marks are reckoned for coal, whilst Germans have to pay twice that sum, it can easily be understood why German soda-makers are chary of setting up sulphur-recovery plant, whilst on the other hand in England there are three works already working the process, and many others engaged in erecting the necessary plant.—H. S. P.

*On the Proportion of Water to Hydrochloric Acid formed by igniting Mixtures of Hydrogen, Oxygen, and Chlorine.* P. Hautefeuille and J. Margottet. Compt. Rend. 109, 641—643.

This research is regarded as preliminary to an investigation of the decomposition of water by means of heat in the presence of chlorine and carbon monoxide.

In a first series of experiments, a known volume of chlorine was added to a given amount of gas obtained by the electrolysis of water. In a second series, a known volume of oxygen was added to the elements of hydrochloric acid. The mixtures were exploded in a thick glass tube containing about 80 cc. by means of a spark. The apparatus is not described in detail.

The pressures of the gases and the amount of chlorine before and after the explosion were determined, the residual chlorine being estimated by means of a solution of sodium arsenite.

These data enable one to deduce from them the amounts of water and of hydrochloric acid formed.

Let  $h$  be the pressure of the hydrogen,  $H$  the pressure of the chlorine, these being introduced into the eudiometer at the same temperature.

$p$  the weight of hydrogen,  $P$  the weight of chlorine introduced.

$p^1$  the weight of hydrogen which unites with chlorine during the experiment.

$p^1$  the weight of chlorine which remains uncombined.

We have—

$$P = p \times \frac{0.069}{2.44} \times \frac{h}{H}.$$

#### SERIES I.—SYNTHESIS OF WATER IN THE PRESENCE OF CHLORINE.

Composition of the gaseous mixture.	Vols. of O	$\frac{1}{2}$	$\frac{1}{4}$	$\frac{1}{2}$	$\frac{1}{2}$	$\frac{1}{2}$	$\frac{1}{2}$	$\frac{1}{2}$	$\frac{1}{2}$	$\frac{1}{2}$
	Vols. of H	1	1	1	1	1	1	1	1	1
	Vols. of Cl	$\frac{1}{2}$	$\frac{1}{4}$	$\frac{1}{2}$	1	$\frac{3}{2}$	$\frac{1}{2}$	$\frac{1}{2}$	$\frac{1}{2}$	2
Value of $\frac{p-p^1}{p^1}$ .....		$\frac{5}{4}$	$\frac{1}{1.8}$ to $\frac{1}{2.9}$	$\frac{1}{2.3}$ to $\frac{1}{2.5}$	$\frac{1}{3.8}$ to $\frac{1}{4.3}$	$\frac{1}{5.2}$ to $\frac{1}{5.7}$	$\frac{1}{9.2}$ to $\frac{1}{11.2}$	$\frac{1}{20}$	$\frac{1}{40}$	0

(1.) As soon as the volume of chlorine is greater than half the volume of hydrogen contained in the electrolytic gas,  $\frac{p-p^1}{p^1}$  is less than unity, and diminishes rapidly with the increase in the proportion of chlorine, so that when

Since  $p^1$  is the weight of hydrogen which unites with chlorine, it is evident that  $p - p^1$  = the weight of hydrogen which unites with oxygen and

$\frac{p-p^1}{p^1} = \frac{\text{number of half molecules of water}}{\text{number of molecules of hydrochloric acid}}$  formed during the experiment.

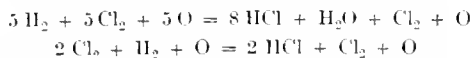
The authors found that  $\frac{p-p^1}{p^1}$  does not vary with the pressure between the limits of  $\frac{1}{3}$  and  $\frac{2}{3}$  of an atmosphere, nor with the nature of the spark.

#### SERIES II.—SYNTHESIS OF HYDROCHLORIC ACID IN THE PRESENCE OF OXYGEN.

Composition of mixture	Vol. of Cl .....	1	1	1	1	1	1	1
	Vol. of H .....	1	1	1	1	1	1	1
	Vol. of O .....	$\frac{1}{2}$	$\frac{1}{2}$	$\frac{1}{2}$	$\frac{1}{2}$	$\frac{1}{2}$	$\frac{1}{2}$	3
Value of $\frac{p-p^1}{p^1}$ .....		$\frac{1}{7.2}$	$\frac{1}{4.3}$ to $\frac{1}{4.7}$	$\frac{1}{3.8}$ to $\frac{1}{4.3}$	$\frac{1}{3.4}$	$\frac{1}{3.1}$ to $\frac{1}{3.3}$	$\frac{1}{3.2}$	$\frac{1}{3.2}$

The ratio  $\frac{p-p^1}{p^1}$  is always less than unity, and varies but little when the ratio, by volume, of oxygen to hydrogen present is increased from  $\frac{1}{2}$  to 3.

The results may be put in the form of equations, e.g.:—



The temperatures to which these states of equilibrium correspond cannot be determined directly. The authors intend to estimate them by means of data furnished by Berthelot and Vieille, and also by some further experiments.

—P. J. H.

*On the Distribution of Hydrogen between Chlorine and Oxygen.* H. Le Chatelier. *Compt. Rend.* **109**, 664–667.

THE experiments of Hauteville and Margottet (preceding abstract) on the distribution of hydrogen in presence of chlorine and oxygen supply important information respecting the general laws of chemical equilibrium. The author has been engaged upon similar researches (undertaken with the intention of applying the results to the Deacon process for the manufacture of chlorine), and points out how the value of the coefficients of distribution can be determined by calculation.

The formula for equilibrium in the case of a mixture of oxygen, chlorine, water, and HCl is—

$$\text{Loge } \frac{p(\text{O}_2) \times p^4(\text{HCl})}{p^{1/2}(\text{Cl}_2) \times p^{1/2}(\text{H}_2\text{O}_2)} + 500 \int \frac{LdT}{T^2} = \text{const.}$$

$L$  is the heat of reaction of 1 molecule of oxygen with 4 molecules of HCl; its value is 28 at ordinary temperatures, and does not vary much up to 1,000°. The value of the constant depends upon the composition of the mixture in equilibrium; its normal value in the Deacon process is 15. In order to apply the formula it is necessary to express the pressures as functions of the number of molecules of each substance.

If we denote these as follows:—

Substance .....	$\text{H}_2$	$\text{O}_2$	$\text{Cl}_2$	$\text{H}_2\text{O}_2$	HCl	$\text{N}_2$
Number of molecules	1	$n$	$n'$	$x$	$2(1-x)$	$N$

and further denote the pressure (in atmospheres) of the mixture by  $P$ , the formula becomes—

$$\text{Loge } P + \text{loge } \frac{16(2n-x)(1-x)^4}{(2n+2n'+2N+2-x)(n'-1+x)^2x^2} + \frac{1,400}{T} = 15.$$

This formula applies up to 1,000°, but is not rigorously correct at higher temperatures when the dissociation of the chlorine molecule becomes appreciable. The hygrometric state, which is of importance, has not been indicated in the experiments of Hauteville and Margottet. We must therefore assume that the gases were saturated with water-vapour at 15°, i.e., that they contained 2 per cent. by volume. The temperature of the mixture corresponding to the state of equilibrium has not been (and cannot be) determined. It depends upon the relative rapidities of the chemical reactions and the cooling effect. The former increases rapidly with the temperature, being practically zero at 500° and only acquiring a noticeable value above 800°. We can only suppose that the temperature in question is higher than 1,000° and lower than 2,000°.

*Influence of Temperature.*—The proportion of water produced ought to decrease as the temperature increases. The following table, calculated from the formula, gives the number  $x$  of molecules of water produced per molecule of hydrogen in a mixture containing equivalent proportions at the atmospheric pressure—

$t$ .....	0°	300°	400°	650°	1,120°
$x$ .....	0.098	0.90	0.80	0.50	0.20

The oxidation of HCl should therefore be practically complete at ordinary temperatures. This has already been proved by M. Berthelot.

**Influence of Pressure.**—The formula indicates a diminution in the proportion of water when the pressure diminishes. This diminution is small, because the change of volume accompanying the reaction is itself small. It must however occur, if the molecular volumes assigned to the various substances are correct, for it follows directly from the second law of thermodynamics. On the other hand the experiments of MM. Hautefeuille and Margottet show that change of pressure produces no effect. This shows that the molecule of chlorine is, under the given conditions,

partially dissociated; if it were completely dissociated the diminution of pressure would, on the contrary, increase the proportion of water produced.

**Influence of the Relative Proportion of Elements present.**—This is the most interesting point, for it enables us to test the correctness of the formula. The following table gives the calculated proportions of water produced from a mixture containing equivalent proportions, and also those obtained by MM. Hautefeuille and Margottet, allowing for 2 per cent. of water-vapour by volume:—

Cl <sub>2</sub>	1	1.25	1.50	2	1	1	1	0.5
H <sub>2</sub>								
O <sub>2</sub>	0.5	0.5	0.5	0.5	0.25	1.25	3	0.5
H <sub>2</sub>								
H <sub>2</sub> O <sub>2</sub> {calculated.....}	0.258 {	0.15	0.07	0.06	0.19	0.25	0.27	0.63
H <sub>2</sub> {observed.....}		0.148	0.08	0.07	0.215	0.275	0.31	0.58

The agreement between the calculated and observed values is thus seen to be most satisfactory; it is even better than could have been expected, inasmuch as no allowance is made for possible dissociation of chlorine. The action of the excess of chlorine or oxygen depends, therefore, solely upon the relative volumes of the bodies present, and is in no way dependent upon their special chemical properties, for these do not enter at all into the formula.

The distribution of oxygen between carbon monoxide and hydrogen may be similarly represented—

$$\text{Log} \epsilon \frac{p(\text{C}_2\text{O}_2) \times p'(\text{H}_2\text{O}_2)}{p''(\text{H}_2) \times p'''(\text{C}_2\text{O}_4)} + 500 \int \frac{L dT}{T^2} = \text{const.}$$

or, neglecting the variation of L with temperature, and using the following notation for a mixture containing a semi-molecule of displaceable oxygen—

Substance..... CO<sub>2</sub> H<sub>2</sub> H<sub>2</sub>O<sub>2</sub> C<sub>2</sub>O<sub>4</sub>  
Number of molecules  $n$   $n'$   $x$   $1-x$ .

$$\text{Log} \epsilon \frac{(n-1+x)x}{(n'-x)(1-x)} + \frac{5000}{T} = \text{const.}$$

In this case it will be observed that the state of equilibrium is strictly independent of the pressure.—D. E. J.

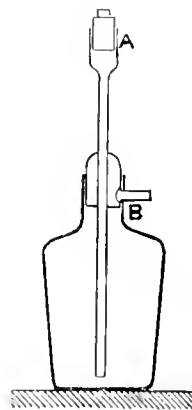
#### *The Preparation and Properties of Anhydrous Platinum Difluoride.* H. Moissan. Compt. Rend. 109, 807—809.

This body is prepared by passing fluorine over a bundle of platinum wires heated to about 500°—600° C., and placed in a stout platinum tube. If no hydrofluoric acid be present no reaction takes place at all up to 100° C. At ordinary temperatures platinum itself is entirely unacted upon by fluorine. A mixture of fluorine and hydrofluoric acid, however, is more energetic in its action than fluorine alone. The fluoride is obtained as a fused mass of a deep reddish colour or as small yellow crystals much resembling those of the dichloride. It is exceedingly hygroscopic and its reactions with water are very characteristic. If placed in a little water it dissolves, but the solution becomes hot and the hydrated oxide is then precipitated. With a comparatively large quantity of cold water the decomposition is retarded, but on warming the salt is at once decomposed. Its peculiar behaviour with water explains the fact long ago observed that the anhydrous difluoride cannot be obtained in the wet way, for, as seen, it rapidly decomposes water to form hydrofluoric acid and the brownish-yellow precipitate recalls to mind Frémy's hydrated dioxide PtO<sub>2</sub>.H<sub>2</sub>O<sub>2</sub>. When raised to a bright red heat the fluoride readily splits up into fluorine and platinum, the latter presenting a distinctly crystalline appearance.

The salt was analysed by dissolving a portion in a large quantity of water, then pouring off from any slight insoluble residue and boiling the clear solution. On drying the precipitate and igniting, the weight of platinum thus obtained gave the formula PtF<sub>2</sub> for the new compound.—S. G. R.

#### *Further Researches on the Preparation and on the Density of Fluorine.* H. Moissan. Compt. Rend. 109, 861—864.

THE fluorine was obtained by the electrolysis of pure hydrofluoric acid, in quantities of about 100 cc. at a time, contained in the platinum apparatus similar to the one formerly described but of a capacity of about 160 cc. Lest the fluorine should carry over with it any traces of hydrofluoric acid, the products of electrolysis were passed through a platinum spiral placed in a bath of methyl chloride giving a temperature of about -50° C. or 70° C. below the boiling point of the acid. The gas then passed over sodium fluoride and, being considered pure, was led to the apparatus shown in the Figure.



This was a platinum flask of a capacity of about 100 cc. and weighing about 70 grms., and closed by a rounded perforated stopper accurately fitted to its seat and serving also as a stopcock. The flask was first filled with pure dry nitrogen and this in turn displaced by the fluorine which was passed in by a platinum tube fitting into A. After that silicon inflamed readily in the issuing gas the current was still continued for five minutes. The stopper was then turned round on itself and the plug being introduced into the tube A, all connexion with the exterior was cut off. The flask was then weighed on a balance turning readily with 0.0005 gm. and its capacity was also determined. From these data the density of fluorine was deduced as (1) 1.262; (2) 1.265; (3) 1.270, the figure adopted being 1.265. The theoretical density of fluorine is  $0.06927 \times 19 = 1.316$ . As was the case with phosphorus trifluoride the observed density is rather low, hence it seems possible that the accepted atomic weight of fluorine, namely 19, may be rather too high.—S. G. R.

*The Colour and Spectrum of Fluorine.* H. Moissan.  
Compt. Rend. 109, 937—940.

By analogy it was considered likely that fluorine would possess very little if any colour, since it stands first in the halogen family and is followed by chlorine, possessed of no great depth of colour in comparison with the highly-coloured bromine and still more highly-coloured iodine. A tabular apparatus of platinum was used for the observation, and care was taken that it was perfectly dry, since water is speedily decomposed by fluorine. On looking through a depth of 0.5 metre of fluorine, it was observed that the gas possesses a greenish-yellow colour much more faint than that of chlorine regarded through a tube of similar depth, and being besides a tint more closely approaching yellow than the greenish-yellow tint of chlorine. Examined through a depth of 1 metre, fluorine was not observed to possess any absorption bands. The following singular observation was made. When the platinum apparatus was filled with fluorine, a very small quantity of water was added, which immediately decomposed with formation of hydrofluoric acid and ozone. This latter gas was produced in a state of such concentration that the whole of the tube assumed the indigo-blue tint observed in ozone of Hautefeuille and Chapuis. After a few minutes, owing to the temperature of the laboratory, the ozone was partly destroyed and the colour reduced; it thus gradually faded and at length disappeared. The spectrum of fluorine was also determined, and a large number of bands, upwards of 13, all in the red part of the spectrum, were found.

*A New Process for the Utilisation of Atmospheric Oxygen and the Compounds upon which the Process is Based.*  
G. Kassner. Dingl. Polyt. J. 274, 136—142; 183—190; 226—231; 270—276.

*Preparation and Properties of Compounds Used.*—

The author found in 1884, when barium manganate was boiled with oxidisable bodies in aqueous solution it parted with its oxygen to these and was converted into a brown substance of the formula  $MnO_3Ba$ , and that this on heating in air took up oxygen and was reconverted to barium manganate. Several drawbacks attended its use; amongst others, in presence of water barium hydrate was formed which rapidly absorbed carbonic acid, and so an increasing amount of barium carbonate was contained. The regeneration, moreover, was very slow.

The author working on the subject has produced three new compounds, and describes their preparation, properties, commercial application, and chemical relationship.

*Barium Plumbate*,  $Ba_2PbO_4$ .—Barium hydrate and lead oxide were heated in a porcelain crucible over a Bunsen flame; a deep black substance was obtained. Dilute hydrochloric acid added to a portion caused effervescence as of carbonic acid; there was a strong odour of chlorine, and a yellow-green solution remained. Dilute nitric acid separated a brown powder. Sulphuric acid was without action until oxalic acid was added, when barium and lead sulphates were formed and carbon dioxide evolved. When oxalic acid was used with nitric acid a clear solution of the respective nitrates was obtained.

By mixing one equivalent of lead oxide and one equivalent of barium carbonate and heating as before, a glassy substance was produced, the colour deepening until quite black. It was powdered, and on testing showed by action of acids that carbonic acid still remained, that the whole of the lead oxide has not been converted into the dioxide, and that all the displaceable oxygen could not be obtained. The presence of lead oxide was proved by adding nitric acid and testing with sulphuretted hydrogen. The displaceable oxygen was determined by mixing three parts of the product with one part of pure oxalic acid and warming with dilute nitric acid till evolution of carbonic acid ceased, when the excess of oxalic acid was determined by titrating back with potassium permanganate,  $H_2C_2O_4 + 2H_2O + O = 2CO_2 + 3H_2O$ . Nitric acid is preferable for the determination, sulphuric acid causes separation of the sulphates, and acetic acid is unreliable.

The displaceable oxygen was found by two tests as 2.32 and 2.59 per cent.

The true barium plumbate was obtained by heating two equivalents of barium carbonate and one of lead oxide. The product was deep black, and contained only slight traces of carbonic acid and lead oxide. The displaceable oxygen was found to be 2.86; theoretically it should have been 2.94. Hypothesis points to a *plumbic acid*,  $H_4PbO_6$ , the anhydride being  $PbO_3$ .

*Strontium plumbate*,  $Sr_2PbO_4$ .—Two equivalents of strontium carbonate and one equivalent of lead oxide treated in a similar way gave a deep-brown chocolate-coloured compound showing the same reactions.

*Calcium plumbate*,  $Ca_2PbO_4$ .—This was made by heating two equivalents of calcium carbonate and one of lead oxide. A lesser heat was required than in case of the other two, and the conversion was effected in about five minutes, over the Bunsen flame. The compound was of a yellowish-red or flesh colour. The abundance and cheapness of calcium carbonate should render this the cheapest of the three compounds.

The properties of the three plumbates were similar, and all were insoluble in water. Examined microscopically, the last-mentioned was seen as an aggregate of yellowish crystals. On treatment with sodium bicarbonate, opaque brownish-black masses ( $PbO_2$ ), having between them colourless crystals ( $CaCO_3$ ), were obtained. The action of acids is similar on all three; nitric acid forms the brown lead dioxide and a nitrate of the alkaline earth. Dilute sulphuric acid gives also lead dioxide and a sulphate of the alkaline earth, though strong acid forms both sulphates. Dilute acetic acid has no action in the cold; on heating, dioxide and the acetate are formed. Hydrochloric acid dissolves them to a yellow-green liquid. To obtain a clear solution, nitric or acetic acids may be used with oxalic acid, or, preferably, hydrogen peroxide.

Carbonic acid or bicarbonates produce carbonates of the alkaline earths and lead dioxide. Sodium bicarbonate acts better than the potassium salt.

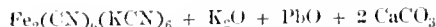
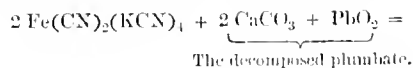
The author found that 350 grms. of calcium plumbate and 400 grms. of potassium bicarbonate in a little water, heated on the water-bath to the boiling point, gave a clear brown liquid deepening to black. The heavy powder, a mixture of lead dioxide and the corresponding carbonate, settled to the bottom, and on being dried and ignited formed again the plumbate. The soluble alkaline salts are easily removed by decanting and washing, and the alkaline carbonate can be obtained as a by-product.

Water alone, at a temperature of 150 °C., under increased pressure, caused formation of the dioxide and the corresponding alkaline hydrate. Ammonium mono- and bicarbonates gave ammonia, carbonates of the alkaline earths and lead dioxide. Ammonium nitrate and acetate gave ammonia, lead dioxide, and the respective salts of the alkaline earths.

*The Technical Application of these Compounds.*—*Calcium plumbate* is most to be preferred, being not only the easiest and cheapest to produce, but because it contains proportionately the highest percentage of displaceable oxygen, viz., 4.56 per cent. It may be used directly, as in the glass industry, for production of lead and lime glass.

Its widest application is, however, indirectly by decomposition and formation of lead dioxide. This can be accomplished, as already noted, in many ways, e.g., by water or steam under increased pressure, by carbon dioxide, mono- or bicarbonates, ammonium salts, or, lastly, by strong acids, such as acetic, nitric, sulphuric, &c.

The oxidation of potassium ferrocyanide (yellow prussiate) to potassium ferricyanide (red prussiate) is rapidly and completely accomplished by means of *calcium plumbate* and carbon dioxide.



In the presence of free caustic alkali the reaction is not complete, and excess of free alkali causes the re-formation of lead dioxide. The process is, however, cheaper than



that by means of chlorine, for the more valuable potassium carbonate is obtained instead of the chloride. Then the insoluble residue is easily separated, washed, and by subjection to a red heat in presence of air, *calcium plumbate* is regenerated.

Potassium ferrieyanide with caustic alkali is known as an excellent oxidising agent, readily oxidising lead oxide to the dioxide, chromic hydroxide to chromic acid, and manganese sesquioxide to manganese dioxide. It has recently received wide application in organic chemistry; such a solution bleaches vegetable fibre as strongly and quickly as hydrogen peroxide (compare this Journal, 1890, 108). The price has hitherto been an obstacle. This objection is now removed, because by simply boiling the reduced solution with the decomposed plumbate, and introduction of carbon dioxide, ferrieyanide is regenerated, and can thus be used again and again.

For the production of oxygen on the large scale the author recommends treating the plumbate with sodium bicarbonate, decanting off the soluble alkali, washing the insoluble residue, pressing into lumps, and drying these in the generating apparatus with superheated steam; as soon as dry, the steam is stopped, and, all air having thus been expelled, heat is applied, when the oxygen is freely given off.

The application of the barium and strontium plumbates is limited on account of the greater cost, the stronger heat required in formation, and the proportionately less amount of displaceable oxygen contained.

Although in the carefully prepared plumbates the lead oxide is entirely converted to plumbic acid, yet this is not so on the large scale, and therefore the alkali obtained (as a by-product) may contain lead in solution. The presence of so poisonous a substance in a compound having such wide applications is highly obnoxious, and care must be taken to eliminate it. This, however, is easily accomplished by means of hydrogen sulphide or any soluble sulphide with which the solution should always be tested. Barium or strontium hydrates should be treated with their respective sulphides. Another method is by electrolysis, when the insoluble dioxide is readily deposited on the anode.

The author contrasts his process with the older methods of Boussingault, of Weldon, and that of Brin's Oxygen Company.

He contends that barium dioxide becomes gradually more or less inert from formation of barium carbonate, and that it requires an extremely high heat to expel the oxygen. That by means of calcium plumbate pure oxygen is obtained; that the plumbate continues unaltered—that is the  $\text{CaCO}_3$  and  $\text{PbO}$  remain in same proportions—and that the much lower heat at which oxygen is given off would mean the use of considerably less fuel. Further, it has a higher specific heat, and although it is heavier in proportion, yet a greater percentage of oxygen is obtained by use of calcium plumbate, while there is the production as a by-product of the alkaline carbonate.

For the much-spoken-of production of chlorine from magnesium chloride, the following simple process is applicable: (1) mixing with the magnesium salt; (2) drying to expel the water of crystallisation; (3) granulating and sifting the hard mass; (4) heating it at a high temperature; (5) collecting or absorbing the chlorine given off.

The author also shows the superiority of the plumbates to the barium manganate before described.

#### *Chemical Relations of the New Bodies.*—

Frémy (Ann. Chim. Phys. 12, 409) prepared a compound by mixing lead oxide and potassium hydrate in a little water and crystallising over sulphuric acid. It was obtained in colourless crystals of composition  $\text{K}_2\text{PbO}_3 + \text{H}_2\text{O}$ . Again, W. Crum (Ann. 55, 212) prepared an almost colourless compound of calcium and lead dioxide by mixing lead nitrate, milk of lime, and calcium chloride solution and heating for some time at 160° F. The formula assigned was  $(\text{CaO}_2)\text{PbO}_2$ .

There would seem to be two hypothetical acids—

*Ortho-plumbic acid*,  $\text{Pb}(\text{OH})_4$

*Meta-plumbic acid*,  $\text{PbO}(\text{OH})_2$

The above bodies,  $\text{Ca}_2\text{PbO}_4$ ,  $\text{Sr}_2\text{PbO}_4$ , and  $\text{Ba}_2\text{PbO}_4$  would thus be *ortho-plumbates*. Red lead might be  $\text{Pb}_2(\text{PbO}_3)$  (see this Journal, 1889, 468).

The *meta-plumbate* being the  $\text{K}_2\text{PbO}_3$  prepared by Frémy, and also by Geuther (Annalen, 219, 68 and 69)—the latter by passing oxygen into a solution of oxide of lead in fused potassium hydrate—the sesqui-oxide may be written  $\text{Pb}(\text{PbO}_3)$ . The author concludes by pointing out that  $\text{PbO}_2$  thus acts similar to  $\text{SnO}_2$  and  $\text{CO}_2$ , and indicating the correspondence in the periodic system of carbon, silicon, zinc and lead.—D. A. S.

#### PATENTS.

*Improvements in the Treatment of Gases and Recovering of Sulphur from Sulphuretted Hydrogen evolved in various Manufacturing Processes.* J. Barrow, Manchester. Eng. Pat. 17,528, December 1, 1888. 8d.

THE gases are deprived of sulphuretted hydrogen by treatment in a scrubber with a solution of perchloride of iron or manganese or both. The precipitated sulphur is collected by settling and filtration. The reduced solutions are regenerated by treatment with peroxides of iron or manganese or with heated air. Plant for conducting the process is described.—G. H. B.

*Improvements in the Manufacture of Chlorine and Chloride of Lime and in Apparatus therefor.* Solvay and Co., Brussels, Belgium. Eng. Pat. 18,574, December 19, 1888. 6d.

A MODIFICATION of a process embodied in several previous patents for the production of chlorine from calcium chloride by heating it with siliceous and aluminous materials. When clay is employed it is found desirable to calcine and powder it before admixture with calcium chloride, in order to drive off combined water, which, if present, would favour the production of hydrochloric acid rather than of chlorine in the subsequent reaction. Hitherto the furnacing of the mixture has been conducted in retorts heated externally, in order to avoid contact with combustible gases containing carbonic oxide, which has a great tendency to combine with the chlorine to form carbonyl chloride, from which chlorine cannot be again recovered in its active form. The amount of fuel required by this method of heating in closed retorts is obviously very considerable, and the chief object of the present patent is to provide a method of direct heating in which the formation of carbonyl chloride shall be reduced to insignificant proportions. To this end the decomposition is effected in a high tower of thick masonry filled with the material to be treated. Gases from a coke gasifier or gas generator are introduced through a ring of short branch pipes situated near the middle of the tower, whilst air is passed in at the bottom. The cold air becomes heated in its passage through the descending hot material until it meets with the generator gas, which is completely burnt in a short space above the supply pipes, termed the zone of combustion. Above this is the zone of decomposition, in which the hot gases containing an excess of air act on the descending material and produce an evolution of chlorine. The upper layers of material serve to abstract the heat of the gases in their passage through it. The tower thus serves as a regenerative element, cool gases leaving the top, whilst the exhausted material arrives cold at the bottom. The chlorine produced in this manner being contaminated with a considerable proportion of carbonic acid, is not suitable for the production of chloride of lime by the ordinary method. The gases are therefore passed into a solution of lime which absorbs all the chlorine, but only a part of the carbonic acid, and this part precipitates from the solution as carbonate of lime. The solution of chloride of lime is then decomposed by the hydrochloric acid which is unavoidably produced in small quantities in the principal reaction, and which is condensed from the chlorine simultaneously produced. Concentrated chlorine gas is thus obtained, which is then directed either upon lime in the ordinary way, or upon imperfect chloride of lime obtained with poor chlorine, and which may then be brought to the desired strength. The dilute chlorine mixed with carbonic acid may also be employed in the manufacture

of chlorates. This process and apparatus are not limited solely to the decomposition of calcium chloride, but are applicable also to the decomposition of magnesium chloride, and of chlorides generally by the dry process. (This Journal, 1885, 345 and 533; 1886, 451; 1887, 661.)—G. H. B.

*Improvements in or relating to the Manufacture of White Lead.* H. B. Condy, Battersea. Eng. Pat. 18,705, December 21, 1888. 6d.

See under XIII., page 199.

*Improvements in Reverting and Utilising By-Products such as are Formed in Processes for Obtaining Chlorine.* R. H. Steedman, Dalmaur, and A. J. Kirkpatrick, Glasgow. Eng. Pat. 18,921, December 28, 1888. 4d.

A SOLUTION of manganese sulphate is treated at a boiling temperature with magnesium carbonate, or magnesia and carbonic acid, manganese carbonate being precipitated and magnesium sulphate remaining in solution.

The manganese carbonate, after filtration and washing, is calcined for the production of oxide suitable for generating chlorine. The solution of magnesium sulphate is evaporated and heated with sodium or potassium chloride, hydrochloric acid being evolved. The residue, consisting of magnesia and sulphate of soda, is treated with water, which extracts sulphate of soda and leaves magnesia ready for use again.

—G. H. B.

*Improvements in the Method of Treating Alkaline Sulphides in Solution or an Aqueous Mixture of the same with Carbonic Acid Gas for the Production of Sulphuretted Hydrogen and in Apparatus therefor.* E. W. Parnell and J. Simpson, Liverpool. Eng. Pat. 19,023, December 31, 1888. 8d.

A SOLUTION or aqueous mixture of alkaline sulphides is charged into a series of receptacles so connected that the contents may be subjected at will to the passage of either of two streams of carbonic acid gas containing an admixture of nitrogen or other inert gas. It is found convenient to have nine receptacles in one system, eight being in operation whilst the ninth is being emptied and refilled. Each stream of carbonic acid is passed through four receptacles at the same time. The first action of the dilute carbonic acid on the alkaline sulphide produces a gas containing very little sulphuretted hydrogen, and this is allowed to escape in any suitable way. As soon as sulphuretted hydrogen is perceptible in the issuing gas, the first receptacle in the series of four forming the preliminary series is disconnected and caused to be the last of the other or final series, whilst a freshly charged receptacle becomes the last in the preliminary series. The other stream of carbonic acid acting on the contents of the final series, each element of which has previously formed part of the preliminary series, produces a gas rich in sulphuretted hydrogen, which is conducted to a gasholder. When the sulphuretted hydrogen in this gas falls below a certain minimum, the first receptacle in the series is disconnected for emptying and recharging. In this way a continuous stream of carbonic acid gas is entering the receptacles at two places and continuous streams of nitrogen and of gases containing much sulphuretted hydrogen are being evolved. Among the advantages of this method of carbonating is the uniform pressure of the gas consequent on the current of carbonic acid gas always passing through the same number of carbonators. The pressure and strength of the sulphuretted hydrogen gas is almost uniform, and the whole plant is kept constantly working except the outstanding carbonator which is being emptied and filled.—G. H. B.

*Improvements in Obtaining Chlorine and in Reverting and Utilising By-Products of the Processes.* R. H. Steedman, Dalmaur, and A. J. Kirkpatrick, Glasgow. Eng. Pat. 881, January 17, 1889. 6d.

COMMON salt, manganese peroxide, and sulphuric acid are mixed together in suitable proportions and furnace for the production of chlorine. The residue, consisting of the

sulphates of soda and manganese, is then dissolved in water and the solution is treated by either of the following processes:—

1. The solution is mixed with magnesium carbonate and heated to boiling, manganese carbonate being precipitated. The solution is evaporated so that the sodium sulphate will crystallise out, leaving the magnesium sulphate in the solution, which is then treated by the process described in Eng. Pat. 18,921 of 1888 (preceding column).

2. The solution is treated with ammonium carbonate, manganese carbonate being precipitated. The solution is evaporated to dryness and heated in contact with steam and carbonic acid; ammonia is evolved and the residue, consisting of sodium bisulphate, is used to decompose sodium chloride in the first operation. Instead of evaporating the solution to dryness, it may be heated with magnesium carbonate for the recovery of the ammonia, the solution of magnesium and sodium sulphates being worked up as before.—(G. H. B.)

*Improvements in the Manufacture of Salt and in Apparatus and Appliances employed therein.* O. E. Pohl, Liverpool. Eng. Pat. 1007, January 19, 1889. 8d.

THE claims in this patent are very numerous and cannot be described without the drawings. The chief feature in the process is the employment of two evaporating pans, one above the other, for the concentration of brine. The two longest sides of the top pan are prolonged downwards so as to dip into the brine of the bottom pan. The products of combustion from the furnace pass through the space thus enclosed, that is, they pass over the surface of the brine in the bottom pan and along the bottom of the top pan, which is suspended in position by rods fixed to overhead cross beams. The ends of both pans are bolted on to the brickwork of the furnace.—G. H. B.

*Improvements in and relating to the Production of Chlorine and to the Manufacture of Sulphate of Soda.* T. Parker and A. E. Robinson, Wolverhampton. Eng. Pat. 2310, February 9, 1889. 4d.

A SUITABLE vessel is divided into two halves by a septum or porous diaphragm. In the one division is placed a solution about half saturated, preferably with sodium chloride, and in the other a similar solution of some sulphate, such as ferrous sulphate. On passing an electric current through the solution, chlorine is evolved at the positive electrode and iron is deposited at the negative, whilst the sulphuric acid which is liberated unites with the soda to form sulphate of soda or salt cake. The chlorine evolved is collected and utilised for manufacturing bleaching powder or for other purposes. (Compare this Journal, 1889, 817, and 1890, 67)

—S. G. R.

*Improvements in Apparatus for the Production of Pyro-ligneous Acid.* J. H. du Vivier, Paris, France. Eng. Pat. 2569, February 13, 1889. 8d.

THIS is a continuous method of treating wood. The retorts are portable and are introduced into and withdrawn from the furnaces by means of specially constructed trucks. The furnaces are set back to back and the waste heat utilised for drying the tarry products. There are six claims made, and two sheets of drawings accompany the specification.—D. A. S.

*Improvements in the Manufacture of Chlorates, and of Magnesium Hydrate suitable for Use therein.* E. K. Muspratt, Liverpool, and G. Eschellmann, Northwich. Eng. Pat. 2786, February 16, 1889. 4d.

THE patentees, in the manufacture of chlorates, prefer to use magnesium hydrate rather than magnesia for the absorption of chlorine. The magnesium hydrate is prepared by treating the oxide or oxychloride, or a mixture of the two, with solutions of the chlorides of magnesium, calcium, strontium

and barium at a temperature of about 80° C. The hydrate formed, when washed and dried, is ready for use. This plan is adopted because the oxide alone does not hydrate itself readily when in contact with water (see this Journal, 1884, 319 and 445).—S. G. R.

*Improvements in obtaining Cyanides from Residuary Liquors formed in Chemical Processes.* J. S. MacArthur, Pollokshields. Eng. Pat. 3072, February 21, 1889. 6d.

THE cyanides may be present in the form of simple, double, or compound cyanides; if zinc be present this should be removed by the addition of ammonium sulphide. To the solution there is added enough acid to make it neutral. If it is alkaline from the presence of soluble carbonates, it can be, if required, made neutral by adding calcium chloride and boiling. To the neutral solution acid is added in sufficient quantity to decompose all the simple, double, and half the compound cyanides present. The solution is then suitably heated, and the evolved hydrocyanic acid absorbed by caustic alkali solutions. To the residual insoluble cyanide in the sludge caustic soda or milk of lime is added and the cyanide thus brought into solution, which after separating the insoluble portion may be either treated as before mentioned or evaporated down and crystallised.

—S. G. R.

## VIII.—GLASS, POTTERY, AND EARTHENWARE.

### PATENT.

*Improvements in the Manufacture of Articles of Pressed Glass.* T. Davidson, Gateshead-on-Tyne. Eng. Pat. 2641, February 14, 1889. 4d.

TO the materials generally used for making flint-glass, e.g., sand 560 lb., alkali 210 lb., nitrate of soda 84 lb., are added phosphate of lime 70 lb., "lime spar" 84 lb., and arsenic 35 lb., and the product melted and moulded in the ordinary way for making pressed glass. The articles formed are then exposed locally to a high temperature, when the parts most heated become opaque, those less so translucent, and those kept comparatively cool remain transparent.—B. B.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

### PATENTS.

*An Improved Mode of and Means for Manufacturing Blocks of Artificial Stone.* A. McLean, Brockley. Eng. Pat. 1262, January 23, 1889. 8d.

THE patentee uses a mixture of such materials, e.g., granite chips and Portland cement, as are generally employed for making artificial stone, but adds a larger quantity of water than is customary. The plastic mass is subjected to a pressure of from 1 to 2½ tons per square inch, whereby a block is produced free from air holes and faced with a skin of fine cement, which may be retained or removed, exposing the granite. The press used has its cover provided with pinions working on racks, along which it can be run into place for pressing and removed after the operation. While in position it is held by tension bars actuated by eccentrics and rests, not on the pinions, but on the mould in which is the stone mixture. The head of the ram is fitted with a grooved and perforated plate covered with a sheet of

perforated zinc, above which is a layer of felt, and finally one of cotton or linen cloth; by this means the water expressed from the mixture is provided with an exit, while the cement is retained.—B. B.

*Improvements relating to the Preparation of Silicious Materials used in the Manufacture of Artificial Stone.* A. C. Ponton, Parkstone, B. L. Moseley and C. Chambers, Hastings. Eng. Pat. 2718, February 15, 1889. 6d.

THE patent refers to the manufacture of the substance termed by the patentees "Tridymite" in Eng. Pat. 5808 of 1888 (this Journal, 1889, 462), and 15,256 of 1888, by which they mean the modification of silica obtained by strongly heating flint or sand, whereby the specific gravity is reduced to 2·3, and conversion into amorphous silica is effected. In order to facilitate the handling of the raw material it is, in the case of sand, slightly moistened with a weak solution of an alkaline silicate and moulded into blocks which can be burnt in a kiln until the change indicated above is produced, and then powdered.—B. B.

*Improvements in and Relating to Blocks or Bricks composed of Silicious Materials.* A. C. Ponton, Parkstone, B. L. Moseley and C. Chambers, Hastings. Eng. Pat. 3351, February 25, 1889. 4d.

"SEA-GRIT, shingle, broken flints, or other similar sea or fresh-water-washed aggregates" are graded by sifting, made into a plastic mass by means of a solution of an alkaline silicate, formed into blocks, allowed to dry and fired.—B. B.

*Improvements in the Process of Manufacturing Artificial Stone.* H. H. Leigh, London. From L. H. Clausen, Hamburg, and J. H. Ehlers, Bahrenfeld, Germany. Eng. Pat. 3357, February 25, 1889. 4d.

ONE part of finely powdered and well dried loam, clay, or chalk, is mixed with 1 part of "common resin" heated to about 212°—260° F., the product well stirred, the temperature raised to about 600° F., 4 parts of loam, chalk, or sand, gradually added and the whole stirred at that temperature for a quarter of an hour, after which it is poured on to polished plates or into moulds heated to 106°—150° F. The colour may be varied by suitable additions such as tar. A marbled appearance may be got by painting the interior of the moulds, which will cause a facsimile of the design thus painted to appear on the blocks; the effect of mosaic work may be obtained by inserting fragments of granite or marble previously oiled—with linseed or fish-oil—and parquetry for floors may be similarly produced. If the mixture be heated to about 940° F. for a quarter of an hour, and 5 parts of chalk, loam, or sand stirred in, the product is somewhat darker, durable, and elastic, and can be used for pavements instead of asphalt.—B. B.

## X.—METALLURGY.

*Iron in Spelter made from Blast-Furnace Flue-Dust.* E. Jensch. Zeits. f. angew. Chem. 1890, 13—15.

IN Upper Silesia flue-dust from the blast furnaces of the iron works is largely used by spelter makers. It does not work well alone, partly owing to mechanical loss from its fine state of division, and partly because the sand and oxides of iron and lead it contains destroy the muffles, but it is a useful source of zinc when smelted with other ores, such as blende and silicate of zinc. If worked alone the spelter formed contains too large a proportion of iron—up to 0·71 per cent.—which renders it brittle and unfit for rolling. Spelter made from blende or silicate does not contain more than 0·02 per cent. of iron, the whole of which is derived from the iron tools, moulds, &c. employed, whilst that made from mixtures of blende or silicate, with from 6 to 8 per

cent. of flue-dust, contains no more than 0.04 per cent. of iron. The quantity of iron in the spelter increases as there is more flue-dust mixed with the ores.

The substitution of Cowper and other fire-brick heating stoves for the old east-iron stoves in iron works has led to a more general recovery of the flue-dust, so that its importance as a source of zinc has lately become greater. But although there collects in certain parts of the flues a small quantity of dust very rich in zinc and almost free from iron, yet on the whole the dust recovered from the fire-brick stoves is much poorer in zinc and contains more iron than that formerly recovered when the old east-iron stoves were in use. Not more than 20 per cent. of the flue-dust now recovered contains more than 20 per cent. of zinc, whereas formerly 80 per cent. of the flue-dust contained at least 20 per cent. of zinc. This increased percentage of iron in flue-dust is an important fact to spelter makers.

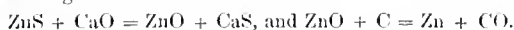
The author has made experiments to find out how the iron is carried over into the spelter. It seemed probable that it is either carried over mechanically in the form of finely-divided ferrous oxide, or that the presence of chlorides leads to the formation of ferrous chloride which volatilises and in contact with the metallic zinc is decomposed with formation of metallic iron and zinc chloride. By smelting various charges of flue-dust and silicate of zinc alone, and mixtures of these, and a mixture of silicate and ferrie chloride, it was seen that the increase of iron in the spelter was to a very small degree due to the presence of chloride, and that this cause alone far from accounted for the whole of the iron carried over. The author ultimately discovered that the finest portions of flue-dust contained the largest proportion of iron, and he concludes that the iron contained in the spelter is to be ascribed to the carrying over of this fine dust by the vapours and gases evolved in the muffle. An attempt to show whether the iron was dissolved as metallic iron or as ferrous oxide, by heating the spelter in a stream of hydrogen, yielded an unsatisfactory result.

—H. S. P.

*On the Influence of Sulphur in Spelter-making.* E. Orgler.  
Zeits. f. angew. Chem. 1890, 15—17.

The remarks contained in this paper are more or less a criticism of a paper by Voigt in the same Journal, 1889, 71 (this Journal, 1889, 986).

The author maintains that to smelters who use blende the presence of sulphur in roasted blende in the form of sulphide is of great importance, as such sulphur is combined with zinc and retains it in the residues. When lime is present in the ores, as occurs often in the case of silicates, especially of the poorer kind, and these ores are mixed with blende, any lime existing uncombined with sulphur exerts a material influence upon the yield of zinc, especially if the blende, from imperfect roasting, contains an undue proportion of sulphur. The lime in contact with zinc sulphide is converted into calcium sulphide and the zinc is liberated according to the reactions—



Voigt's analyses, showing that in zinc residues one-third of the zinc is combined with sulphur and two-thirds with oxygen, do not agree with numerous analyses relating to Rhenish-Westphalian works, where chiefly blende alone is smelted, which show on the contrary that two-thirds of the zinc in the residues are combined with sulphur, and only one-third with oxygen. The author explains this by the suggestion that, in Upper Silesia, to which Voigt's remarks refer, the ores, which there are found in the Muschelkalk, all contain large quantities of lime and magnesia. If, now, a roasted blende and a poor silicate (which class of ore is specially rich in lime) are smelted together, the lime decomposes any badly roasted blende; but if blende alone be smelted any sulphur present due to incomplete roasting remains in combination with its equivalent of zinc. The more "available" lime (*i.e.*, lime not existing as sulphate or sulphide) there is in the ores smelted the less important is it to a "dead-roast" the blende, an important point when it is remembered that the roasting is the most costly operation as regards both fuel and labour.—H. S. P.

*Outbursts of Gas in Metalliferous Mines.* B. H. Brough.  
Trans. North of England Institute of Mining and Mechanical Engineers, 1889, 59—72.

THIS paper is a collection of the various recorded instances in which gases have been discovered in metalliferous mines. Details of the circumstances attending these discoveries are given, and from a consideration of them the author concludes that the presence of gases in metalliferous mines is not in every case due to the same cause, but may be accounted for by the following hypotheses:—(1.) The decomposition of timber in a mine, and the accumulation of the fire-damp so produced in cavities that are ultimately broken into. (2.) In iron mines, where the iron is not entirely in the state of peroxide, water might be slowly decomposed with the production and accumulation of hydrogen. (3.) The production of fire-damp by the decomposition of the bituminous matter sometimes found in mineral veins or which may be present in beds underneath the ore deposit, the gas finding its way through fissures into the mine workings. (4.) The production of fire-damp by the decomposition of organic matter, in the same way as the hydrocarbons met with in salt mines. (5.) Outbursts of sulphuretted hydrogen, due to the action of acid waters on pyrites ore. (6.) The presence of carbonic acid caused by the action of acid waters, produced by the oxidation of pyrites on limestone or metalliferous carbonates.—A. W.

*Loss on Roasting Gold Ores and the Volatility of Gold.*  
S. B. Christy. Berg- und Hüttem. Zeit. 1889, 224, 290, 318.

THE experiments showed that the volatility in a current of chlorine at 100° is almost nothing. Above this temperature the loss commences to be perceptible, and a maximum is reached at about 250° C., and diminishes to a minimum about the temperature below a red heat, and then again increases, although slowly, to a maximum at something above the fusion point. The increase of volatility is steady between a red and white heat. At incipient red heat the standard loss is nearly 0.05 per cent. in half an hour, at a lower red heat it amounts to the double of this, at a cherry-red to five up to seven times, at a yellow heat to eight times, and becomes at the fusion temperature almost 30 times as great.

From this it is seen how important it is to regulate the temperature in the case of a chlorination roasting process. The action of chlorine on other metals, such as silver, copper, iron, and lead is discussed.

*The Behaviour of Copper at the Temperature of the Reverberatory Furnace.* M. Stahl. Berg- und Hüttem. 1889, 323.

WITH increasing temperature the formation of cuprous oxide diminishes, and in the case of very high reverberatory heats, takes place only to a very limited extent. On the contrary, the absorptive power of copper for gases at the different temperatures of the reverberatory furnace increases. It increases with the temperature and with the purity of the copper. These facts explain why one and the same kind of copper cast at different temperatures exhibits such entirely different properties.

*Analysis of Tungsten, Ferro-Tungsten, Tungsten Steel, Ferro-Chromium and Chromium Steel.* H. Ziegler.  
Dingl. Polyt. J. 274, 513—528.

See under XXIII., page 216.

*Quicksilver Deposits on the Pacific Coast.* Eng. and Min. J. 1890, 137.

THE United States Geological Survey has recently published monograph XIII. on the Geology of the Pacific Coast, by Mr. George F. Becker. This is a quarto volume of 486 pages, and is accompanied by an atlas of 11 sheets.

The investigation has been the most detailed of any yet made on the American quicksilver deposits, and for

comparison some of the more important European mines were visited and examined.

The scope of the work has been almost altogether limited to the geological, chemico-geological and lithological points involved.

The Engineering and Mining Journal criticises the report as follows:—

"In the determination of the geological age of the coast range and in his interpretation of the genesis of cinnabar ores, Mr. Becker takes ground at variance with Whitney and other geologists who have preceded him. The controversy about the geology of the portions of California related to the quicksilver districts is still an open one, and Mr. Becker's conclusions will probably be questioned and discussed by students in the same field. In the economic direction Mr. Becker raises a point in his first chapter which will cause some astonishment among mining men. In brief, it is an attempt to deduce the probable amount of quicksilver in the earth's crust, as compared with other valuable metals, from the relative price, cost of production and quantity produced, the conclusion being that quicksilver must be three or four times as abundant as silver, for example. Quicksilver deposits are far less numerous than occurrences of silver (of which latter metal it is very probable that enormous quantities are held in solution in sea water, to say nothing of the known ore deposits and associations with lead and copper ores). And if we compare it with gold, the facts are even more against the theory, for gold occurs in all, or nearly all, quartz, whether in veins, gravel or sand, and in great masses of eruptive, metamorphic and sedimentary rocks, though not in such concentrated form as to be available for mining. So also comparisons with many other metals of similarly wide distribution would be misleading. It so happens that cinnabar, meta-cinnabar, native mercury, &c. when found at all, are usually found in a relatively more bunched condition—that is, more favourably placed for the miner.

"Nor can we agree with Mr. Becker in the broad assertion that the price of a metal is only slightly in excess of its average cost of production. The author assumes a ratio of 100 to 90. Following this comparison out, it would, therefore, appear that the quicksilver which sold at 1.50 dols. must have cost 1.35 dols. per pound to produce; that at 1 dol. would have cost 90 cents; and that at 40 cents 36 cents only. This is manifestly not the case. The quicksilver market is peculiar in being one of limited and widely fluctuating demand, depending mainly on the amount which can be absorbed in amalgamating gold and silver, and in vermilion-making. It is also affected by the policy of foreign monopolies. If quicksilver were a metal of almost universal application, with a practically unlimited market, like iron and steel, there would be more force in the argument."

The following is a summary of Mr. Becker's principal conclusions:—

"Quicksilver appears to be rather more than three times as abundant in nature as silver. The quicksilver produced in the world from 1850 to 1885, inclusive, weighed 1.74 times as much as the silver produced, but the value of the silver was about 16.4 times that of the quicksilver. The great quicksilver-producing localities of the world have been Almaden in Spain, Idria in Austria, Huancavelica in Peru, California, and the province of Kwei-Chau in China. No statistics are known to exist of the Chinese product. The total known products of the other regions take rank in the order in which they are named above, but of late years Peru has produced nothing, and from 1850 to 1885 California yielded about half the total product. The production of Italy is more important than it is usually assumed to be. In 1886 the yield was 7,478 flasks. The production of California, which was nearly 80,000 flasks in 1877, was only about 30,000 in 1886. [It was 25,650 flasks in 1889.—*Ed. E. and M. J.*]

"A chain of quicksilver deposits of very greatly varying commercial importance almost girdles the world. Beginning in Spain, these deposits are distributed along the great chain, including the Alps, Caucasus, and Himalayas, to China; thence through Japan along the eastern edge of the Asiatic continent to the Arctic circle. Beginning again in Alaska,

the deposits follow the western Cordilleras down to Chili. Brief descriptions of the more important or more interesting of these deposits are given in Chapter II., and serve as an introduction to the discussion of the deposits of the Pacific slope.

"The sedimentary rocks of the coast ranges of California are almost all composed of granite detritus. A portion of these have been subjected to very intense metamorphism, and have been converted into thoroughly crystalline rocks, in part schistose. These rocks are of Cretaceous age, and are grouped as pseudodiorite, pseudodiorite, glaucophane-schists, plthanites, and serpentine. Very elaborate field studies, microscopical examinations, and chemical analyses of these rocks are given in Chapter III., which is mainly devoted to the investigation of their origin and the processes by which they have become recrystalline. The conclusion reached is that dynamical action, together with warm waters carrying magnesian salts and silica in solution, effected the metamorphosis at the epoch of an exceedingly violent upheaval. This chapter also includes an investigation of concretions in sandstone, which are referred to the action of organic matter, and the analysis of the conditions under which decomposition will produce rounded nodules like pebbles.

"The massive rocks of the quicksilver areas include granite, ancient porphyrites, andesites, rhyolite, and basalt. A new group of andesites is discussed, for which the name *asperites* is suggested. It is shown that these rocks are of variable mineralogical composition, even in the same eruptions, while all of them share a trachytic habitus. The name is simply a latinised equivalent of trachyte. Very remarkable andesitic and basaltic glasses occur near Clear Lake in areas of unusual size. These glasses are extremely acid, but contain also a very high percentage of alkalis, and it is because of this peculiar chemical composition that they have failed to crystallise, not because they have cooled more rapidly or under less pressure than the accompanying crystalline rocks. An attempt is also made to show that the original crust of the earth was granite, and reasons are given for believing that the primeval rock is exposed in California. The lavas burst through the granite, and the conclusion is reached that they cannot possibly consist of remelted sediments.

"The historical and structural geology of the quicksilver belt is discussed in Chapter V. It is shown that the metamorphosed rocks pass over into early Cretaceous beds containing a very characteristic fossil of the genus *Ancella*. Soon after the era in which this mollusk lived—the Neocomian—occurred the great upheaval which induced the metamorphism. The next strata in point of age comprise a hitherto undetected group of the middle Cretaceous called the Walla beds. They were laid down unconformably on the already metamorphosed Neocomian. At the very end of the Cretaceous the Chico series were deposited for the most part on the metamorphic rocks and unconformably with them. Following the Chico are the Tejon beds, which are here regarded as Eocene; but there was continuity of life and of sedimentation from the Chico to the Tejon, or from the Cretaceous to the Tertiary—a state of things detected nowhere else in the Northern Hemisphere. Upon the Tejon lie the Miocene rocks with no notable nonconformity. The close of the Miocene was marked by an important upheaval, which was recognised by early observers. The volcanic period seems to have begun nearly at this time. The end of the Pliocene was also marked by disturbances, and most of the *asperites* seem to have been erupted at this epoch. The ore deposits stand in close relation to the volcanic phenomena and are probably nearly or quite all Post-Pliocene.

"The gold belt of California contains *Ancella*-bearing beds in Mariposa and Tuolumne counties. This shell is of the same species as that in the coast ranges, and the first known upheaval of these mountains was contemporaneous with an important addition to the Sierra Nevada. A description of various forms of *Ancella* from different portions of the world, by Dr. C. A. White, with plates, forms an appendix to this chapter.

"Descriptive chapters follow dealing with the various districts, of which detailed surveys were made. Each of these districts affords special facilities for the study of some

special topic. The Clear Lake region contains freshwater Pliocene beds, and in it the age of audesities can be determined. It also contains remarkable areas of volcanic glass. At Sulphur Bank cinnabar is being precipitated from heated waters largely by the action of ammonia. At Knoxville, besides the ore deposits, there are admirable opportunities for determining the age of the metamorphic rocks and for studying the process of alteration. At New Idria the nonconformity between the metamorphic rocks and the Chico and the continuity between the Chico and Tejon appear. The New Almaden mine is particularly well adapted for the study of the structure of the ore deposits. At Steamboat Springs cinnabar is being deposited without the complications introduced by the presence of ammonia.

"In Chapter XII. the Great Western, Great Eastern and Napa consolidated mines are described, and in the next chapter more or less information is giving concerning each of over fifty minor deposits on the Pacific slope. Some of these have been productive mines, while others are mere prospects of only a geological interest.

"A general description of the deposits described follows, including the enumeration of the gangue minerals, the microscopical character of ores, &c. It appears that the cinnabar has been deposited solely in pre-existing openings, and never by substitution for rock. The fissure systems, which are always present, are very irregular, and deposits cannot be conveniently classified according to existing systems. A new descriptive term, 'chambered vein,' is suggested, which would include nearly all the deposits. A chambered vein is defined as a deposit consisting of an ore-bearing fissure and of ore bodies contiguous with the fissure which extend into the country rock. It appears that all of the deposits described have probably been deposited in the same way from hot sulphur springs.

"Chapter XV. deals with the processes by which the ore has been dissolved and precipitated in nature. It is shown by experiment and analysis that cinnabar unites with sodium sulphide in various proportions, forming soluble double sulphides, and that these compounds can exist in such waters as flow from Sulphur Bank and Steamboat Springs either at ordinary temperatures or above the boiling point. Metallic gold, iron pyrites, copper pyrites, and other minerals found with cinnabar are also soluble in the same solutions.

"A discussion of the origin of the ore concludes the investigation. It is shown that the quicksilver is probably derived from granitic rocks by the action of heated sulphur waters which rise through the granite from the foci of volcanic activity below that rock.

"For the convenience of those who consult the report the separate chapters are made as far as possible independent of one another, a plan involving a certain amount of repetition. Further, to facilitate the use of the volume, the last chapter presents a summary of those which precede it."

#### PATENTS.

*Improvements in the Manufacture of Iron and Steel alloyed with Nickel.* H. Schneider, Le Creusot, France. Eng. Pat. 16,567, November 14, 1888. 4d.

NICKEL scrap in any form is melted in any suitable furnace with cast iron or steel, with the production of alloys of steel and nickel containing from 5 to 30 per cent. of the latter. These alloys are said to be suitable for the manufacture of armour plates, projectiles, &c.—A. W.

*Improvements in the Manufacture of Iron and Steel alloyed with Copper.* H. Schneider, Le Creusot, France. Eng. Pat. 16,568, November 14, 1888. 6d.

THE patentee claims the manufacture in a blast-, cupola-, or reverberatory furnace, of cast iron or steel, containing variable percentages of copper, such alloys being applicable to the production of armour plates, &c. Copper ore, scrap, or a mixture of matte and scrap, may be introduced into the ordinary blast furnace or cupola, or the copper may be first made into a cuprous coke. The product generally contains from 5 to 20 per cent. of copper, but the patent is not confined to these proportions.—A. W.

*Improvements in the Wet Method of extracting Gold from Crushed Ores or other finely divided Auriferous Material.* J. H. Pollok, Glasgow. Eng. Pat. 16,796, November 19, 1888. 4d.

THE patent relates to the extraction of gold from crushed quartz or other auriferous material and especially from refractory ores, float gold ores or tailings, more particularly in connexion with the process of extracting gold from crushed ores by means of chlorination under hydraulic pressure as described in Eng. Pat. 17,195 of 1887 (this Journal, 1888, 678). The improvements consist in removing the air from the vessel in which the ore is placed for chlorination, and in forcing in water heated to about 60° C.

The ore, suitably crushed, and if desirable concentrated and roasted, with or without salt or other reagent according to its nature, is charged together with the requisite reagents into a suitable revolving cylinder properly protected from the corrosive action of the chlorine evolved in the operation. The reagents used to generate chlorine are charged into the cylinder, the one before and the other after the ore, to avoid the liberation of chlorine before the closing of the cylinder and the revolving of the same. After the ore and reagents have been charged, the air is removed from the cylinder by a suitable stop-cock placed for that purpose, and this is effected either by any air-removing device such as an air-pump or by driving out the air by the admission of water. After the air is removed, or simultaneously with its removal, water is forced into the cylinder, and this water is heated to 60° C. either during its introduction (by means of a steam injector) or previously to or during the revolution of the cylinder. "As soon as the cylinder is completely filled with ore, reagents, and water, the steam acts as a very convenient and economical water-forcing device for it presses on the condensed water in the supply pipe, and forces it into the cylinder, thus exerting hydraulic pressure in the cylinder equal to the steam pressure in the boiler." The cylinder is next revolved, and in the absence of air and at or about the temperature of 60° C., and under the hydraulic pressure, the chlorinating liquid rapidly attacks and dissolves the gold. When the action is complete the chlorine is blown or aspirated off and the ore and solution discharged into a suitable filter. In cases where copper and silver are present with the gold the liquid may be made, by means of a steam ejector, to circulate through the ore in a hot state for some time before removal from the filter, as this greatly assists the solution of the silver.

This method is equally applicable when bromine or iodine is used in the place of chlorine.—H. S. P.

*An Improved Process of Extracting Iron from its Ore and of Refining or Purifying Iron.* A. Q. and A. Brin, London. Eng. Pat. 17,858, December 6, 1888. 4d.

IRON ore, charcoal, lime, and sodium chloride are heated together in any convenient furnace. The nascent chlorine from the salt is stated to remove the impurities from the iron, especially the phosphorus and the sulphur. The process can also be applied to the purification of wrought iron and the refining of steel.—A. W.

*A New or Improved Apparatus for Treatment of Auriferous Sand and other Matters containing Free Gold.* A. Clement-Conti, Foleto, Italy. Eng. Pat. 18,004, December 10, 1888. 8d.

THIS is an apparatus, which is said to be inexpensive and portable, for washing auriferous sand or other alluvial deposits, or mixtures containing particles of free gold. The sand is first introduced upon a wooden or metallic shoot, and is then washed over a grating, so as to separate pebbles and coarse sand. The fine sand and gold which pass through the grating are then carried by the flow of water into a funnel provided with two overflows, so as to keep the level of the water in it constant, and to obviate vibration and regulate the force of water passing through the funnel. This funnel is adapted to a tube passing nearly to the bottom of a cylindrical vessel, and it can be raised or lowered so as to regulate the water-pressure in the cylinder.



The floor of the cylinder is in the shape of an inverted cone, and a plug is fitted into a hole at the apex. The particles of gold settle on this floor, whilst the lighter sand passes out of the cylinder by a number of syphons, which empty into an annular trough surrounding the cylinder, in which some mercury is placed, to catch any fine particles of gold that pass over with the sand. On the top of the cylinder is placed a trough or cistern, kept filled with water, which is allowed to flow into the cylinder by pipes passing through the bottom of the trough, and reaching nearly to the bottom of the cylinder. More water being allowed to flow into the cylinder than can be carried away by the syphons, overflow pipes are fitted at a level near the top of the cylinder, which discharge into the same annular trough as the syphons. The gold which collects on the floor of the cylinder is, when necessary, allowed to pass into a receiver placed below by removing the plug in the cylinder-bottom. The operation may be carried on by a successive number of concentrations.

Drawings of the apparatus accompany the specification. The size of cylinder represented is only 36 centimetres in diameter, but this size is calculated to treat 10,800 litres of sand passing through the sieves in 10 hours. These 10,800 litres of sand are reduced to 10 litres of rich sand, and this is then treated in a much smaller apparatus, where it is reduced to  $\frac{1}{2}$  or  $\frac{2}{3}$  litre, and then again in a still smaller apparatus to 50 cc., consisting of gold and iron, and a few grains of sand. It is then dried, and the iron removed by a magnet.

Another way of using the apparatus is to introduce mercury into the washing cylinder.—H. S. P.

*Improvements relating to the Extraction of Gold, Silver, and Lead from Substances containing the same, and to Apparatus therefor.* A. B. Cunningham, Islington. Eng. Pat. 28, January 1, 1889. 8d.

THE ore is heated with caustic soda or potash to fusion of the caustic alkali. If an insufficient quantity of lead be present, a requisite quantity of galena or other substance containing lead is added.

The operation may be carried out on the hearth of an open reverberatory furnace provided with an iron bottom and sides, or lined with a fused caustic bottom, or with any other lining calculated to resist the action of caustic alkali. But in preference it should be carried out in a cast-iron (or wrought-iron) vessel or pot. When sulphides are treated, this pot is lined with rings of cast iron, placed one on top of the other, and adapted to the internal form of the pot. This lining is easy of adjustment, and can be replaced when required. A drawing of a suitable pot and furnace for heating the pot accompanies the specification.

In treating galena, the patentee finds 30 per cent. by weight of caustic alkali gives a good result. The lead collects at the bottom of the vessel, and carries down with it the gold and silver, which is then recovered by cupellation or other suitable process.—H. S. P.

*Improvements relating to the Extraction of Aluminium from Cryolite and other Substances.* C. Netto, Dresden, Germany. Eng. Pat. 359, January 8, 1889. 6d.

THE basis of this patent is founded on the fact that during the extraction of aluminium by sodium the impurities generally associated with the alumina are reduced in the early stages of the operation. The process, therefore, is divided into two parts. The mixture of cryolite and salt is first treated with one-third of the theoretical amount of sodium and is then run off from the metal containing the impurities thus produced, and subjected to the action of the remainder of the sodium, in which latter operation pure aluminium, containing only 0.5 to 1.0 per cent. of impurities, is obtained. This fractional reduction, which is not necessarily confined to two parts, or to the division of the sodium as above, is claimed for application to all processes for obtaining aluminium. The crucibles or other vessels in which the operations are performed are preferably lined with basic material as a safeguard against silicon and other impurities.—A. W.

*Improved Means of Extracting Precious Metals from Ores.* J. B. Hannay, Loch Long. Eng. Pat. 841, January 16, 1889. 8d.

RELATES especially to the extraction of gold from its ores in the form of chloride. The patent is for an apparatus devised for this purpose, which consists of a chlorinating vessel, a set of circulating pumps, a filter-press, and a chlorine pump, or sets of these with suitable communicating pipes, cocks, and valves. The ore is first reduced to a fine powder and then mixed with water or chlorinated water, so as to form a sludge capable of being pumped. The chlorinating vessel is charged with this sludge, and the pumps cause the latter to circulate by drawing it from the upper part and discharging it into the lower part of the vessel. Meanwhile, chlorine gas is pumped into the vessel, preferably at a pressure considerably above that of the atmosphere. After circulation has gone on until the most part of the gold is dissolved, the sludge is delivered by the circulating pumps to the filter-press, additional pressure being obtained if needed by using the chlorine pumps to force air into the upper part of the vessel. The liquid issuing from the filter-press may be treated in any of the known ways for separating the gold from it, or it may be used over again with a fresh quantity of ground ore until it is of a desirable strength. It is advantageous to charge the chlorinating vessel with an excess of chlorine above that required for combination with the gold contained in the sludge; and to recover this excess, before discharging the contents of the chlorinating vessel into the filter, a little steam is blown into the vessel and the expelled chlorine collected in a gasholder or led to another chlorinating vessel; or an aspirator may be used for drawing off the excess of chlorine. When silver is present the blocks which are taken from the filter-press are reduced again to a sludge and subjected to a similar treatment with a suitable solvent instead of chlorine. Other means of filtering may be used instead of a filter-press.—H. S. P.

*An Improved Method and Means for separating Metals and Heavy Minerals from Ores.* R. Stanfield and T. Clarkson, Battersea. Eng. Pat. 890, January 17, 1889. 8d.

THE ore, after being brought to a fine state of division, is fed, with or without water (preferably without water) into a suitable receptacle revolving about a vertical or suitably-inclined axis, and provided at its circumference with holes through which the particles of ore are ejected. These holes may be straight or curved and arranged radially or otherwise, and may be in one place so as to project the material horizontally, or on the surface of a cone so as to project it upwards or downwards as desired. The vessel may be also arranged with straight or curved projecting tabular arms. If the particles of ore are of the same size they will be separated according to their densities—the denser particles being projected farthest; if, however, the particles differ in size, it is advisable to perform a preliminary sizing operation. A receiver is placed around this centrifugal separator at a lower level. This receiver may be divided into any convenient number of concentric compartments, so as to divide the material deposited upon it into any required number of parts, and brushes or scrapers may be provided to remove the contents of each division of the receiver. The receiver may also be kept moist by water or steam or any other suitable fluid. A current of air may be made to operate upon the particles of ore as they fly from the rotating vessels so as to assist their deposition upon the receiver.

The separation may be assisted by adding a heavy substance (such as mercury) which would combine with the metallic portion of the material.

The machine is applicable to ores of gold, silver, tin, lead, copper, or any other metal. It may be either used for sizing purposes or for separating metals and minerals from ores, and the improved receiver may be used with any form of centrifugal separator or sizing apparatus.—H. S. P.



*Improvements in the Manufacture of Iron and Steel.* J. Richardson, Pocklington. Eng. Pat. 1099, January 21, 1889. 4d.

THREE solutions are prepared: the first is prepared by dissolving in water with the aid of heat certain proportions of nitrate of soda, nitrate, prussiate, and bichromate of potash, and mixing with the solution "naphtha, paraffin, or certain other hydrocarbons;" the second solution is made by dissolving gum catechu and raw sugar in water; and the third is a solution in water of pearlash, acetic acid and chloride of lime. These three solutions are mixed together and fed into a boiler, and there heated. The vapour obtained is then passed through, over or under the molten iron, until the quality of the metal is sufficiently improved.—A. W.

*Improvements in or appertaining to the Process of Galvanising Iron and Steel.* T. B. Saunders, Cleckheaton. Eng. Pat. 1571, January 29, 1889. 6d.

THE improvements relate to the removal and recovery of the ammoniacal fumes formed in the galvanising tank when sal-ammoniac is being used. The tank is covered with a convenient hood, connected by means of a pipe with a condensing tower. The fumes are drawn through into the latter, where they are condensed by means of water or acid spray.—A. W.

*Improvements in the Manufacture of Aluminium and Alloys thereof.* L. Petit-Devancelle, Paris, France. Eng. Pat. 7013, April 26, 1889. 8d.

THE metal with which the aluminium is to be alloyed is melted in a suitable furnace, preferably in a rotatory furnace such as a Dank's furnace, the aluminium being then added in the form of a salt of aluminium. To assist the thorough mixing of the contents of the furnace, a helical rib or flange is fixed upon the inside wall of the furnace. This rib may be made of iron or fire-brick.

For producing alloys of iron and aluminium, any of the ordinary compounds or substances containing aluminium may be used with the iron; for alloys of aluminium with other metals, for example, copper, a silicate of aluminium only should be used.

If it is desired to obtain the aluminium in an unalloyed condition, the aluminium is recovered from the alloy by ligation, distillation, or oxidation of the metal used to form the alloy, the aluminium remaining as a residuum.

—H. S. P.

*An Improved Process for the Treatment of Auriferous and Argentiferous Antimony Ores.* C. Schreiber and H. Knutsen, Broken Hill, New South Wales. Eng. Pat. 14,235, September 10, 1889. 6d.

IN the usual method of roasting these ores before amalgamation there is a considerable loss of both gold and antimony. The gold occurs in metallic state, and generally on the surface of the antimony at the points of contact with the quartz. During the roasting the particles of gold become coated with a film of oxide of antimony which prevents the subsequent action of the mercury. Moreover, a certain quantity of antimony becomes amalgamated, and causes the mercury to "sicken." If the ore be roasted with salt a basic chloride of antimony is formed which forms a protective coating on the particles of gold, so that a similar difficulty in amalgamating is experienced.

The improved process consists in "leaching" or lixiviating the sulphides and oxides of antimony with a solution of a sulphide of the alkalis or alkaline earths, preferably with a solution of sodium sulphide.

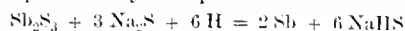
The ore is first hand-picked into three classes, more or less, viz., No. 1 ore containing upwards of 25 per cent. of antimony; No. 2, containing from 5 to 25 per cent.; and No. 3, containing less than 5 per cent. It is then crushed and passed through a sieve of not less than 900 meshes to the square inch. It is then taken to the lixiviating pans or agitators, where it is treated with a solution containing

sulphide of sodium in a rather larger proportion than is required by the equation  $\text{Sb}_2\text{S}_3 + 3 \text{Na}_2\text{S} = 2 \text{SbNa}_3\text{S}_3$ ; e.g., in the proportion of 12 parts of  $\text{Na}_2\text{S}$  to 17 parts of  $\text{Sb}_2\text{S}_3$ . If sulphide of barium or calcium be used an amount is taken proportionate to the molecular weight of the salt used.

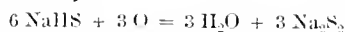
In practice, to obtain a concentrated solution of the antimony double salt, it is best to wash the ore first with a solution that has been twice used, then with one that has been once used, and finally with fresh liquor.

The ore being now free from antimony can be easily amalgamated for the recovery of the gold and silver.

The antimony is recovered from the solution of the double salt by means of electrolysis. For this purpose care must be taken to regulate the strength of the current to the degree of concentration of the solution. The reaction at the cathode may be represented by the equation—



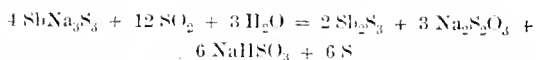
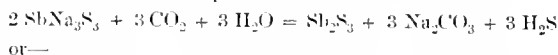
and at the anode by—



During the process a quantity of caustic soda is from time to time added to convert the sodium bisulphide into sodium sulphide:— $\text{Na}_2\text{S}_2 + 2 \text{NaOH} + 2 \text{H} = 2 \text{Na}_2\text{S} + \text{H}_2\text{O}$ .

To increase the conductivity of the solution a small quantity of common salt is added to it. The liquors are circulated continuously from cell to cell, until the whole or nearly the whole of the antimony is precipitated. The residual solution of sodium sulphide is then returned to the lixiviating pans. "In practice it is considered that all solutions containing a certain quantity of the double salt of antimony will repay treatment by electrolysis," strong solutions being diluted with weak ones, and weaker ones increased in strength by dissolving in them sulphide of antimony from another part of the process. In the third class the liquor obtained by treating 5 per cent. and lower grade ores is worked otherwise. From this it is proposed to separate the antimony by allowing the solution to drip continuously down a tower about 20 ft. high, so as to come in contact with carbonic acid, or preferably with sulphurous acid, which is allowed to enter at the bottom of the tower.

The reactions are represented as follows:—



When carbonic acid is used, and especially in the case when sulphide of calcium or barium is employed, the precipitate at the bottom of the tower contains insoluble carbonates. The whole precipitate is then reduced in a muffle or reverberatory furnace and smelted for antimony. But when sulphurous acid is used there are formed soluble hyposulphite and bisulphite of sodium, from which the precipitated sulphide of antimony is removed by filtration and the whole of the sodium sulphide originally used can be recovered from the filtrate by evaporation and reduction with powdered coke or charcoal. The sulphide of antimony is placed in canvas bags and suspended in the electrolytic cells for strengthening a weak solution or for replacing the dissociated trisulphide. The metallic antimony is collected, washed with hot water, pressed into cakes, and smelted in crucibles for the market.—H. S. P.

*Improvements in the Manufacture of Iron and Steel.* G. Archbold, New York, U.S.A. Eng. Pat. 15,824, October 8, 1889. 4d.

WITHERITE or other salt of barium is introduced with the ordinary charge into the blast furnace with the object of removing the phosphorus and sulphur and producing pig iron free therefrom. These impurities can also be removed from iron by treating the latter with the barium salt in the Bessemer or other converter, the quantity used being the theoretical amount necessary to produce sulphide and phosphide of barium. The theory is as follows: The barium is converted at the temperature of the furnace

into cyanide which reacts on the iron phosphide, producing barium phosphide and ferrocyanide of iron, which latter is converted into "refractory carbide of iron which mixes with the slag." When much silicon is present it is found advantageous to use also barium nitrate in addition to the carbonate.—A. W.

*Improvements in the Manufacture of Iron and Steel.* R. F. Ludlow, Springfield, U.S.A. Eng. Pat. 16,474, October 18, 1889. 8d.

This patent applies to the oxidation of the impurities in iron by air, the claim being that instead of the air being directed on to or through the metal from any one position, it is blown upon the surface at an angle and at constantly varying places, which gives the mass a "practically uniform disturbance."—A. W.

## XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

### PATENTS.

*Improvements in the Method of and Means for separating Matters in Solution by Osmose, Dialysis, or Diffusion, or by Electrolysis, or by a Combination of those Processes.* J. Marx, Ulm, Germany. Eng. Pat. 2367, February 16, 1888. Amended October 22, 1889. 8d.

THESE improvements have been already described in this Journal, 1889, 288. The emendations appear to consist in the correction of printer's errors in the text of the original specification—no alteration being made in the claims.—O. H.

*Improvements in the Production of Oxygen and Hydrogen in Large Quantities by Means of the Electrolysis of Water, applicable for Ballooning and other Uses.* D. Latchinoff, St. Petersburg, Russia. Eng. Pat. 15,935, November 3, 1888. 11d.

To obtain oxygen and hydrogen in large quantities a large current, say 600 ampères at a potential difference of 110 volts, is led through three parallel set of electrolytic baths, each set containing 44 separate cells connected in series for this particular number of volts. Each cell contains either water acidulated with sulphuric acid or a 10 per cent. solution of caustic soda, and immersed in the solution are two carbon cathodes and a lead anode, or iron plates may be used. Suitable gas collectors are arranged over each cell enabling the two gases to be dried and carried off to suitable gas-holders. By this means, the patentee states, 640 cubic metres of hydrogen can be obtained in 60 hours, and the value of the oxygen at the current price more than pays all expenses.—B. T.

*Improvements in Electro-Chemical Generators.* F. J. Brougham, London. From La Société Perreux Lloyd and Fils, Paris, France. Eng. Pat. 18,858, December 24, 1888. 8d.

A NOVEL form of battery is described in which the heat generated by the internal electrical resistance is utilised to heat and concentrate the solutions employed to such a degree that the salts formed will crystallise out upon cooling. The battery is so constructed that the solutions can be heated by steam if the heat obtained electrically be not sufficient. The salts obtained are of more value than the original materials employed, so that the electricity is obtained as a by-product. The depolariser, nitric acid, is used over and over again, the nitric oxide formed being converted by the oxygen of the air into hyponitric acid, and then reconverted into nitric acid in suitable condensing towers.—B. T.

*Improvements in and Appertaining to the Manufacture of Metals or Metallic Alloys by Means of Electricity.* G. C. Dymond, Liverpool. From J. M. A. G. Lécuyer, Courbevoie, France. Eng. Pat. December 29, 1888. 8d.

THE raw material is incorporated with carbon and made into electrodes; an electric arc is then formed between a pair of these electrodes over the bed of a reverberatory furnace. The ore in the electrodes is reduced and the metal falls into the hot bed of the furnace. In some cases alloys are formed by mixing other metals, as powders, with the materials of which the electrodes are composed. Details of construction of apparatus are described, one of which consists of jointing on fresh electrodes to the unburnt ends of the partially used ones, so as to enable the process to go on continuously.—E. T.

*Improvements in Secondary or Storage Batteries.* T. Cuttriss, Leeds. Eng. Pat. 997, January 19, 1889. 6d.

A PLUG or core is put into the containing vessel, of such a size and shape as to leave a narrow space all round between it and the walls of the vessel. This space is then filled with a paste of oxide of lead and sulphuric acid, which when hardened forms the active material of one electrode of the cell. When the containing vessel is non-metallic it is lined with platinum or lead, or strips of these materials are bedded into the paste so as to make good electrical contact with it.—E. T.

*Improvements in Secondary Battery Plates, Elements, or Supports.* J. S. Sellon, London. Eng. Pat. 1460, January 26, 1889. 6d.

To prevent the active material falling out of the plates a compound plate is used, so constructed that the ribs on one side are not coincident with those on the other, but are situated reversely so that the active material interlaces between these ribs, and is at the same time made up of one continuous mass instead of being made up of separate individual pellets as usually constructed.—B. T.

*Improvements in the Construction of Secondary Batteries.* G. Philippart, Paris, France. Eng. Pat. 1673, January 30, 1889. 8d.

THE supports are made of lead, compressed, or cast under pressure. The active materials are made into a thin paste with glycerin or some such substance and spread over a piece of fabric, such as felt, asbestos cloth, &c. This layer of paste is pressed into the fabric, the prepared fabric being then heavily pressed into contact with the lead supporting plate. The layer of active material may be further protected by an external sheet of perforated celluloid or such material, and the electrodes may be of any desired shape.—E. T.

*Improvements in and Relating to the Production of Chlorine and the Manufacture of Sulphate of Soda.* T. Parker and A. E. Robinson, Wolverhampton. Eng. Pat. 2310, February 9, 1889. 4d.

See under VII., page 189.

*Improvements in Electrical Accumulators or Secondary Batteries.* W. E. Langdon, Derby. Eng. Pat. 5816, April 5, 1889. 8d.

THE object is to get rid of the spray given off during charging. Each cell has a cover whose edge fits into a groove running all round the top of the cell, and filled with liquid. The cover has a small aperture in the crown, through which any spray-laden gas passes into a "decomposing cell." This cell may be said to consist of three concentric tubes. The first, or innermost, is covered as by a cap, by the second, which is closed at the top. The gas given off passes up the first tube, down between the first and the second, and then up between the second and third. The space between the two latter is packed with lime or some such material capable of absorbing the spray.—E. T.

*Improved Supports of Secondary Batteries.* G. E. Heyl, Charlottenburg, Germany. Eng. Pat. 13,800, September 2, 1889. 6d.

THE object of the improvement is to get an elastic support that can expand and contract with the active material. Using a perforated plate, the ribs forming the boundary of each mesh, are themselves perforated by narrow slits so as to split what was a common boundary to two adjacent meshes into two distinct boundaries, one for each mesh. The thin boundary walls may afterwards be turned inwards so as to make the opening to each mesh smaller than the interior. Neither the perforations nor the slits need extend right through the plate, and there may, if preferred, be perforations and slits on each side separated from each other by a thin continuous septum of lead.

—E. T.

*Improvements in Thermostats.* H. Cortland, Toronto, Canada. Eng. Pat. 15,800, October 8, 1889. 6d.

A METALLIC tongue free at one end but fixed at the other is stretched diametrically across an annular metal case, but electrically insulated from the case, the tongue carrying at its centre an adjustable screw, one end of which presses against the centre, on the concave side, of a concavo-convex hard rubber diaphragm carried by the same metal case. As the diaphragm expands or contracts with changes of temperature, the position of the free end of the tongue alters and makes or breaks an electrical contact, which can be used to give a signal in any well-known way.—B. T.

*Improvements in Devices for Connecting the Plates of one Electrical Battery with another Battery or Batteries, and with a Switch-board or Transmitting Device.* W. F. Smith, Philadelphia, U.S.A. Eng. Pat. 15,819, October 8, 1889. 8d.

THE coupling device is such as might be made in the following manner. A short length of tube, screwed over a part or the whole of its outside, is split longitudinally into two, and one-half attached perpendicularly, near its middle, to the end of the strip from one cell, while the other half is similarly connected to the strip from an adjacent cell. To couple the two cells the two halves of the tube—each attached to its strip—are brought together and a "thimble" screwed over them, so as to connect them rigidly together. Instead of the outside of the tube and the inside of the thimble being screwed, they may be slightly coned to fit each other. A little mercury is put at the bottom of the thimble to ensure good connexion, and any parts may be electro-plated to resist amalgamation. Branch or other connexions can be made by a plug fitting into the jointed tube and provided with a flexible conductor.—E. T.

*Improvements in Frames for Supporting the Plates or Elements of a Secondary or Storage Battery.* W. F. Smith, Philadelphia, U.S.A. Eng. Pat. 15,820, October 8, 1889. 8d.

A FRAME of lead or suitable material is used to support a series of plates to be formed, the said frame being provided with a lug for connecting up in the usual manner, and also with an auxiliary loop or extension, two forms of which are shown, to be used during the process of formation of the plates, both as a connexion and also as a means of lifting the plates, thus avoiding all damage to the lug which is to be used when the plates are in their finished state.—B. T.

*Improvements in the Method of Electrolytically Reducing Plates of Metallic Salts to a Metal State to "Form" the Electrodes of Secondary or Storage Batteries.* S. C. C. Currie, Philadelphia, U.S.A. Eng. Pat. 15,821, October 8, 1889. 8d.

IN the ordinary methods of reducing a salt of lead to the metallic state, the salt is held in a stout frame of suitable metal and treated electrolytically; as, however, the salt is usually a poor conductor, the formation is but slow, and

does not take place uniformly throughout the plate. To obviate this, and to accelerate the formation, a perforated plate of conducting metal is pressed down upon the surface of the salt, and the whole is treated electrolytically in the usual manner, the plate to be formed being connected to the negative pole of the source of electricity.—B. T.

*Improvements in Galvanic Batteries.* W. E. Irish, Brooklyn Village, Ohio, U.S.A. Eng. Pat. 16,036, October 11, 1889. 8d.

ZINC treated with an amalgam of mercury and tallow is used for the positive electrode, and is immersed in a solution of sulphuric acid, Mecca, or other suitable oil, and sodium sulphate in suitable proportions, while the negative electrodes are composed of carbon in the form of plates or pencils. Similar plates or pencils of carbon are also placed outside the porous pots, close to but not in contact with the zincs, to reduce the resistance of the element, or auxiliary plates of iron are used adjacent to and connected with the zinc in order to prevent waste of the latter while the battery is not working.

The negative electrode is immersed in a solution containing the following in suitable proportions:—Bichromate of potash, chlorate of potash, nitrate of soda, flour of sulphur, chromic acid, nitric and sulphuric acids. The battery solutions may be covered with a suitable oil or paraffin wax to prevent escape of fumes.—B. T.

*Improvements in Moulds for Casting the Plates of Secondary or Storage Batteries.* W. Shapleigh, Camden, New Jersey, U.S.A. Eng. Pat. 16,662, October 22, 1889. 8d.

THE improvements are for casting small blocks or plates of active material, round which a frame of better conducting material may afterwards be cast. The mould is made in two symmetrical parts hinged together. The liquid material is poured down a central feeder to the bottom of the mould, and thence passes into vertical risers. "Knife edge" slits extend from the sides of these risers to the matrices in which the blocks are to be cast, and branch air-ways extend from the top of each matrix, to which they are connected by other knife-edge slits, to vertical main air-ways communicating with the exterior. It is claimed for this mould that very solid homogeneous blocks can be cast without air-bubbles forming, and that such blocks can be readily separated from the mould and from each other.—E. T.

*Improvements in Treating and Rectifying or Ageing Alcohol or Alcoholic Liquors or the like by Electricity.* A. de Meritens, Paris, France. Eng. Pat. 16,947, October 26, 1889. 4d.

See under XVII., page 205.

## XII.—FATS, OILS, AND SOAP MANUFACTURE.

*Mineral Lubricating Oils.* A. Kunkler. Dingl. Polyt. J. 274, 276—284.

THE author, in giving his experience of testing oils, does not consider an oil-testing machine necessary.

He gives in tabular form the results obtained on testing a large number of Russian, American, and German mineral oils, and also of rape, olive, castor, linseed, and other oils.

The points determined were:—1. Specific gravity at 17.5° C., in most cases by the pycnometer, in a few by the Westphal balance. 2. The temperature at which vapour is first given off. 3. The flashing point. 4. The burning point. Both the latter were determined in a small porcelain

crucible. 5. The freezing point was taken in an ordinary sized test-tube. 6. The colour. 7. The viscosity at 20°, 30°, 50°, 60°, 70°, 100°, and 150°, the viscosimeter used being that of C. Engler. The following are some of the results obtained:—

Class of Oil.	Sp. Gr. at 17.5°.	Flashing Point.	Viscosity.	
			50°	100°
Russian cylinder oils.	911—923	183°—238°	10.2—16.2	2—2.8
Russian machine oils.	893—920	138°—197°	5.8—6.3	1.5—1.8
Russian spindle oils.	893—895	163°—167°	3.1—3.1	1.4—1.5
American cylinder oils	886—899	280°—283°	..	4.1—4.8
American machine oils	884—920	187°—206°	4.2	1.6
American spindle oils	908—911	187°—200°	3.1—3.3	1.4—1.6
Rape oil, crude.....	920	265	4.0	1.7
Rape oil, refined.....	911	395°	4.9	2.0
Olive oil.....	914	245°	3.7	1.8
Castor oil.....	963	275°	16.4	3.0
Linseed oil.....	959	285°	3.2	1.7
Tallow.....	951	265°	5.2	2.5

—D. A. S.

#### *Mineral Lubricating Oils.* A. Kunkler. Dingl. Polyt. J. **274**, 323—328.

For the first portion of this article, see preceding abstract. From the considerations previously adduced, it appears that the ordinary method of judging the lubricating value of an oil from its specific gravity is untrustworthy, and does not permit the classification of oils from various sources as light, normal, and heavy oils. Even in conjunction with the flashing point, a separation into groups is not practicable in the case of American oils; with Russian oils a partial classification is possible. The experiments show that no direct relation exists between the viscosities and flashing points of oils, even from the same source. American and Russian oils may be distinguished by their behaviour on cooling. American oils all solidify at about 0° C., previously depositing solid paraffin and, in the case of clear samples, becoming turbid. Most Russian oils remain liquid down to about -10° C., and solidify to a clear transparent mass, without previous precipitation of paraffin. Cylinder oils contain, on an average, 23 per cent. by volume of substances boiling at 310° C.; whilst machine oils only contain 7 per cent. of these bodies. The variations in colour of the different oils is very slight. Dark oils exhibit a greenish fluorescence, and in thin layers appear blackish-brown and transparent. The paler shaft and machine oils of American origin generally possess a greenish fluorescence; whilst those of Russian origin have a bluish fluorescence. Pale Russian cylinder oils exhibit a blue shine, whilst the American oils have no fluorescence. American oils may be often distinguished by their superior purity as regards taste, smell, and colour. Pale Russian oils of 0.903 to 0.909 sp. gr., almost without exception, give a flocculent precipitate on standing; and many samples are not clear and possess a disagreeable sweet odour.

The temperature at which vapour is formed can only be approximately determined, and in conjunction with the flashing and burning points affords the means of judging the volatility of an oil, *i.e.*, its stability as a lubricant. Below are given the vapour evolution temperatures, the differences between these and the flashing points, and the differences between the flashing points and the burning points respectively, for two oils of each variety:—For Russian shaft oil, 105°—110°, 54°—62°, 26°—29°; for American, 110°—112°, 77°—80°, 40°—47°; for pale Russian machine oil, 120°—128°, 60°—75°, 37°—45°; for American, 125°, 81°, 39°; for

Russian cylinder oil, 110°—142°, 76°—108°, 27°—58°; for American, 185°—185°, 100°—103°, 53°—64°.

The determination of viscosity gives a means of dividing oils into groups according to their lubricative value. Oils possessing the same specific gravity, flashing point, &c., may yet differ considerably in viscosity. The probable source of an oil may be deduced from its viscosity and other data. Cylinder oils and dark machine oils of Russian origin exhibit with a higher flashing point and specific gravity, also a higher viscosity; whilst American oils possess with a higher viscosity and flashing point, a lower specific gravity. Pale Russian machine oils compared with American samples of the same flashing points and specific gravities, possess a distinctly higher viscosity. The German mineral oils from Alsace and Oelheim (Hanover) are similar to the Russian oils in so far that the viscosity increases with the specific gravity and flashing point. As regards their behaviour on cooling, they approximate more to American oils. Concerning the diminution of viscosity with increase of temperature, it is to be noted that between the temperatures 20°—50° C. and 50°—100° C. it is more rapid for Russian shaft and pale machine oils than for the corresponding American oils. On the other hand the viscosity of American cylinder oil diminishes more rapidly than that of Russian cylinder oil between the temperatures of 70°—100° C. and 100°—150° C. The diminution in viscosity is proportionately larger for cylinder oil between 70°—100° C.; and for shaft oil between 20°—50° C., than between 100°—150° C. and 50°—100° C. respectively. Several animal and vegetable oils used for lubricating purposes were examined. It was found that, with the exception of castor oil, they all had at 50° C., a smaller viscosity than mineral oil of the same or lower specific gravity. On the other hand there was a much less tendency to vaporisation, and, particularly at high temperatures, a smaller diminution in viscosity than in the case of mineral oils. Finally, the author describes some precautions to be observed in the determination of viscosity at different temperatures.—H. T. P.

#### PATENTS.

*Improvements in the Preparation or Manufacture of Petroleum for Domestic and other Purposes.* P. Smith and J. Pearson, Manchester. Eng. Pat. 3044, February 20, 1889. 8d.

This invention is for making solid or semi-solid petroleum or paraffin oils. After the oil has been deodorised by electrolysis, it is heated to 250° F., and 3 to 5 per cent. of powdered desiccated soap is stirred in, the temperature raised to 400° F. for a few minutes, and the product run into moulds to cool, when it solidifies.

The claim is made for the products and for the method of preparation.—D. A. S.

*Improved Cleansing, Scouring and Bleaching Compound for Wool and other Fabrics, also applicable to Cleansing Purposes generally.* A. H. Parker, London. Eng. Pat. 9410, June 6, 1889. 4d.

See under VI., page 174.

### XIII.—PAINTS, PIGMENTS, VARNISHES, AND RESINS.

#### PATENTS.

*Improvements in or relating to the Manufacture of White Lead.* H. B. Condy, Battersea. Eng. Pat. 18,705, December 21, 1888. 6d.

LEAD suitably granulated is covered with dilute acetic acid, which is run off after 4 to 6 hours, and the lead left to oxidise by contact with air during another 4 to 6 hours. On covering the metal again with the dilute acetic acid, the oxide formed becomes dissolved and a fresh quantity is produced by running off the acid and further exposure to air. The treatment is continued until the solution of basic acetate of lead has acquired a specific gravity of 1.145, when it is treated in a separate vessel with carbonic acid. The carbonate of lead is separated in a filter and then suspended in water, to which sufficient alkaline carbonate is added to ensure precipitation of all soluble lead compounds. The white lead is finally filter-pressed and dried. Carbonic acid may be obtained by heating bicarbonate of soda, which yields the readily saleable carbonate as a by-product, or from well washed chimney gases. The liquor filtered from the washed white lead is evaporated for the recovery of the acetate of the alkali.—G. H. B.

*Improvements in the Manufacture of Paints and Pigments.* R. Stone, London. Eng. Pat. 1578, January 29, 1889. 4d.

THE "invention consists in novel methods of manufacturing paint for general purposes," "China clay, marble, spar, shale, partly matured river mud, Devonshire or Staffordshire clay, or any other substance of a fine or silky nature," being used for the body of the paint; one or more of these substances is then mixed with one or more of the following liquids:—petroleum oil, petroleum spirit, sulphuric acid, nitric acid or sodium silicate. After burning, the material is ground and mixed with oil and dryers, or with sodium silicate only in the usual manner. One ton of the clay or marble, &c. requires about 12 gallons of any of the liquids.—W. M.

*Improvements in and relating to Plastics, more especially designed for Imparting to Glue a Permanent Elasticity, and adapted to be used for various Purposes.* R. W. Johnson, New Brunswick, New Jersey, U.S.A. Eng. Pat. 18,503, November 19, 1889. 4d.

THE composition consists of glue or gelatin, glycerin, and sulpho-oleate of sodium or potassium. When the composition is to be used for printers' rollers, stoppers, &c. the proportions taken are: 1 lb. of glue and  $\frac{3}{4}$  lb. each of glycerin and sulpho-oleate of sodium or potassium. To prepare the compound, the glue is dissolved in as small a quantity of hot water as possible, the glycerin added, and the two thoroughly mixed. The sulpho-oleate is then added, and the mixture heated till all the water used to dissolve the glue has been driven off.

For many purposes the glycerin may be omitted, and the composition be made of 1 part of glue to  $1\frac{1}{2}$ —2 parts of sulpho-oleate of sodium. The composition may also be used as an ointment, in combination with vaselin or oil or other fat and suitable drugs.—B. H.

*A New Composition to be used as a Substitute for Ivory, and for other Purposes.* I. Appletree, Sheffield. Eng. Pat. 18,090, November 13, 1889. 4d.

THE material is composed of the following constituents:—granulated or pulverised ivory, 80 parts; isinglass, 20 parts; French chalk, 5 parts; water, 40 parts; gin, 2 parts; cotton wool, 2 parts. The mixture is placed in a boiler, heated, and constantly stirred for about 10 hours, until it assumes the consistency of paste. It is then removed from the

boiler, placed upon a cool slab, and subjected to a short process of rolling. The prepared paste is then put into a suitable mould or moulds, and subjected to great pressure. Instead of genuine ivory dust, that from walrus or similar teeth may be used, and for an inferior quality, the ivory may be substituted by bone of the kind now used for handles.—B. H.

### XIV.—TANNING, LEATHER, GLUE, AND SIZE.

#### PATENTS.

*A New or Improved Machine for Extracting the Hair from Skins.* L. D. S. Cullin and G. Hughes, London. Eng. Pat. 16,545, November 14, 1888. 8d.

THIS invention relates to an improved machine for extracting the hair from skins. The skin is carried along on a band or web of leather or other suitable material, mounted on a frame, to which it is held by a clip, between two feeding rollers. The front end of the frame is provided with a roller of india-rubber, over which operates a helicoidal nipping and pulling device, furnished with four blunt knives, and revolving in a reverse direction to that of the india-rubber roller. A curved knife is mounted on the frame, and serves to raise the hair on its way to the pulling device. Drawings are given.—B. H.

*Improvements in or connected with Cylinders or Rotating Vessels for Use in Tanning or otherwise Treating Hides or Skins, Washing, Emulsifying, Churning, or like Operations, applicable also for the Treatment of Materials in Vacuo or under Fluid Pressure or the like.* B. Nicholson, South Norwood, and T. Palmer, Sevenoaks. Eng. Pat. 1345, January 24, 1889. 11d.

THE object of this invention is to provide cylinders or drums for use in treating hides or skins. The cylinder or drum is provided with hollow axles at one or both ends, through which a pipe, divided by a longitudinal partition into two passages, passes to the interior of the drum, through one of which passages air or other fluid pressure may be introduced or withdrawn, the other passage being employed for the introduction or withdrawal of the tanning or other liquor employed. The hides are supported in the drum by means of buttons carried on angled rods attached to lengths of tubing mounted on the tie-rods, which are connected to the opposite heads or ends of the drum.

For details, the drawings accompanying the specification must be consulted.—B. H.

*A New Process for "Discolouring" Tannic Acid Solutions.* H. J. Haddan, London. From J. Landini, Hamburg, Germany. Eng. Pat. 17,436, November 2, 1889. 4d.

THE object of this invention is to "discolour" tannin solutions, such as are used in tanneries, by treatment with nitrate of lead, afterwards adding alum and borax. The nitrate of lead solution is added to the boiling solution of tannin in small quantities at a time, and with continual agitation, till the "discolouring" is complete. The solution is then withdrawn, cooled down, neutralised, and filtered.

—B. H.

## XV.—AGRICULTURE, MANURES, Etc.

*On the Relation between the Physical Characteristics of Plants and the Richness of the Soil in Fertile Constituents.* G. Ville. *Compt. Rend.* **109**, 628—631. (Compare this Journal, 1890, 85.)

THE author wishes to determine the changes undergone by plants when the soil is deficient in some one of the four constituents necessary to vegetable life, viz., phosphate of lime, potash, lime, and nitrogenous matter. The characteristics studied are—

(1) Colour; (2) Height; (3) Total weight; (4) General appearance. The plant chosen as the type for experiment was hemp.

(1) *Colour*.—This subject has been dealt with in the former note, and the author only adds that the effects of change of nutrition on colour are generally most evident at some particular period of its development for each plant; for certain species the month following germination gives the maximum of contrast.

(2) *Height*.—A most important characteristic, affording a good criterion of the fertility of the soil. The height of each plant under similar conditions varies but little from one year to another if taken at the same period of each year. The same order of height is maintained throughout the year by plants under dissimilar conditions. With plants of which the nitrogenous matter is the most important constituent, the want of this matter makes the greatest difference in the height.

(3) *Weight*.—The weight of the crop in the dry state gives no less sure an indication than colour and height. The variations in absolute weight from one year to another of plants under the same treatment amount to from one

on two tenths; but as before, the relative order for plants under different treatment is the same.

(4) *General Appearance*.—Determined by photography.

A coloured plate is inserted in the text showing the photographs, the colour, height and weight of yield of hemp plants grown under different conditions in the year 1884.

	Height in Metres.	Weight of Yield in Kilogrammes.
Manure, "stimulating" (100 kilos. of nitrogen).....	1'53	11'220
Manure, "complete" (75 kilos. of nitrogen).....	1'20	11'150
Manure containing no nitrogen ..	0'61	4'740
Manure containing no phosphates	0'97	8'220
Manure containing no potash ....	0'40	5'220
Manure containing no lime.....	1'15	10'570
Soil without manure.....	0'18	2'175

The author has found that the amounts of carotene and chlorophyll in plants seem to be always proportional to one another, and afford a further criterion with regard to the nature and quantity of the fertile constituents contained in the soil.

It remains to be decided if the colour of chlorophyll solutions is proportional to their strength, as is the case with carotene. By a colorimetric method  $\frac{1}{10}$  milligramme of carotene can be estimated in its solution in bisulphide of carbon. [Carotene is extracted from the plant by petroleum ether, and after evaporation *in vacuo* the residue is redissolved in bisulphide of carbon to form an orange solution.]

### ESTIMATION OF CAROTENE (in milligrammes per 100 grms. of leaves) IN THE FOLLOWING PLANTS:—

	Plants in which the Nitrogenous Matter predominates.				Plants in which the Potash predominates.	
	Hemp.	Wheat.	Rape.	Betroot.	Potato.	Vine.
Manure, "stimulating" .....	350	195	183	183	204	..
Manure, "complete".....	315	167	..	164	173	200
Manure containing no nitrogen .....	199	74	114	120	159	177
Manure containing no phosphates .....	281	97	..	131	162	179
Manure containing no potash.....	254	104	..	140	124	125
Manure containing no lime.....	21	114	..	183	154	167
Soil without manure .....	250	66	..	143	94	121

The author hopes that farmers will adopt his plan of observing the four chief characteristics of plants to define the nature of their crops each year. He proposes to continue his researches on the estimation of carotene and chlorophyll.—P. J. H.

*On the Role of Ammonia in the Nutrition of the Higher Forms of Plant Life.* A. Müntz. *Compt. Rend.* **109**, 646—648.

THE object of this paper is to prove that plants can obtain their nitrogen directly from ammonium salts without these being first converted into nitrates.

Kühnmann at one time maintained that nitrates were always converted into ammonium salts (by means of

reducing actions going on in the soil) before they could yield their nitrogen to plants, and these views were held even after Boussingault had shown that nitrates are directly absorbable by plants.

At the present time the exactly opposite theory tends to prevail, and it is generally supposed that ammoniacal nitrogen must be converted into nitrate before it can be absorbed.

The difficulty of deciding as to the correctness of this theory lies in the presence of the nitrification ferment, which always converts a certain portion of the ammonium salt used as manure into nitrate.

The author has therefore devised the following means for growing plants on a soil in which the only nitrogen supplied as manure is in the form of sulphate of ammonia, all nitrification being prevented.

A certain quantity of natural soil was freed from nitrates by washing, manured with sulphate of ammonia, put into large pots, and then heated in an oven to 100°, a temperature amply sufficient to kill the nitrification ferment. To prevent future inoculation by the ferment the pots were placed in large cages partly closed in with glass and partly with cloths, which served to filter the air from all germs. The sides of the cage were smeared with glycerin, so that the arrangement was exactly similar to that devised by Tyndall.

The seeds were immersed for a short time in boiling water before being sown, so as to sterilise their surfaces. All these operations were performed in a closed room, of which the atmosphere had been purified by spraying with water, and then leaving the dust to settle. The cages containing the pots were put under an open shed, and were watered with sterilised water.

In order to have some means of comparison, another set of pots was prepared in exactly the same way, and these were then inoculated with the nitrification ferment by means of a minute quantity of compost.

Experiments carried out during the years 1885—88 gave identical results. No trace of nitrate was ever formed in the sterilised pots. Two examples are quoted in illustration. The results are given in milligrammes of nitric acid per kilogramme of earth.

Earth free from Nitrification Ferment.		Earth inoculated with Nitrification Ferment.	
Initial State.	Final State.	Initial State.	Final State.
Mgr. 0.0	Mgr. 0.0	Mgr. 0.0	Mgr. 91.2
0.0	0.0	0.0	420.0

The growth of the plants was as a rule satisfactory, maize, the bean, kidney bean, and hemp attaining a height of over 1 metre.

By determining the proportion of nitrogen in specimens of the plant, and subtracting the amount present in the seed, the total quantity directly absorbed in the form of ammoniacal nitrogen could be determined. The following examples are quoted:—

	Nitrogen		
	In the Seed.	In the Plant.	Derived from Ammonium Salt.
Bean.....	37	956	915
Kidney bean....	16	105	89
Maize.....	3	211	208
Barley.....	7	50	49.3
Hemp.....	5	115	114.5

These experiments prove conclusively that the higher plants are capable of absorbing ammoniacal nitrogen directly by means of their roots, and hence that the nitrification of ammonia manures is not an indispensable condition for their being utilised.—P. J. H.

#### *The Artificial Manure Manufacture.* W. Cohn. Chem. Ind. 12, 459—461.

As the author predicted two years ago (Chem. Ind. 10, 444), the successful use of the cheaper potash salts and Thomas phosphate powder has been accompanied by an increase in the use of artificial manures generally. In 1888, 3,508,450 centner (about 173,650 tons) of Chili saltpetre was imported into Hamburg; in 1887 the imports of this article had been as much as 5,084,850 centner (250,325 tons); and in 1889 the imports had again increased over 1888; at the beginning of September the amount was 4,427,900 centner (217,896 tons).

The use of sulphate of ammonia has increased in like manner, and renewed efforts have been made to collect the ammonia formed in the process of coke-burning, efforts which are becoming more and more successful. Maercker has recently (Dent. landw. gesell. Mittheil. 11, 1889—1890) published the results of experiments on the effect of nitrate of soda and sulphate of ammonia when used both in small and large quantities for corn, beetroot, and potato crops, which show that ammonium sulphate has at least an equal value to the sodium nitrate. In the case of superphosphates an improvement in the process of manufacture is hardly looked for. In the price, however, such an improvement has been realised, the manufacturers having combined together to obtain a proper remuneration for capital invested. Bone meals, both raw and boiled, continue to hold the position they have attained, the steamed bone being more and more sought after. At the recent annual meeting of the Deutsche Versuchs-Stationen at Speyer the question of adulteration of bone meals was fully discussed and a definition of what bone meal should be was arrived at. The property of absorbing ammonia said to be possessed by gypsum formed in the manufacture of superphosphates, has encouraged several manufacturers to introduce this waste material into the market.—J. W. L.

#### PATENTS.

*Improvements in the Means or Method of Treating or Preparing certain Refuse Matter to increase its Fertilising Properties.* W. Riddiough, Bradford. Eng. Pat. 1710, January 31, 1889. 4d.

The improvement consists in grinding or crushing "night-soil," whereby its activity and value as a manure are increased.—E. J. B.

*A Compound of Animal Substances and Earths containing Phosphate of Iron or Alumina for Fertilising Purposes.* N. B. Powter, New York, U.S.A. Eng. Pat. 14,633, September 17, 1889. 4d.

The fertilising compound is formed by mixing some powdered phosphatic rock containing alumina or iron, such as Redonda phosphate, with acid and wet animal matter such as undried meat, skin, blood, hair, animal filth, &c.

—E. J. B.

*A New or Improved Insecticide for the Treatment of the Diseases of the Vine and other Trees and Plants.* J. B. Alzugaray, Porto, Portugal. Eng. Pat. 15,539, October 3, 1889. 4d.

This insecticide, entitled "Phylloxerine or Anti-phylloxerine Liquor," consists of a mixture of petroleum, shale or tar oils, with sulphur or sulphides of calcium or sodium.

—D. A. S.

*Improvements in Fertilisers.* E. and H. Saxl, Vienna, Austria. Eng. Pat. 18,918, November 25, 1889. 4d.

This invention consists in drying and then pressing into cakes the excrements of domestic animals, in which form the material can be easily and cheaply transported. The cakes may be ground and reduced to powder of any required degree of fineness.—W. M.

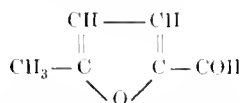


## XVI.—SUGAR, STARCH, GUM, Etc.

*New Relation between Sugars and Furfuryl Derivatives. Constitution of Methylfurfural and Isodulcitol.* Maquenne. *Compt. Rend.* **109**, 603—606.

THE author has shown (this Journal, 1889, 974) that *fucus* when distilled with dilute sulphuric acid yields furfural and methylfurfural. There are four possible formulæ for this latter, one in which a  $\text{CH}_2$  group is introduced into the side chain, and the other three in which a  $\text{CH}_3$  group is attached to one of the carbon atoms of the ring, thus displacing a hydrogen atom. It yields acetic acid on oxidation, and so it must be one of these latter.

Isodulcitol distilled with four times its weight of sulphuric acid  $15^\circ\text{--}20^\circ\text{B.}$ , yields a methylfurfural identical with that obtained as above, together with small quantities of acetone, but not a trace of furfural. Fischer and Tafel have shown that isodulcitol is an aldehyde of normal hexane, and Herzig found that it yielded acetic acid on oxidation, showing that it contained a methyl group at the extremity of the chain. The production of methylfurfural from isodulcitol must be brought about by joining the chain so that this methylfurfural must be represented by the formula—



As arabinose yields, by similar treatment, furfural, isodulcitol may be considered as  $\omega$ -methylarabinose.

Although the yield of methylfurfural from isodulcitol is small, yet its production may be employed as a test when it is not possible to crystallise the sugar.

On distilling a quantity of any body containing isodulcitol with sulphuric acid ( $20^\circ\text{B.}$ ), the less volatile portion of the oily distillate gives a green colouration with alcohol and sulphuric acid, which is characteristic of methylfurfural.

—A. L. S.

*On the Fermentation of Raffinose in Presence of different Kinds of Yeast.* D. Loisean. *Compt. Rend.* **109**, 614—615.

THE author gives extracts from a sealed paper deposited by him with the Academy in March 1888, which contains an account of similar experiments to those described recently by Berthelot (this Journal, 1889, 933), showing that raffinose under the influence of a weak yeast yields a glucose and saccharose, the former of which only ferments.

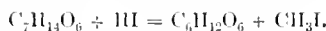
—A. L. S.

*On Two New Sugars from Quebracho Bark.* G. Tanret. *Compt. Rend.* **109**, 908—910.

COARSELY powdered quebracho-bark (*Aspidosperma quebracho*) is mixed with milk of lime and lixiviated with 50 per cent. alcohol. The solution is reduced by distillation and evaporation to a bulk of 500 cc. per kilo. of bark used, neutralised with acetic acid and treated with "Extract of Saturn." From the filtered liquid the sugar is precipitated by means of ammoniacal lead acetate. The lead precipitate is decomposed with dilute sulphuric acid, the solution is concentrated to a syrup, and then dissolved in the least possible quantity of 90 per cent. alcohol. Ether is now added, which precipitates a sticky mass. The precipitate is redissolved in water, concentrated and left in a cool place for several days, when fine crystals separate. The crystals are washed with a little dilute alcohol, dissolved in water and decolourised by means of animal charcoal. The concentrated solution is dissolved in boiling 60 per cent. alcohol, which on concentration deposits crystals of the new sugar. The yield is about 1 grm. per kilo. of bark used. The author names this sugar *Quebrachite*. Its

formula is  $\text{C}_7\text{H}_{14}\text{O}_6$ . It crystallises in anhydrous rhombic prisms, and is very sweet to the taste. It is very soluble in water, and pretty soluble in boiling alcohol, even when absolute, but insoluble in ether. Its density is 1.54. It melts at  $186^\circ\text{--}187^\circ\text{C.}$  and boils in vacuo at about  $210^\circ\text{C.}$ , at the same time subliming in beautiful needles. It is levo-rotary,  $(\alpha)_D = -80^\circ$ .

Quebrachite is only fermentable under the action of beer-yeast. It has no action on Fehling's solution, but reduces ammoniacal silver nitrate in the cold. Boiling dilute solutions of acids and alkalis appear to have no effect on it. It is not precipitated by basic acetate of lead, but only by ammoniacal lead acetate. Strong sulphuric acid dissolves it very slowly at ordinary temperatures with but little colouration; but at  $100^\circ\text{C.}$  it dissolves more quickly, quebracho-sulphuric acid being formed. This acid is levo-rotary and forms soluble in crystallisable calcium and barium salts. Quebrachite yields an acetic ether melting at  $89^\circ\text{C.}$ , and also a very unstable nitro-derivative, both of these compounds are levo-rotary. Heated with hydriodic acid, quebrachite forms methyl iodide and a new inosite, thus:—



This body crystallises in brilliant prismatic needles, containing two molecules of water of crystallisation, which it loses rapidly on exposure to air. The author names it "*Levo-inosite*." It is soluble in 2.3 parts of water at  $12^\circ\text{C.}$ , slightly soluble in boiling alcohol, but insoluble in ether. It melts at  $238^\circ\text{C.}$ , and boils in vacuo at  $250^\circ\text{C.}$  with volatilisation, being, however, less volatile than quebrachite. Its rotary power,  $(\alpha)_D$ , is  $-55^\circ$ .—H. T. P.

*Advances in the Manufacture of Starch, Dextrin, Glucose, &c.* Dingl. Polyt. J. **274**, 328—334.

THE combined grater and grinding mill used in the manufacture of potato starch seems to have no mean rival in the shape of H. Schmidt's *compound-grater* (Ger. Pat. 45,284). O. Saare (Zeits. f. Spiritusind. **12**, No. 24) publishes the results of a number of comparative trials made with the two forms of apparatus at H. Schmidt's experimental works. The compound-grater employed consisted of a mantle of perforated sheet-iron surrounding the inner rasping drum. The mantle was grated alternately inside and outside by striking the perforations alternately from the outside and inside respectively. The whole apparatus was so arranged that the compound-grater could be fixed in its place or removed with very little trouble. The rasping drum had a diameter of 400 mm., and was provided with new gratings having 18 teeth to the inch. Four experiments were made, 10 kilos. of washed and superficially dried potatoes being used each time. The potatoes contained 17.7—17.9 per cent. of starch. The rate of grinding was the same in each case. The ground potatoes and washings were collected in a barrel and filtered through two sieves, the second being the finer of the two. The milky filtrate was allowed to settle for a day in another barrel, when the starch was separated, dried, and weighed. The pulp remaining on the sieves was also dried and weighed, and the starch left in it estimated. The first experiment was made with a carefully-mounted rasping drum without the compound-grater; the second was made with a well-set drum fitted with the compound apparatus. The third and fourth experiments were made with a badly-fitted-up rasping drum, provided without and with the compound-grater respectively. The results obtained are given below. In the first experiment there were obtained 395.5 grms. of pulp and 1,521.9 grms. of starch; in the second trial 356.3 grms. of pulp and 1,563 grms. of starch. Further, in the first case, the pulp contained 264 grms. of bound starch, and, in the second case, 223 grms. of bound starch. The third experiment yielded 446.5 grms. of coarse and 42.8 grms. of fine pulp, and 1,428.4 grms. of starch. The fourth trial gave 346.1 grms. of coarse and 46.3 grms. of fine pulp, and 1,532.5 grms. of starch. These results show that the compound-grater compensates for the defects of a badly-mounted rasping drum, and raises the yield of starch on a level with that obtained by means of a first-class

resp. As regards the extra power required on addition of the compound-grater, experiments show it to be 1.2 indicated horse-power, whilst a grinding mill requires 1.5—2 horse-power. Saare condemns the use of centrifugal machines in damp-starch factories, chiefly on economical grounds; besides which centrifugal starch is said to become mouldy very readily.—H. T. P.

## XVII.—BREWING, WINES, SPIRITS, Etc.

### PATENTS.

*Improvements in the Treatment of Commercial Alcohol.*  
H. E. Sorel, Paris, France. Eng. Pat. 17,338, November 28, 1888. 11d.

In this invention the following two operations are carried on concurrently:—

- (1.) Partial and successive distillations of concentrated products.
- (2.) Partial and successive condensations at high temperature.

These two operations follow from the same physical law, which may be stated:—There exists for each volatile body, and for each temperature, in the presence of a given solvent which does not act chemically on the body when brought to the same temperature, a definite and fixed relation between the quantity of body existing in the form of vapour in a certain weight of vapour in contact with the liquid, and the quantity of the same body dissolved in the same weight of the liquid. This relation is defined as the *partage coefficient*. When the partage coefficients of a volatile liquid and its solvent are very nearly equal, it is impossible to separate them by distillation.

Now, by so arranging the strength of the alcoholic liquid to be treated, the temperature of distillation can be varied between 79° and 100° C., and so the relative partage coefficients may be altered as desired.

Under the conditions under which the invention is worked, alcohol of such a concentration is used that one-half or two-thirds of the alcohol may be distilled off without materially altering the boiling point.

Employing a continuous distillation column, the walls of which are protected from radiation, and passing into it at the top alcohol, between 90° and 70° Gay-Lussac, previously raised to its boiling point, the alcohol passing from shelf to shelf to the boiler at the bottom, the inventor has verified the following relation:—

$$S_1 = S_m \left\{ 1 + \frac{\left( \frac{Kp}{P} \right)^{m-1} - 1}{\frac{Kp}{P} - 1} \right\}.$$

between  $S_1$  = the proportion of a certain impurity existing in a state of solution in the liquid of the first upper shelf, and  $S_m$  = the proportion of the same impurity in the liquid of  $m^{\text{th}}$  shelf,  $K$  being the partage coefficient for the liquid and  $P$  being the proportion of this impurity in the vapours passing from bottom to top to that in the liquid passing from top to bottom.

The inventor has verified this in practice, and finds that he obtains under the above conditions all the impurities having a partage coefficient greater than unity in the products of distillation, whilst those having a partage coefficient less than unity are obtained in the non-distilled alcohol.

It must be noted that in this case the distillation column does not work as an ordinary distillation column because there is not, so to speak, any expenditure of liquid until it arrives in the vaporising boiler. The vapour traverses all the shelves without notable condensation, and the proportion between the amount of vapour and liquid remains approximately constant.

The vapour escaping from the top of the distillation column, and containing, as above stated, only those impurities having a partage coefficient greater than unity, are either passed into an ordinary distillation column worked in the usual way or into the partial condensers. These consist of a series of tubes of elliptical section arranged so that the liquid condensed in each series may be collected separately. They are immersed in water contained in a series of tanks, the temperature of the one containing the condenser into which the vapours first pass is kept at 77°—79° C., and the temperature of each succeeding one a few degrees lower until the last one, which is kept cold.

The inventor even finds it advantageous to so arrange the heating of the tanks that the bottom of one is kept at the same temperature as the top of the next, the temperature gradually rising from the bottom to the top of each tank and he separates the series of condensers contained in each tank into elements, each element consisting of one horizontal layer of pipe, and the liquid from each element is collected separately.

The products condensed at a low temperature, and containing a large quantity of volatile products, are treated with oxide of lead, or plumbate of soda or potash. As there is some difficulty in separating the lead by filtration, he adds some such neutral salts as chloride of magnesium or manganese, soluble in alcohol, and capable of forming a flocculent precipitate with the alkaline earths or their carbonates.

One of these chlorides is added to the liquid containing the lead and then milk of lime or some similar substance, when a flocculent precipitate is formed which rapidly settles, taking the lead with it. The clear liquid may now be distilled.

The claims are:—

“1. Partial distillation in a continuous distillation column of the crude phlegms or alcohols in course of manufacture more or less diluted with water, so as to separate these phlegms or alcohols into vaporised products containing principally the more volatile constituents of the mixture, and non-vaporised products containing principally the less volatile constituents.

“2. Application to distilling apparatus and to rectifiers, of condensers forming series, each maintained at a constant temperature, each condenser of each series having its separate outlet.

“3. Successive additions to the crude phlegms or alcohols in course of treatment of a composition of oxide of lead, then of a salt soluble in alcohol and capable of being precipitated in flakes by magnesia, lime, the alkalis or the alkaline carbonates, and, finally, one of these reagents; this succession of chemical operations having for its object the combination into a filterable precipitate, of a part of the ethers and analogous bodies.”—A. L. S.

*Improved Process for Aërating and Purifying Beer Worts and Apparatus therefor.* O. Imray, London. From A. Bergh, Copenhagen, Denmark. Eng. Pat. 86, January 2, 1889. 8d.

In order to effect the cooling and aërating of wort in a sterile condition, the hot wort is led into a rotating centrifugal machine which is contained in a closed case. From this the wort is taken by a pipe, whose inlet is placed just below the rim of the machine, into some convenient closed cooling apparatus and thence to the fermenting vessels. The wort is free from grounds as these remain behind in the centrifugal machine. Air is admitted into the case surrounding the centrifugal machine through a cotton-wool filter and the whole apparatus is sterilised by steam before use.

The centrifugal machine is also useful for removing the yeast remaining when fermentation has proceeded as far as necessary, the after-fermentation being brought about by the introduction of a suitable quantity of some pure ferment.—A. L. S.

*A Vinegar Generator and Filter.* R. H. Herder, Chicago, U.S.A. Eng. Pat. 570, January 11, 1889. 8d.

THIS patent has for its object the more speedy acetification of the mash and the filtration of the product. The apparatus consists of a cask hung on trunnions so as to swing in a vertical position. At one end of the cask are fitted three perforated false bottoms, the space between the two nearest the head of the cask is filled with some filtering material, such as sand, charcoal, &c. Bungholes and stoppers are inserted where necessary. The interior between the inside false bottom and the opposite head is filled with wood shavings. Four check-valves for admitting air are inserted, one in each head and the other two in the side of the cask about two-fifths of the length of the cask from each end, and they are so arranged that the two lower ones are always closed and the two upper ones open. The cask is half filled from the top with a suitable fermented or distilled mash. Acetification takes place rapidly in the upper part, where a certain amount of liquid adheres to the shavings, the air finding its way in through the check-valves. After four hours or so the cask is inverted and acetification takes place in the other half of the cask, the other pair of check-valves coming into operation, and so on until the acetification is completed, when the vinegar is run out through the filter at the bottom of the cask.

—A. L. S.

*Improvements in the Treatment of Commercial Alcohols.* H. E. Sorel, Paris, France. Eng. Pat. 770, January 15, 1889. 8d.

ACCORDING to the formula given in Eng. Pat. 17,338 of 1888 (see previous page) for determining the ratio between the impurities on an upper stage of the column to the impurities on a lower stage, *i.e.*, the velocity of conveyance of impurities from the bottom to the top of the column, it is seen that this ratio is not dependent on the term  $K, p, P$ , taken separately, but on the ratio  $K \frac{P}{p}$ . The inventor has

found by experiment that the ratio  $\frac{P}{p}$  is almost independent of the absolute value of the reflux returning from the top of the column, but depends essentially on the temperature; being low, between 95° and 100° C., increasing very rapidly to 80°—82° C., and then decreasing rapidly; whilst as the temperature falls  $K$  diminishes. On the whole the ratio  $K \frac{P}{p}$  increases from 90° to 80—82° C., and then decreases very rapidly to 78·7° C., the boiling point of strong alcohol. So, the lower the temperature of the lower stages of the rectification column, the better will the tail products be retained, as the term  $K \frac{P}{p}$  is then kept as small as possible.

It may also be noted from the above considerations that the stage where the temperature of the column ranges from 80°—81° C. serves to mark off the part where the tail products are constantly returned and turned back towards the bottom, and that part where they tend to rise faster than the alcohol; so that it is advantageous to be able to separate the vapours condensing at this stage from the rest.

In putting these considerations into practice the inventor reduces the functions of the analyser to simply maintaining the temperature of the vapours at the proper point, and so create as little reflux as possible. The portion of the column which is not refrigerated is surrounded with some non-conducting covering.

The vapours enter at the bottom of the column, passing over the surface of the return liquids; they then rise into the regulating refrigerator which consists of two sections. The lower one is filled with a number of hollow earthenware cylinders, open top and bottom, and arranged in quincunx form, one above the other in horizontal stages. The upper section contains several horizontal series of copper tubes, traversed by hot water from the analyser, the tubes being placed very close together and their surfaces plaited and undulated, so as to distribute the condensed liquid as regularly as possible over the cylinders of the lower stage.

Between these two sections the temperature is 80°—81° C., the stage at which it is pointed out above the tail products tend to accumulate, and an arrangement is inserted for separating off at will the liquid condensing here.

The vapours then enter the rectifying column properly so called, which consists of two sections of similar construction to those forming the regulating refrigerator, except that the one filled with the earthenware cylinders is longer. The vapours issuing from the top of the column are either passed into an ordinary refrigerator or some such arrangement as is described in the abstract above referred to.

The claims are:—

"1. The improvement in rectification or dephlegmation columns for the treatment of commercial alcohols consisting in adapting at the lower part thereof a refrigerating apparatus.

"2. In combination with refrigerating apparatus applied as aforesaid, provision for the continuous withdrawal of impure alcohol.

"3. The method of and the apparatus for the treatment of commercial alcohols."—A. L. S.

*Improved Alcoholic Beverages.* A. Malzy, London. Eng. Pat. 962, January 18, 1889. 4d.

THE invention consists in the preparation of mixtures of alcoholic liquids and aerated or other waters in definite proportions, having regard to quantity and quality, in vessels so that the liquid can be easily and quickly retailed ready mixed. The process of mixing can be effected before, during, or after aeration. (Compare this Journal, 1887, 221.)

—A. L. S.

*Improvements in Apparatus for promoting Circulation and Aeration of Brewer Worts.* G. G. Cave, Fenny Stratford, and J. H. Howell, Bristol. Eng. Pat. 1942, February 4, 1889. 6d.

TWO "steam boiling coppers" are employed, they are placed side by side, and are surmounted by a "curb" or pan which extends over both vessels, communication being maintained between the bottom of the coppers by a pipe. The wort in one of the coppers is maintained at a higher temperature than the other, and consequently in boiling overflows and is conducted by the "curb" into the other copper, whilst an equal quantity passes by the pipe into the first copper, thus maintaining a constant circulation. A second pipe provided with a valve connects the coppers just below the curb, so that when, owing to evaporation, the contents of the first copper do not overflow into the second, the circulation may still be kept up by opening up this pipe. Instead of using two coppers the low-temperature one may be replaced by a vat or other vessel of copper or any other suitable material.—A. L. S.

*Improvements in Shavings for Vinegar Generators and for Clarifying Beer.* R. H. Herder, Chicago, U.S.A. Eng. Pat. 3362, February 25, 1889. 8d.

THE improvements consist in the production of shavings to be used in the manufacture of vinegar, &c., which shall expose more surface than the ordinary flat coiled shaving.

To this end the shavings are planed with transverse or longitudinal ribs, with corrugations, serrations, or any combination of these. In this way there remain, when the shavings are coiled, channels between the coils formed by the ribs, &c., through which the wort and air may pass. The patentee claims that the wort takes much longer to trickle over these shavings, thus exposing it a much longer time to the atmosphere and thereby increasing the working capacity of the generator.—A. L. S.

*Improvements in or relating to the Preservation of Beer and the like.* A. J. Boulton, London. From H. F. L. Otto and G. B. Schwerdtfeger, Doebeln, Saxony. Eng. Pat. 15,401, October 1, 1889. 4d.

THE inventors coat the interior of the cask with paraffin wax which has been impregnated with salicylic acid, which they state prevent the entrance of all organisms from the exterior air into the beer.—A. L. S.

*A New or Improved Pasteurising Apparatus.* W. A. Vollmer, Apenrade, Germany. Eng. Pat. 15,810, October 8, 1889. 6d.

THIS apparatus is for the pasteurising of beer in bulk. It consists of an air-tight vessel surrounded on all sides except the front by a water-jacket, the temperature of which may be raised or lowered by passing steam or cold water through a coil contained in it. Two thermometers, a manometer, a gauge for determining the level of the liquid, a safety-valve, and manhole are attached to the front of the vessel.

The apparatus is filled with beer and the temperature raised by the steam coil to 60° F. and kept constant for some little time, and then by replacing the steam by cold water the beer is cooled until the manometer indicates the normal pressure.—A. L. S.

*Improvements in the Treatment of Brewers' Grains for Cattle Food.* C. P. Goode, London. Eng. Pat. 16,325, October 16, 1889. 4d.

THE dried grains, whilst still hot, are subjected to great pressure in a mould divided into compartments; by this treatment the grains adhere and when cooled form dry coherent cakes suited for transport. When the grains to be treated are cold, they must be heated in the mould. In some cases other nutritious matters are added to the grains before compression.—A. L. S.

*Improvements in Treating and Rectifying or Ayeing Alcohol or Alcoholic Liquors or the like by Electricity.* A. de Meritens, Paris, France. Eng. Pat. 16,917, October 26, 1889. 4d.

THE present patent has reference to Eng. Pat. 13,293 of 1888 (this Journal, 1888, 860). The inventor states that alternating electric currents assist chemical combination between bodies which are in a condition to combine; and that ammonia, aniline, the phenylene diamines, &c., which will combine with the aldehydes at 80° to 100° C. combine at ordinary temperatures with the help of an alternating current. He therefore adds to the alcoholic liquor two equivalents of ammonia or aniline, &c., to each equivalent of aldehyde to be combined, and passes an alternating current, when combination and consequent neutralisation immediately take place.—E. T.

## XVIII.—CHEMISTRY OF FOODS, SANITARY CHEMISTRY, AND DISINFECTANTS.

### (A)—CHEMISTRY OF FOOD.

*Foods and Food Adulterants.* Investigations made under direction of Dr. H. W. Wiley, Chief Chemist. Part Fifth. *Baking Powders.* C. A. Crampton, Assistant Chemist, United States Department of Agriculture; Division of Chemistry; Bulletin 13, 1889, 557—627.

NO recognised standard for the composition of a baking powder exists, and as long therefore as no injurious ingredients are present in such a powder it cannot properly be looked upon as adulterated, whatever its composition. In a case like this, therefore, of a food adjunct, the composition of which is to a certain extent arbitrary, it is desirable that the public should be in possession of data which will enable it to form some opinion as to the relative value of a baking powder. At present the sale of baking powders is not regulated, but it seems desirable that manufacturers should be required by law to use a label giving approximately the composition or analysis of the powder sold. Some regulation of this sort seems only fair to the consumer and the manufacturers of a first-class and therefore high-priced article.

The bulletin under consideration is a step in the right direction, for before useful legislation can be carried out on a matter like this, it is necessary that analytical data should be carefully collected and discussed.

The oldest, and in a sense the best method of leavening bread is by means of yeast. By its means the carbohydrates of the flour are partially decomposed, and the carbonic acid evolved in the process lightens or "leavens" the dough. Excepting the yeast no foreign materials are introduced into the bread, but the process is a slow one, and the road has therefore been paved for the introduction of mechanical and chemical methods to accomplish the aëration, or charging of the dough with gas, more rapidly. One advantage of yeast-raised bread is that the albuminoids get peptonised. In Dauglish's method of aërating, the dough is made in a closed vessel, with water charged with carbonic acid under pressure. When the pressure is relieved the expansion of the gas leavens the bread. A weak wort, in which fermentation has set in, and which absorbs the carbonic acid gas more readily, is also used instead of water. The above method of leavening is therefore largely a mechanical one.

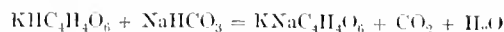
No reliable data exist with regard to the consumption of baking powders; there seems, however, some ground for believing that some 25,000,000 lbs. are paid annually for baking powders by consumers in the United States. Baking powders consist essentially of an alkaline carbonate—bicarbonate of soda is almost exclusively used for this, sometimes bicarbonate of ammonia—mixed with a solid acid or acid salt. Usually some dry inert material, such as starch, is added to prevent action between the chemicals during the time the powder is kept. This is called the "filling."

Baking powders can be broadly and conveniently classified according to their acid constituents:—

- (1.) Tartrate powders.
- (2.) Phosphate powders.
- (3.) Alum powders.

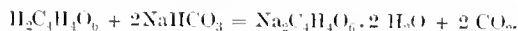
Many powders are mixtures of at least two of these three classes.

*Tartrate Powders.*—In these bitartrate of potassium or "cream of tartar" is usually employed to supply the acid. Sometimes free tartaric acid is used. The reaction brought about on the addition of water to dissolve the constituents would be—



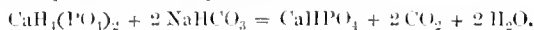
The products of the reaction are therefore potassium, sodium tartrate (Rochelle salt), carbon dioxide and water.

With free tartaric acid the reaction is—



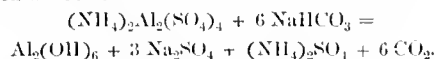
Here the products of the reaction are sodium tartrate and carbon dioxide. Both sodium tartrate and Rochelle salt are slight purgatives. The quantity of Rochelle salt present in a loaf of bread (2 teaspoonfuls of powder to a quart of flour) is probably somewhat more than would be contained in a scidlitz powder. In other respects these salts are harmless, and the tartrate baking powders may on the whole be looked upon as the most satisfactory class of baking powders. Tartrate powders prepared with free tartaric acid probably do not keep so well as those in which the bitartrate of potash is used, hence the latter class is almost invariably met with.

*Phosphate Powders.*—In these monocalcium phosphate is the acid salt employed. It is difficult, however, to obtain this salt pure, and most phosphate powders therefore contain a considerable amount of sulphate of lime. The following equation, probably, fairly well represents the reaction which takes place with these powders:—



The phosphate of soda is a mild purgative, but otherwise, no particular objection can be raised to these powders, and perhaps, in cases of malnutrition, scrofula, rickets, phthisis, &c., the presence of phosphates may be advantageous. Acid phosphate of soda is said to have been used in former years as a constituent of baking powders, but appears to have been entirely superseded by the lime salt.

**Alum Powders.**—In these the salt used in place of an acid is either potash alum ( $K_2Al_2(SO_4)_4 \cdot 24 H_2O$ ), or ammonia alum ( $(NH_4)_2Al_2(SO_4)_4 \cdot 24 H_2O$ ) according to their relative cheapness. At present the ammonia alum is exclusively used. As burnt alum, *i.e.*, alum deprived of its water of crystallization, dissolves more slowly in water; it is probably in this condition that it is used for baking powders. The reaction would be—



The residue therefore consists of sulphate of soda, ammonium sulphate and alumina. Sodium sulphate is a slight aperient. Little is known about the physiological action of ammonium sulphate. With regard to the alumina the following conclusions are drawn :

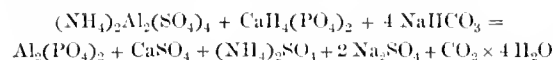
- (a.) It must be expected that small quantities, at least, of alum will be absorbed by the digestive fluids, where any form of powder containing it is used.
- (b.) Whether the absorption of small quantities of alum into the human system would be productive of serious effects is still an open question, and one that careful physiological experiments alone can decide.
- (c.) That form of alum powder in which sufficient phosphate is added to combine with all the aluminium present is a better form, and less apt to bring alum into the system than where alum alone is used.

Most American alum powders are of this latter description. There are few "straight" alum powders, *i.e.*, alum powders containing alum only as the salt which reacts with the carbonate.

In England and France, which have food adulteration laws, the use of alum in bread-making is prohibited.

Sometimes tartaric acid or bitartrate is added to alum powders. This practice is objectionable, as the acid tends to prevent the precipitation of the aluminium hydrate and facilitates the absorption of alumina into the system.

Conclusion (c.) above has reference to the mixed alum and phosphate baking powders. In this case the character of an alum powder would, it seems, be improved. The reaction would be—



and, as will be seen, insoluble calcium sulphate partially replaces sodium sulphate, and the less soluble aluminium phosphate will to a greater or less extent replace alumina.

The following comparison of the different classes of powders is then given: "It is assumed, of course, that the ingredients are combined in exactly the proper proportions, and that all the chemicals used are of full purity and strength.

Powders.	Carbon	Total Residue
	Dioxide.	of the Weight
	Per Cent.	of Chemicals
		used.
Tartrate .....	16	104
Phosphate .....	22	123
Alum .....	27	128
Alum and phosphate .....	17	111

"From this it will be seen that a tartrate powder, theoretically, gives the lowest percentage of carbonic acid gas in proportion to the weight of chemicals used in its composition, together with the least weight of residue. It is assumed that burnt alum is used in both the alum and the alum and phosphate powders. The residues are calculated to hydrated salts in all cases. No account is made of inert "filling," as that would be the same in each case. It should, of course, be remembered that in the above calculation the *total weight* of residue is reckoned in each case without regard to solubility or relative effect upon the system of the various salts formed."

Carbonate of ammonia is sometimes used alone by bakers as a chemical aerating agent. If it were entirely driven off during the baking of the bread there would be no objection to its use. But that this is the case is open to doubt, and it is not therefore advisable to use it alone in baking. An objection can hardly, however, be raised to the small quantities of this substance present in some baking powders.

Then follow analyses of banking powders found in the market and their discussion by Professor H. A. Weber, forming part of the annual report of the Ohio State Dairy and Food Commissioners for 1887. The discussion does not add materially to what has been said so far, and the analyses of 30 samples cannot, of course, be given. Of these, Mr. Crampton says: "Professor Weber's analyses are rather superficial and incomplete, probably being made under conditions that did not admit of thorough quantitative work. He has overlooked entirely, for instance, the fact of the presence of phosphoric acid in many alum powders."

Professor H. B. Cornwall, in his "Report of the Dairy Commissioner of New Jersey, 1888," p. 82, gives the results of the examination of a large number of baking powders, but excepting for the "strength," *i.e.*, the yield of carbon dioxide which was estimated quantitatively, the examination was merely of a qualitative nature.

Sulphates, ammonium salts, phosphates, alumina, tartaric acid and tartrates, and potash were tested for, and the results enabled him to classify the powders into—

1. Cream of tartar powders.
2. Acid phosphate of lime powders.
3. Alum and phosphate powders.
4. Alum powders.
5. Unclassed powders.

Professor Cornwall's conclusions agree with those already given at the beginning of this abstract.

We now come to a consideration of the analyses of the United States Department of Agriculture. The samples examined were purchased at retail stores in Washington, D.C.

For classifying the samples, qualitative analyses, similar to those used by Professor Cornwall, were employed.

A detailed description of the quantitative methods employed would take up too much space; suffice it to say that for the carbon dioxide two determinations were always made, one with the addition of acid for determining the *total* amount of carbon dioxide present, the other with the addition of water only for determining the amount of *available* carbon dioxide present, *i.e.*, the carbon dioxide actually liberated when the powder is used for baking purposes.

Other ingredients subjected to quantitative analysis were—

Starch (present as filling).  
Phosphoric acid.  
Tartaric acid.  
Alkalis.  
Aluminium.  
Calcium.  
Sulphuric acid.  
Ammonia.  
Moisture.

The results of each analysis are given in the first instance as acid and basic radicals in percentage composition. An attempt is then made to combine these into salts showing the constitution of the powder. In the latter case, however, the results arrived at are at best but approximately correct, for the difficulties in the way of arriving at the true constitution of a powder are very great, if not insurmountable. The analyses agree fairly well in a general way with those obtained by Professors Weber and Cornwall. The value of a baking powder with regard to its available carbon dioxide diminishes with age, so that the results obtained cannot strictly be taken as showing the relative values of the powders; but, generally speaking, the tartrate and phosphate powders give higher percentages of carbon dioxide than the alum and alum and phosphate powders. Mr. Crampton says: "Professor Cornwall's average for 20 samples of alum and phosphate powders (no straight powders included) is 8.97 per cent.; for eight samples of tartrate powders, 11.60. Professor Weber's average for 19 samples of alum powders is 7.58 per cent.; for eight samples of tartrate

powders, 11.20 per cent. My average for 20 samples of both alum and alum and phosphate powders is 8 per cent.; for eight samples of tartrate powders, 10.10 per cent. The only straight phosphate powders sold seem to be the various preparations made by the Rumford Chemical Works, and the 'Wheat' powder; at least these are all obtained by any of the investigators. The carbonic acid strength of the former is uniformly good, slightly higher than the tartrate powders; the latter is a peculiar preparation, made up without any filling whatever, and gives a low percentage of carbonic acid, except in one of Professor Cornwall's samples, which seems to have been obtained quite fresh."

A point of importance in connexion with baking powders is the percentage of filling or inert material they contain. The object of the filling is mainly to prevent the active constituents in the powder from reacting on each other during the time the powder is kept. It is possible that too much or too little filling may be present in a powder. In the former case the value of a powder is diminished by the presence of too large a proportion of inert material. In the latter case the powder is liable to rapid deterioration during keeping, so that when used its carbon dioxide value is very low. In the case of bitartrate powders, Mr. Crampton found on an average 14.04 per cent. of filling, 24.57 per cent. being the highest, 5.32 per cent. the lowest quantity; the latter evidently not being sufficient to prevent deterioration. Powders made up with free tartaric acid contained much more filling, namely, 40.05 and 45.63 per cent., possibly on account of the hygroscopic nature of the free acid. 26.41 per cent. was found in a phosphate powder, another contained no filling, evidently to its detriment. The alum and alum and phosphate powders contained large percentages of filling, 40.76 per cent. being the average, 52.29 per cent. the maximum, 31.54 per cent. the minimum.

Probably this high percentage is necessary to prevent deterioration, especially if but a small proportion of the water of crystallisation has been driven off from the alum. Professor Prescott has given it as his opinion that anything over 20 per cent. of filling should be looked upon as dilution.

Mr. Crampton finally has attempted to make up a domestic baking powder of fair keeping quality, and gives the following formula as answering the purpose well:—

	Oz.
Cream of tartar.....	8
Baking soda.....	4
Corn starch.....	4
	Per Cent.
Total carbon dioxide.....	12.63
Available carbon dioxide.....	10.91

The author's views as to the lines on which legislative regulation of the sale of baking powders should proceed have been given at the commencement of this abstract.

—F. W. T. K.

## (B.)—SANITARY CHEMISTRY.

### PATENT.

*Improvements in the Means of and Apparatus for Treating Refuse.* W. Warner, Nottingham. Eng. Pat. 18,719, December 21, 1888. 8d.

THESE improvements have reference to the apparatus commonly known as "Destructors." At the back of the grate is situated a drying hearth on to which the refuse is delivered from a hopper above, placed at the rear end of a reverberatory arch which covers both grate and drying hearth. The construction of the apparatus is such that the main flue for taking off the products of combustion is caused to pass under the drying hearth, which thus receives heat both from above and below. The fire and ash-pit doors are made air-tight so that a forced draught can be used if desired. When employed it is delivered partly below the fire-bars and partly above. The latter current enters the furnace through perforated metal plates so

arranged as to protect the brickwork on each side of the grate, and having air-channels situated behind them. The improvements also have reference to the form of hopper used, the nature of the damper arrangements, and to contrivances for intercepting dust in the flue and facilitating its removal.—A. R. D.

## XIX.—PAPER, PASTEBOARD, Etc.

*Study of the Quality of Paper.* Bull. Soc. d'Encour. pour l'Ind. Nat. 1889, 430—444.

THE increase in the production of paper has led to the introduction of a variety of raw materials in addition to rags, such as hemp, straw, wood-pulp, &c. Of these hemp alone can be considered as an improvement. Attention has been more and more directed to the qualities of paper, its surface, its colour, and at the same time its selling qualities, the result of which is the production of paper of inferior quality, of little strength, and oftentimes destined to a short life. These circumstances have led to the establishment in Germany of a specially appointed laboratory at the Imperial Technical Institute of Charlottenburg, under the direction of Dr. Martens, where papers are systematically examined. The principal points examined are strength, elasticity, nature of sizing, material, thickness, &c.

To determine (1) *the strength*, a number of strips of the paper taken in different directions are tested in a specially constructed machine known as the Gatty-Reisch machine, and the mean taken. The results are expressed in terms of the length of the paper itself necessary to produce the rupture. This is easily obtained by calculation when the weight of the paper and its breaking strain in grammes are known. The same machine indicates automatically the elasticity or extensibility of the paper.

(2.) *Resistance to Rubbing*.—A sample is taken and rubbed between the hands. With a weak paper minute holes soon make their appearance, the number of rubbings determining the quality.

Papers are classed as follows:—

1. Extremely weak.
2. Somewhat less weak.
3. Less weak.
4. Medium.
5. Moderately strong.
6. Strong.
7. Very strong.
8. Extremely strong.

(3.) *Thickness*.—This is measured by means of a micrometer.

(4.) *Ash*.—Cellulose contains less mineral matter than 1 per cent. Any increase in the amount of ash indicates the addition of mineral matter.

(5.) *Microscopical Examination*.—The sample is moistened with a solution of iodine, and the following colourations are obtained:—1. Fibres coloured yellow, in the case of wood pulp. 2. Fibres of cinnamon colour, hemp, linen, cotton. 3. Fibres, uncoloured, pure cellulose. To prepare the paper for microscopic examination the specimen moistened with iodine solution is placed in a porcelain crucible with a little water, a little soda solution added, and the whole boiled. The liquor is coloured yellow if the paper was sized; after a quarter of an hour's boiling, the residue is washed with water. The pulp is then triturated in a mortar with a little water and a portion placed afterwards on the object glass of the microscope. It is easy under the microscope to distinguish to which of the three groups named the specimen belongs.

(6.) *Examination for Chlorine and Free Acids*.—These bodies are very rarely found in papers. The former is recognised by its action on potassium iodide and starch,

The detection of the latter is a matter of great difficulty. Gertzberg recommends treatment with a solution of Congo-red.

(7.) *Determination of Sizing Material.*—Gelatin is detected by the formation of a cloudiness or precipitate when a solution of tannic acid is added to an infusion of the sample of paper in boiling water. Vegetable size is examined for by warming the paper with alcohol and pouring the solution into water; a precipitate is formed if rosin size be present.

To determine the degree of sizing lines are drawn on the paper with a solution of ferric chloride and the other surface is floated in a solution of tannic acid. Ink is formed with less or greater rapidity, according as the paper is well sized or otherwise.

The percentage of water in a paper is also usually determined.

The following are some of the methods of classification adopted at Charlottenburg:—

#### CLASSIFICATION ACCORDING TO STRENGTH.

Classes.	1.	2.	3.	4.	5.	6.
a. Mean breaking length in metres.....	6,000	5,000	4,000	3,000	2,000	1,000
b. Mean elasticity percentage extension .....	1.5	4.0	3.0	2.5	2.0	1.5
c. Resistance to rubbing..	6	6	5	4	3	1

#### CLASSIFICATION ACCORDING TO COMPOSITION.

1st Class.—Paper composed entirely of cellulose, giving a maximum of 2 per cent. of ash.

2nd Class.—Paper composed of cellulose, straw, but without wood pulp, giving 5 per cent. of ash.

3rd Class.—Paper composed of various substances, but without wood pulp, giving less than 15 per cent. of ash.

4th Class.—Paper composed of various substances, whatever the percentage of ash.

Though many objections have been raised to the above classifications, their value has been shown by the fact that of late years the average quality of paper manufactured in Germany has risen and the intervention of the Government has had a good effect on the industry.

The report concludes with the following notes on the microscopic examination of paper:—

The fibres of plants do not present the same appearance in paper as in the natural state, as the processes which they have undergone, to some extent modify them.

*Straw.*—The various kinds of straw usually employed are oat, wheat, barley, rye, maize, and, in China, rice. The fibres present but few distinguishing features; they, moreover, resemble those of hemp and linen. On the other hand, it is easy to recognise in a paper the epidermic cells of straw, and even to determine their source. Fig. 1 represents

Fig. 1.



Fig. 2.



Fig. 3.



a cell from rye straw, magnified 250 times; Fig. 2 that from rice, and Fig. 3 that from maize. The latter has been subjected to the action of chromic acid. The following table gives the dimensions of the various kinds of cells:—

	Dimensions of Cells (in Millimetres).	
	Length.	Breadth.
Barley.....	*103—*214	*012—*014
Rye .....	*086—*315	*010—*016
Wheat.....	*152—*449	*018—*024
Oats.....	*187—*443	*012—*017
Maize .....	*108—*252	*033—*030

These epidermal cells are always accompanied by vascular cells in the form of spirals and isolated rings (Figs. 4 and 5.)

Fig. 4.



Fig. 6.



Fig. 7.

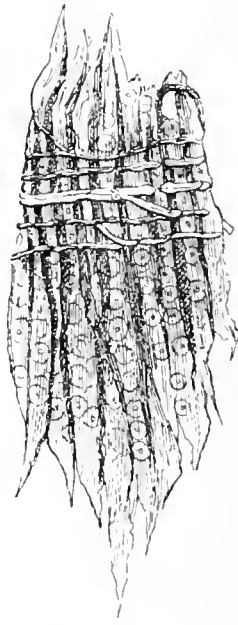


Fig. 5.



Fig. 8.

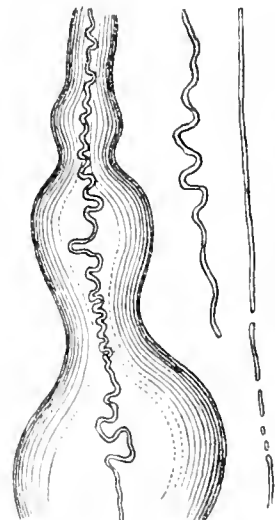
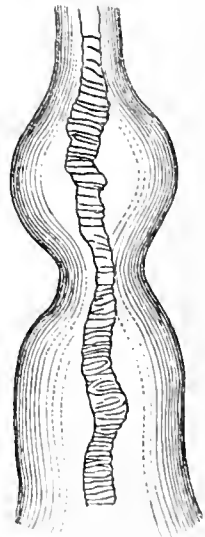


Fig. 9.





**Wood Fibre.**—There are two kinds of wood fibres usually met with in paper, one from the various kinds of pine, showing very characteristic round pitted vessels (Figs. 6 and 7), the other derived from birch and poplar, having equally characteristic, but totally different, markings.

**Fibres of Dicotyledonous Plants.**—Of these the most important are flax and hemp, and they are very difficult to distinguish from each other. Weisner has lately endeavoured to distinguish them by means of the action of ammonia-copper solution. This solution causes the fibres to swell, and ultimately dissolves away all but the interior canal. The different appearances presented are shown in Fig. 8 (flax) and Fig. 9 (hemp).

This reaction cannot be applied to the fibres in paper, as the mechanical treatment they undergo entirely masks their characteristic features.

The action of the beating rod causes the fibres to split up until they present somewhat of the appearance of Fig. 10 or Fig. 11, if it has not been carried on to the same extent. Occasionally transverse markings make their appearance (Fig. 12); these markings never occur in the case of fibres from monocotyledonous plant.

**Cotton.**—The general microscopical appearance of the cotton fibre is shown in Fig. 13. The spiral appearance,

Fig. 10.

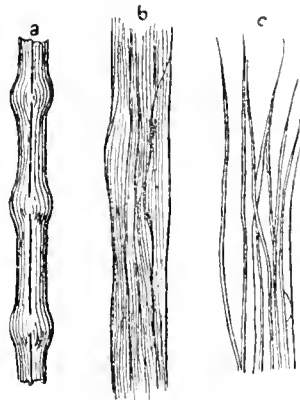


Fig. 12.

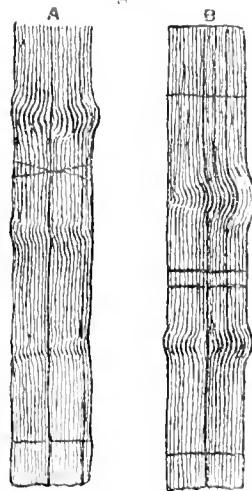
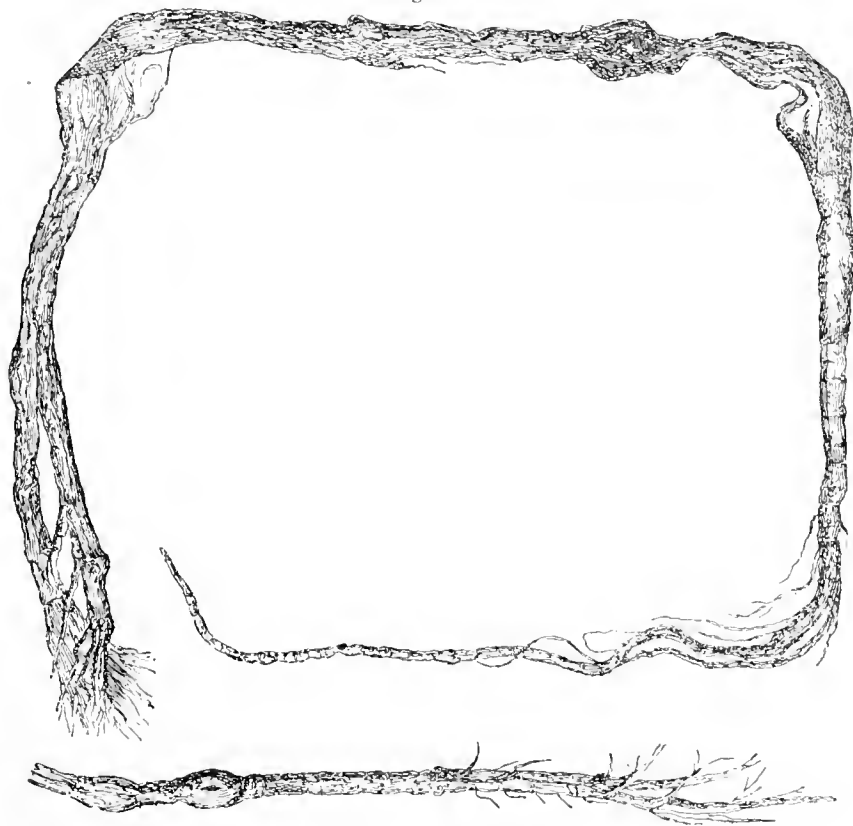


Fig. 11.



however, is sometimes simulated by the flax fibre (Fig. 14), and on the other hand occasionally cotton fibre is met with

Fig. 13.

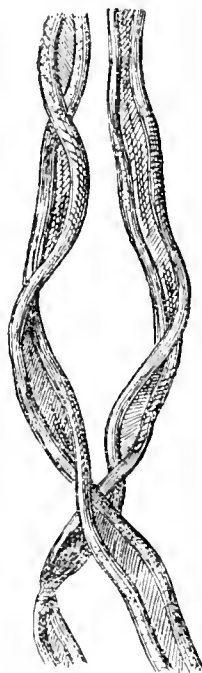


Fig. 14.



Fig. 15.

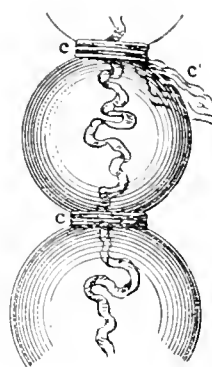


Fig. 16.



in which it is absent. The ammonia copper reagent causes the cotton fibre to assume the appearance of Fig. 15, the

interior canal having the appearance of a flat ribband. The action of the roll sometimes causes cotton fibre to assume the appearance of Fig. 16.—E. J. B.

*Manufacture of Paper in Japan.* M. Martens. *Bull. Soc. d'Encour. pour l'Ind. Nat.* 1889, 495—498.

THE German Consul at Gioko-Osaka lately sent over a collection of samples of papers made in Japan, which have been examined at the Institute at Charlottenburg according to the methods in use there. The results are given in the following table:—

Name.	Composition.	Ash.	Weight of Square Metre.	Breaking Length.	Extensibility.
		Per Cent.	Grms.	Kilometres.	Per Cent.
Gampi.....	Gampi.....	1.2	12.6	7.2	2.9
Kodsu.....	Kodsu.....	2.3	22.1	4.25	3.9
Mitsumata .....	Mitsumata .....	1.4	18.4	4.08	2.8
Kopigampi .....	Gampi.....	2.4	8.2	7.05	2.1
Ussuyossi.....	( $\frac{2}{3}$ Gampi..... ) ( $\frac{1}{3}$ Mitsumata..... )	1.5	10.0	7.12	2.2
Y'su nedzumi .....	0.8 Gampi .....	4.3	23.1	7.78	3.2
Programi .....	0.2 Silk.....				
Hiro Suki.....	Kodsu .....	1.9	7.8	8.18	2.6
Murasaki.....					
Srogami .....					
Atsu Hinkirigami .....	Gampi.....	1.8	37.8	10.8	3.3
Usu Hinkirigami .....	Gampi.....	1.8	23.9	10.7	3.1
Tu Hyoshi .....	Gampi.....	2.2	84.9	10.5	4.3

By "breaking length" is meant the length of a band of paper suspended vertically, the lower end being free, which the fixed end will support before breaking.

It is remarkable that with European papers the breaking length varies from one to six kilometres, whereas with the Japanese papers it rises to as high as 10.8 kilometres.

The principal materials used for making paper in Japan are: 1. *Wickstomia canescens*; 2. *Edgeworthia pagrifera*; 3. *Broussonetia pagrifera*. Their common names in Japan are Gampi, Mitsumata and Kodsu. Efforts have been made to acclimatise these plants in Europe, but without success. Nearly all the paper is made in Japan by hand labour, the

methods being handed down in families. The methods are very simple. The bark is removed from the tree and allowed to soak for some days to loosen the outer coating. This is removed and the pure bast fibre is allowed to bleach in the sun. It is then boiled in lime water or in a lye made from the ashes of the plants themselves, well washed in water and then beaten into pulp with sticks. The frame on which the paper is made is of thin strips of bamboo and silk.

The paper is sized with boiled rice or some similar starchy material.—E. J. B.

## PATENTS.

*Improvements in Treating or Cleansing Paper-making Materials and other Fibrous Materials and Fabrics.*  
R. Hamilton, Edinburgh, and I. C. Hamilton, Linlithgow.  
Eng. Pat. 1433, January 26, 1889. 11d.

The inventors assist the action of caustic soda in the preparation of paper pulp from rags, esparto, &c., by the addition of the soda or potash salts of certain sulphonic acids obtained by treating olefines with sulphuric acid.—E. J. B.

*Improvements in the Manufacture and Bleaching of Wood and other Pulps and in Apparatus for that Purpose.*  
C. Ramsey, Leeds. Eng. Pat. 1834, February 1, 1889. 8d.

The inventor boils wood chips with lime water in a vomiting boiler. When sufficiently acted upon he treats with a solution of hypochlorite of magnesia at 3° B. for eight hours, the circulation of the liquid being maintained by means of steam. The pulp so obtained is then bleached in a separate vessel with another solution of hypochlorite of magnesia. The solutions are prepared by adding solution of sulphate of magnesia to one of bleaching powder.—E. J. B.

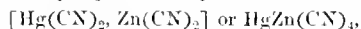
## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

*On the So-called Double Cyanide of Zinc and Mercury.*  
W. R. Dunstan. Pharm. J. February 15, 1890, 653.

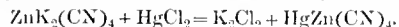
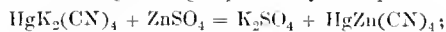
SIR JOSEPH LISTER has recently drawn attention to the valuable antiseptic properties possessed by a material which is described in many chemical treatises as a double cyanide of zinc and mercury prepared by precipitating a solution of the cyanide of mercury and potassium with a solution of zinc sulphate (Lancet, November 9, 1889, and January 4, 1890).

During the manufacture of this substance several facts were observed which seemed to throw doubt on the statement that it is constituted of a chemical compound of the two cyanides.

It is stated on the authority of Rammelsberg, in Gmelin's "Handbook," that when the soluble double cyanide of mercury and potassium,  $\text{HgK}_2(\text{CN})_4$ , is decomposed with a solution of zinc sulphate, or when the soluble double cyanide of zinc and potassium,  $\text{ZnK}_2(\text{CN})_4$ , is decomposed with a solution of mercuric chloride, an insoluble double cyanide of mercury and zinc is precipitated, the composition of which is probably represented by the formula—



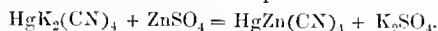
the chemical changes being expressed by the equations—



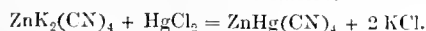
It is this substance, freed from soluble salts by washing with cold water, that has been prepared for Sir Joseph Lister. It was observed that in washing the precipitate with cold water large quantities of mercuric cyanide are dissolved from it, only a relatively small proportion, varying from one-fourth to one-fifth of the calculated quantity, remaining in the precipitate. In the absence of knowledge as to the cause of this action, and as to the nature of the precipitate, the manufacture of the substance becomes an entirely empirical operation.

The methods which suggested themselves were—

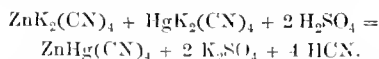
1. The precipitation of a solution of mercury potassium cyanide with a solution of zinc sulphate—



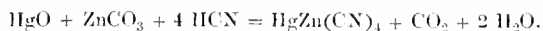
2. The decomposition of a solution of zinc potassium cyanide with a solution of mercuric chloride—



3. The decomposition of a solution containing a formula-weight of mercury potassium cyanide and a formula-weight of zinc potassium cyanide with just sufficient dilute sulphuric or acetic acid to convert the whole of the potassium present into potassium salt; the cyanides of zinc and mercury would thus be precipitated together:—



4. The simultaneous dissolution of equivalent quantities of freshly precipitated mercuric oxide and freshly precipitated zinc carbonate in strong aqueous hydrocyanic acid—



Having prepared the pure reagents the four above-mentioned processes were successfully put into operation for the purpose of ascertaining whether mercuric cyanide will unite with zinc cyanide in any proportion.

*Method 1.*—This method is substantially the same as that by which the substance used by Sir Joseph Lister has been prepared. Sir Joseph Lister informed the author that the process adopted has been as follows: Cyanide of mercury, 24 oz.; cyanide of potassium (about 95 per cent.), 12 oz. 171 grains; zinc sulphate, 27 oz. 159 grains. The potassium cyanide and the mercuric cyanide are dissolved in 4 pints of cold water, and the liquid is precipitated by the addition of the zinc sulphate dissolved in 12 pints of cold water. These quantities are calculated from the equation given above. The precipitate is collected and washed with two successive quarts of water (see also "Lancet," January 4, 1890).

The substance prepared in this manner is a nearly white powder. The composition of a specimen furnished by Sir Joseph Lister was ascertained by analysis. By washing with cold water, until nothing further is removed, the amount of soluble mercuric salt is determined. The mercury, zinc, and cyanogen were determined by known reliable methods.

The results of the analysis were—

	Per Cent.
Total mercuric cyanide .....	17.77
Mercuric salt removed by cold water ..	2.86
Zinc cyanide .....	67.85

There were also present about 10 per cent. of zinc hydrate, or more probably hydroxy-cyanide, originating partly from the alkali contained in the potassium cyanide, and partly, no doubt, from the decomposition of zinc cyanide.

The supposed double cyanide of zinc and mercury,  $\text{ZnHg}(\text{CN})_4$ , would contain 68.6 per cent. of mercuric cyanide; the substance analysed contained less than one-fourth of this quantity, the remainder having been dissolved by the water present during precipitation and by that used in washing the precipitate.

The substance was now prepared by the same method, but more water was added to the solutions before precipitation. The precipitate was washed until quite free from soluble mercuric salt and dried over caustic soda in a vacuum desiccator, to avoid any possible decomposition by heat. The results of the analysis of this specimen were—

	Per Cent.
Total mercuric cyanide .....	6.77
Mercuric salt removed by cold water ..	None.
Zinc cyanide, including hydroxy-salt ..	33.3

The quantity of water present during precipitation is thus shown to exercise an important influence on the composition of the precipitate. A large quantity of water leads to the retention of much less mercuric salt in a form in which it is not dissolved by water. The amount of mercuric cyanide in this specimen is only about one-tenth of that contained in the compound  $\text{HgZn}(\text{CN})_4$ .

*Method 2.*—Seven grms. of the double cyanide of zinc and potassium were dissolved in water and precipitated by the addition of a solution containing 7.7 grms. of mercuric chloride. The entire quantity of water present was rather less than in the preceding experiment. The precipitate

was washed with cold water until free from soluble mercuric salt, dried over caustic soda and analysed. The following results were obtained :—

	Per Cent.
Total mercuric cyanide .....	9.67
Mercuric salt removed by cold water .....	None.
Zinc cyanide .....	80.61
Zinc hydroxy-compound (calculated as hydroxide) .....	7.3

This substance contains about one-seventh of the quantity of mercuric cyanide, represented by the formula  $\text{ZnHg}(\text{CN})_4$ .

**Method 3.**—Equivalent quantities of the mercury potassium cyanide and the zinc potassium cyanide were dissolved in water, and just sufficient diluted sulphuric acid was added to remove the whole of the potassium. The precipitate was collected, washed until free from soluble mercuric salt, and dried over caustic soda. The following are the results of its analysis :—

	Per Cent.
Total mercuric cyanide .....	8.46
Mercuric salt removed by cold water .....	None.
Zinc cyanide .....	81.9
Zinc hydroxy-compound (calculated as hydroxide) .....	6.9

This method also failed to produce the compound  $\text{ZnHg}(\text{CN})_4$ ; the amount of mercuric cyanide contained in it is only about one-eighth of that represented by this formula.

**Method 4.**—Equivalent proportions of freshly precipitated mercuric oxide and zinc carbonate were thoroughly incorporated and added to a small quantity of concentrated aqueous hydrocyanic acid. Reaction was immediate, carbon dioxide being copiously evolved. The mixture soon lost the odour of hydrocyanic acid. More was then added, until the odour remained permanent after some hours, the mixture being frequently stirred during this time. The precipitate was filtered, washed with cold water until no mercuric salt could be detected in the washings, and dried. It yielded on analysis the following results :—

	Per Cent.
Total mercuric cyanide .....	1.6
Mercuric salt removed by cold water .....	None.
Zinc cyanide, including a little hydroxy-compound .....	98.4

This method is no more successful than those previously tried, in producing a double salt of the formula  $\text{ZnHg}(\text{CN})_4$ . It seemed, however, to be a method which might possibly be useful in the preparation of the antiseptic material, since the product is remarkably pure. In the experiment described above, a saturated aqueous solution of hydrocyanic acid was employed, and it was thought that the large quantity of water present had prevented the retention of much mercuric cyanide in an insoluble form. Equivalent quantities of mercuric oxide and zinc carbonate were therefore suspended in a little alcohol, and the vapour of hydrocyanic acid passed into the liquid until reaction was complete. On filtering and thoroughly washing the precipitate it was found that even under these favourable conditions nearly the whole of the mercuric cyanide had been dissolved.

The analyses which are recorded above of the precipitates obtained by four distinct methods, each of which is likely to afford the supposed compound, provided that it is not decomposed by water, conclusively disprove the existence of a double cyanide of the formula  $\text{ZnHg}(\text{CN})_4$ . Gmelin's suggestion that such a compound may be prepared by precipitating a solution of mercury potassium cyanide with a solution of zinc sulphate or a solution of zinc potassium cyanide with mercuric chloride is clearly erroneous.

Although there can no longer be any doubt that the compound  $\text{ZnHg}(\text{CN})_4$  cannot exist, yet there remains to be explained the remarkable fact disclosed by the foregoing results that the precipitated zinc cyanide retains some quantity of mercuric cyanide so firmly that it cannot be removed by washing the substance with cold water. In each of the precipitates produced by the four different processes the mercuric cyanide appears to be contained in two forms. The greater part of the mercuric cyanide is

evidently in the free state, and is therefore easily dissolved by cold water. A smaller part is not removed by cold water, although mercuric cyanide is a soluble salt. How is this smaller amount of mercuric cyanide retained by the zinc cyanide? Is it (1) chemically combined as a double cyanide? or (2) as a compound of mercuric hydroxy-cyanide with zinc cyanide? or (3) as a compound of zinc hydrate (or hydroxy-cyanide)? or (4) is it merely *occluded*\* by particles of zinc cyanide during precipitation, so that in consequence of a protective coating of insoluble zinc cyanide the water is unable to come into contact with the mercuric cyanide? Experimental investigation alone can furnish a conclusive answer to these questions.

The author speculates on the cause of the presence of mercury cyanide not in a state of combination, and gives an account of experiments to determine this point.

#### Report on New Fine Chemicals of Therapeutic Value.

Merek's Bull, 1889, 85—92; also 93—102.

THE MYDRIATIC ALKALOIDS OF THE SOLANACEÆ. — *Hyoscyamine* (this Journal, 1888, 584).

This alkaloid is principally obtained from the leaves and seeds of *Hyoscyamus niger*, though more recently from *Atropa belladonna*. In the trade three modifications are to be found at present:—*Amorphous Hyoscyamine*, not colourless, from *Hyoscyamus niger*; *Crystallised Hyoscyamine* from *Hyoscyamus niger*, chemically pure, perfectly white, and very light, and consisting of the pure alkaloid; and *Commercial Hyoscyamine*, chemically identical with the preceding kind, but obtained from *Atropa belladonna*. The sulphates, hydrochlorides and hydrobromides are also to be met with, but it is the alkaloid which is principally used.

*Amorphous Hyoscyamine* is a light brown, syrupy liquid of alkaline reaction, but little soluble in water though readily so in acidulated water, alcohol, ether and chloroform.

*Crystallised Hyoscyamine* is met with in silky crystals, permanent in the air, melting at  $108.5^\circ \text{C}$ ., and possessing the same solubility as the preceding kind.

*Commercial Hyoscyamine* (*Atropa belladonna*) in its physical and chemical properties is similar to the crystallised variety; its physiological and therapeutical behaviour has not yet been reported on.

Generally speaking, the physiological and therapeutic effects of hyoscyamine are similar to those of atropine, especially as regards mydriatic action. In recent practice its use in ophthalmology has been almost abandoned, and it is now principally employed as a hypnotic in mental disorders, or as an anodyne and anti-spasmodic in asthma, epilepsy, &c. According to *Gauke* the pure crystallised alkaloid is the best form for administration.

*Hyosine*.—This alkaloid was discovered by Ladenburg in the mother-liquors from the manufacture of hyoscyamine. Hyosine has never yet been obtained in the crystalline, or even solid form. It is a syrupy liquid, with difficulty soluble in water, though easily so in alcohol and ether. It yields well crystallised salts, among which the following are at present recognised as possessing therapeutic value:—*Hyosine hydrochloride*, *hydrobromide*, and *hydriodide*.

In consequence of its physical properties the pure alkaloid is not used in medicine.

All the salts of hyosine are soluble in water, and insoluble in strong alcohol and in ether.

In 1881 Hirschberg discovered that hyosine is a very powerful mydriatic, but he added that owing to its ready liability to act on the organ generally, caution must be exercised in its ophthalmologic use. Emmert (Archiv. für Augenheilkunde, 1882, 183) and Walter (Inaugural Dissert. Dorpat, 1887) fully endorsed Hirschberg's conclusion. Emmert recommends hyosine in all cases where energetic and prompt mydriatic action is desired. C. Walter found

\* It has been suggested to the author that some explanation of the use of this word should be given, in view of its special application by the late Professor Graham to designate another, but apparently analogous, class of facts. He therefore takes this opportunity of stating that the word is here used in its strictly etymological sense, being derived from *occludo*, "to shut up" or "lock in."

that a more rapid and greater dilation of the pupil was obtained with a  $\frac{1}{4}$  to a 1 per cent. solution of hyoscyne than with a 1 per cent. solution of atropine, the duration of *mydriasis* (dilation of pupil) however being briefer.

Remarkable hypnotic effects with the hyoscyne salts are reported especially in the case of insane patients, and most especially in extreme cases of excited or raving insanity.

In the tremors of *paralysis agitans*, even in very grave cases, hyoscyne has been found by *Erb* to act as a most excellent palliative. In other nervous disorders it has been successfully used.

*Tertiary Amyl Nitrite* (Bertoni's "amylonitrous ether"),  $C_5H_{11}NO_2$  (see this Journal, 1889, 1003).—The boiling point of this "ether" is not as stated (*loc. cit.*), "about 30° C.," but as Bertoni now states "93° C."

*Antipyrine and Nitrites. Their Antagonism.*—According to Knorr, when an acid aqueous solution of antipyrine is brought into contact with potassium nitrite, a slightly soluble crystalline substance of bluish-green colour is formed, viz., isonitroso-antipyrine,  $C_{11}H_{11}N_3O_3$ .

H. C. Wood and J. Marshall have shown this latter compound to be devoid of any action either on dogs or rabbits; also a dog when given antipyrine followed very soon by ethyl nitrite, showed no abnormal condition whatever. Antipyrine would thus seem to be capable of diminishing and even wholly neutralising the possible detrimental action of a nitrite, whilst, on the other hand, antipyrine itself is debarr'd from all action by the presence of sufficient nitrite.

*Bromoform*,  $CHBr_3$ .—A colourless limpid liquid of a peculiar, sweetish, agreeable taste. It is easily soluble in alcohol, but only slightly so in water, five or six drops dissolving in 100 grms. of water only after long and violent shaking. This aqueous solution, if well corked, may be preserved for a long time.

Bromoform is an anæsthetic like chloroform, but it does not irritate the mucous membrane of the mouth as chloroform does. Moreover, experiments by Horroch (Oesterreich. Med. Jahrb. 1883, 497) have proved that complete anæsthesia is obtained by much smaller respiratory doses of bromoform than of chloroform. By internal and subcutaneous treatment with bromoform, warm-blooded animals were placed in *narcoses* lasting from four to eight hours. These were induced within 5 to 36 minutes after subcutaneous administration of 0.1—1.0 grm. of bromoform, and were always accompanied by a fall of temperature of from 3° to 5° C.

Great success attended the use of a solution of bromoform in water to which a little alcohol had been added, in cases of whooping cough complicated with pneumonia, by Stepp and Goldschmidt (*Deutsch Med. Wochenschr.* 1889, No. 31).

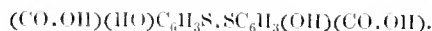
*Bryonia alba* and its active constituent, *Bryonidin*, have recently been found to act as a most powerful *Hæmostatic* (capable of arresting hæmorrhage).

Petresen and Eliau (*Deutsch. Med. Zeit.* 1889, 487) commend the merits of white bryony (*Bryonia alba*) as a hæmostatic of prime efficacy. This is a new use of the drug. An infusion is made of 20—25 grms. of the dry root in 300 grms. of hot water, and the liquid is evaporated down to 150 grms. This liquid is mixed with syrup for administration. The above investigators succeeded in isolating from bryony root, besides several resins and acids, also a glucoside which they considered to be *Bryonin*. Its general action corresponds to that of the root. However, more recently still, A. Maukowsky (Inaug. Dissert. Dorpat, 1889) reports that he has found two glucosides in *Bryonia alba*, viz., bryonin and bryonidin, and he further states that only bryonidin possesses toxic, and therefore therapeutic properties. It is to be specially noted that red bryony (*Bryonia dioica*), easily confounded with the preceding, possesses no hæmostatic virtues whatever. The physiological action of *Bryonia alba* consists of a progressive contraction of the capillaries, which may be continued until a complete arrest of the circulation is secured. For antihæmorrhagic effect *Bryonia alba* appears to far transcend any other agent as yet known.

*Chloral-amide*,  $CCl_3.CH(OH)(NH.CHO)$ , a recently discovered and synthetically prepared hypnotic formed by the addition of anhydrous chloral ( $CCl_3.CHO$ ) to formamide ( $CHO.NH_2$ ). Chloral-amide is prepared in colourless crystals, soluble in 9 parts of water, or in  $1\frac{1}{2}$  parts of 96 per cent. alcohol. Its taste is mildly bitter, and not acrid. Its aqueous solution must be prepared cold, since the compound decomposes when heated above 60° C. Its aqueous and alcoholic solutions are not affected either by weak acids or silver nitrate solution. By caustic alkalis they are decomposed rapidly, and slowly by alkaline carbonates. Therapeutic value has been tested by Hagen and Hübner, Lettow, Rabow, Reichmann, and Kny (*Wiener Med. Presse*, 1889, 1361). As a hypnotic it has been proved superior to chloral hydrate. Of most value in cases of nervous insomnia and similar disorders. Disagreeable accessories not noticed (Rabow). This hypnotic may be taken internally as a powder, prepared by triturating 1—3 grms. of the amide with 1 grm. of fennel-oil sugar (*Eulosaccharum Feniculi*).

*Mercury Preparations.*—Their relative toxic strengths compared. As the result of a series of experiments by Zeising of Breslau (First Congress Germ. Dermatolog. Soc. Prague, June 1889) it was shown that the absolute mercury strength of any preparation used has less share in the toxic result produced than the degree of absorbability of the preparation into the organism.

*Dithiosalicylate of Sodium*—



*Dithiosalicylate of Sodium I. and II.* prepared by Baum are two isomeric substances constituted as above. The salt No. II. has been experimented upon by Lindenhorn, who found it to be a valuable antiseptic and disinfectant, much more powerful than salicylate of sodium.

It is a greyish-white powder, very hygroscopic, and soluble in water. Any acid added to the solution produces a precipitate of yellow viscid drops, consisting of dithiosalicyllic acid. Ferric chloride added to the aqueous solution of the soda salt produces a faint violet colouration. According to Hueppe a 20 per cent. solution of dithiosalicylate of sodium destroys the spores of *anthrax* in the course of 45 minutes. Salicylate of sodium has no such action. Lindenhorn claims the following advantages over ordinary sodium salicylate: The dithiosalicylate need not be given in such large doses as salicylate. It has no ill effect on the stomach, heart, or large vessels; does not produce collapse nor singing in the ears. It promises to be a more valuable anti-rheumatic.

## PATENTS.

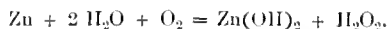
*Improvements in the Production of Oxygen and Hydrogen in Large Quantities by Means of the Electrolysis of Water, applicable for Ballooning and other Uses.* D. Latchinoff, St. Petersburg, Russia. Eng. Pat. 15,935, November 3, 1888. 11d.

See under X1., page 196.

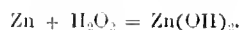
*A Process for making Hydrogen Peroxide.* Dr. Traube, Breslau, Germany. Ger. Pat. 48,542, December 5, 1888.

SCHÖNBEIN has shown (*Verhand. d. Naturforsch. Ges. in Basel*, N.F., Part II., 426) that when zinc amalgam is shaken up with water and air, there is formed, together with zinc hydrate, a small quantity of hydrogen peroxide, which even after long shaking does not rise above  $\frac{1}{33000}$  part. According to Traube the reactions are as follows:—

I.



II.



Hydrogen peroxide therefore is found in such a small proportion only because it is again almost completely decomposed by the zinc. This does not, however, occur if an alkaline earth is present. In such a case the hydrogen peroxide forms a superoxide of the alkaline earth. Accordingly zinc amalgam and milk of lime are shaken up with air when a precipitate of zinc hydrate and calcium superoxide separates, which is decomposed by an acid that forms an insoluble compound with zinc and lime. Lime water does not give such good results as milk of lime. Cadmium amalgam acts in the same way as zinc amalgam. The amalgam should contain at the most 1 part of zinc or cadmium to 1,000 of mercury.—H. S. P.

*An Improved Process for Producing Ozone Water in which the Ozone is Retained for a Considerable Period without Alteration.* B. Graf and F. Pickenbrock, Berlin, Germany. Eng. Pat. 15,526, October 3, 1889. 4d.

A SMALL quantity of hydrochloric acid and of an alkaline chloride is added to the water, through which ozonised oxygen is passed, to improve the keeping properties of the product. (Compare this Journal 1890, 3—10.)—G. H. B.

## XXII.—EXPLOSIVES, MATCHES, Etc.

*Determination of the Detonating Point of Explosives.* Bein. Zeits. f. angew. Chem. 1889, 667—669.

THE author has been in the habit of using Horsley's method, which consists of floating on an oil-bath a small dish containing the powder and heating until the powder fires, noting the temperature of the bath at the time. To avoid sublimation of sulphur to a certain extent, he did not introduce the powder until after the bath had been heated. The method has disadvantages as for higher temperatures (200° to 450°); special baths must be used; and, again, the thermometer does not necessarily give the true temperature of the powder. An apparatus is described which would seem not to possess these disadvantages. A test tube is fixed in the axis of a sheet-iron cylindrical vessel with conical top. This sheet-iron vessel is about 30 cm. in height and 8 cm. in diameter; it is surrounded by another similar sheet-iron vessel covered with asbestos, and about 15 cm. in diameter. The bottom of the outer vessel contains a hole, which admits of the entrance of a Bunsen flame. The whole forms a sort of air-bath. When the temperature has been raised trials are made by dropping a few grains of the powder under examination into the test tube. The result is watched through spy holes in the side of the apparatus, and the temperature is registered by a thermometer in the tube. The apparatus seems to give very good results, and as many as 20 or 30 determinations may be made in an hour.—T. L. B.

*Report upon Questions relating to the Employment of Explosives in Presence of Firedamp.* Annales des Mines, 14, 1888, 197—376; and Proc. Inst. Civil Eng. 99, 100.

THIS is an exhaustive report of a special sub-commission appointed by the French Government to determine the behaviour of the different explosives capable of use in mines in the presence of firedamp. The sub-commission was composed of 11 members, of whom Mr. Mallard was president. The report is divided into five chapters, preceded by an historical note, and followed by tables and appended notes. Chapter I. describes the apparatus and mode of experiment. Chapter II., explosives freely suspended in firedamp mixtures. Chapter III., explosives exploded in a closed vessel. Chapter IV., mode of firing shot in the mine; and, Chapter V., conclusions.

The conclusions are as follow:—

1. Even explosives under water can inflame firedamp mixtures with air by means of the dust of the mine.

2. The greater number of known explosives are capable of igniting firedamp mixtures when exploded freely in the atmosphere. Among these explosives are dynamite, gun-cotton (either military or mining, particularly the latter), dynamite gelatin, and Paulille's ammoniac dynamite.

3. It is, however, possible to find explosives which detonate at a temperature sufficiently low to avoid inflammation of firedamp mixtures, at least in the great majority of cases, when freely exploded in the atmosphere. Among the explosives experimented on which approximately fulfil this condition are: (1), the intimate mixture of 50 parts dynamite with 50 parts of crystallised carbonate of soda, or sulphate of soda with 10 equivalents water of crystallisation, ammonia alum, and ammonium chloride; (2), Monlin-Blanc pyroselin powder; (3), mixture of 20 parts dynamite, at 75 per 100, and 80 parts of nitrate of ammonia; (4), mixture of 20 parts of gun-cotton titrating 173 cc. nitrogen dioxide and 80 parts nitrate ammonia; (5), bellite, of which the composition is not known with certainty, and the experiments have not been sufficiently numerous; (6), Favier's explosive, containing 90 parts of nitrate of ammonia, 10 parts mononitronaphthalene, which appears to equal bellite in security. It requires, however, further experiment.

4. Because of the complexity and variability of the phenomena occurring during the detonation of explosives free to air, it will be prudent to avoid firing shots in the mine, even with charges considered the safest, at points where the mixture of air and firedamp is inflammable. The choice of explosives must be considered as diminishing danger, but not as absolutely suppressing it.

5. It is necessary to employ the explosives under conditions such as to develop from them the maximum useful work. Economy and security are in accordance to recommend this rule. To accomplish this the following conditions are necessary. The explosive must be rammed with care, and the hole must be sufficiently deep. No void space must be left either in front, behind, or round the cartridge. The Bickford fuse must not be placed in contact with the explosive if it is used, and the dangers of the fuse are sufficiently great to make it desirable to replace it by some more certain mode of ignition.

The commissioners further remark that their conclusions led to abandoning the use of mining-powder in mines where firedamp is known to exist, and even to place under suspicion ordinary dynamite, dynamite gum, ammoniac dynamite, such as is actually manufactured. Of these, dynamite gum appears to be the most dangerous. The explosives which give greatest security are the binary mixtures of dynamite, gun-cotton, dinitrobenzene with nitrate of ammonia; but the best mode of manufacturing and protecting these mixtures from atmospheric moisture has still to be experimented on. The breaking up of the coal and rock by them also requires practical study. The commission recommend the Government to prepare sample cartridges and issue them to mining engineers who may be willing to conduct practical trials with them in ordinary work.

To the main report is added a supplementary one dealing more minutely with the conditions of the explosion of firedamp, and in the conclusions it is stated that the temperature of inflammation of firedamp is 500° C., but it is necessary that the action of this temperature should be prolonged to produce ignition. Because of this fact, and the almost instantaneous mixture of the products of combustion with the atmospheric air which causes them to cool rapidly, explosives in which the temperature of explosion is less than 2,200° C. are incapable of inflaming firedamp mixtures when detonated under normal conditions.

The hole, however, must be carefully tamped, as the greater the imperfection of wadding, the greater the danger, and free explosion in air is the most dangerous of all. In the supplementary report the experiments with binary mixtures are detailed minutely, and it is again stated that nitrate of ammonia is the best substance to be used in connexion with the explosives. The reports are illustrated with figures of the apparatus used, and numerous tables are given.

## PATENTS.

*Improved Machine for Making Cartridges of High Explosives.* W. D. Borland, London. Eng. Pat. 1321, January 24, 1889. 8d.

THE invention consists essentially of a contrivance for causing an intermittent feed of the explosive material into the cartridge case or tube or other suitable mould in which the explosive has to be formed, and also causing the plunger which presses the material to work on and not in the material, and to act with a certain maximum force which can be determined beforehand. For details of the machine, the specification, which is furnished with drawings, must be consulted.—W. M.

*A Process for Granulating and Glazing Gelatinous Explosives.* O. Imray, London. From F. C. Glaser, Berlin, Germany. Eng. Pat. 2078, February 5, 1889. 4d.

THE compound, while still in a plastic condition, is moulded into sheets or rods, which are cut into small pieces, dried, and subdivided by passing them between grooved rollers. The dust is sifted out for subsequent compounding and the granules are polished in an inclined revolving vessel and glazed with plumbago.—W. M.

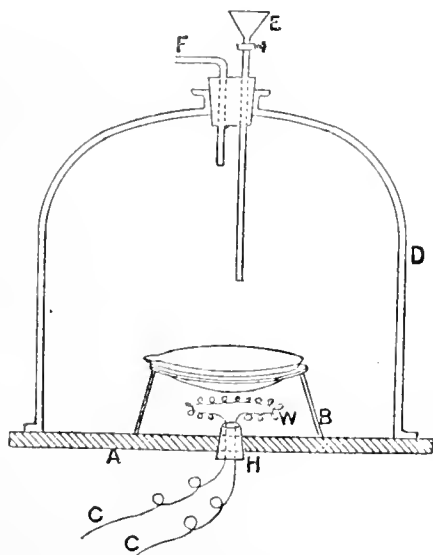
## XXIII.—ANALYTICAL CHEMISTRY.

*Electricity in Chemical Manipulation.* R. Fessenden. Chem. News, 61, 4—5.

DESCRIPTION of chemical apparatus in which electricity is employed.

1. *For Rapid Evaporations in Vacuum.*—A is a piece of plate glass, with hole bored in centre, and fitted with a cork, H, carrying two wires which are connected to the ends

Fig. 1.



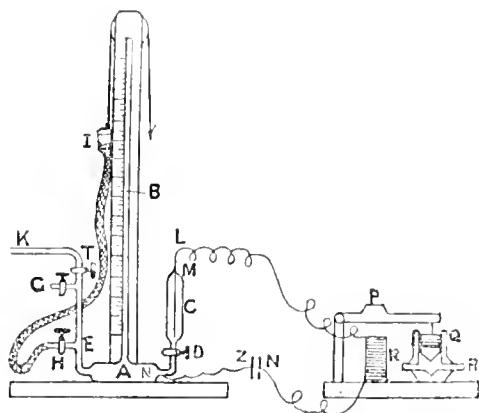
of the platinum coil W, packed in magnesia. The dish, with the liquid, is placed on stand B, and covered by bell-jar. F is connected to a filter pump, and the ends of the coil to a primary battery. More liquid is added from time to time through E.

2. *Automatic Heat Regulator.*—A is a piece of glass tubing, connected, first, to vertical piece of thermometer

tubing B, second, to reservoir C, having stop-cock at D, and, third, to the T-piece E.

K is connected to the bulb of an air thermometer. The branch H is connected by india-rubber tubing to the vessel, I, containing mercury, which may be raised or lowered by means of the pulley. Through C a carbon rod resting on

Fig. 2.



the capillary portion of C; through A, a platinum wire, X is fused. O is an electro-magnet, and the connexions are as shown. Q is a plunger, held by a spring in a piston, so that when the magnet is not acting, the gas can pass freely through R to R<sub>2</sub>. The apparatus is used as follows: the cocks H and T being open, whilst G and D are shut—it is then a simple air thermometer. When used as a regulator, cocks G and D are opened, and the height of mercury adjusted so that it nearly touches the carbon rod M; cocks G, H, and T are now closed; if the temperature rises the electric circuit will be complete, and the plunger will diminish the gas supply. This apparatus will maintain the temperature constant to 0.5°, it may be used with any pressure of gas, and with one galvanic cell, will work for months.

—W. W. H. G.

*New Apparatus for Testing Materials in Tension and Compression.* H. Bonnami. Le Génie Civil, 15, 1889, 475; and Proc. Inst. Civil Eng. 99, 7.

MR. BUGNET, of Havre, has succeeded in producing a simple, accurate and automatic machine, suitable for testing Portland cement, in a very ingenious manner.

His apparatus consists essentially of two vertical tubes, the one being rigid and the other flexible; the latter terminating in a reservoir provided with an escape valve. Both tubes are connected to a pan, closed above by an india-rubber diaphragm. The tubes and pan are filled up to a datum line with some suitable liquid, preferably mercury; the space between the mercury and the diaphragm is filled with water.

On raising the reservoir no pressure is exerted on the diaphragm if the escape-valve be closed; care being taken that the height of the reservoir above the datum never exceeds 760 millimetres (29.92 in.). On opening the valve the mercury slowly escapes, and exerts a pressure on the diaphragm in virtue of its height in the rigid tube, to which it is proportional.

The whole of the apparatus is mounted on a cast-iron stand. The pan, which is a part of the same casting, has two cavities; one is in communication with the rigid tube, and the other with the flexible tube and reservoir. The upper part of the flexible diaphragm is covered by a brass disc, on which a bent standard is pivoted; one of the pulling slips is suspended from the upper part of this standard, and the other clip is provided with an adjusting screw for bringing the clips up to their work when the briquette is inserted.

The load on the briquette is measured by the height of the mercury column in the rigid tube. The height



being indicated and registered by means of a float, a string attached to it passes over a ratchet-pulley, and is kept tight by a balance weight on the other end. The ratchet allows the index to rise, but not to fall, unless the pawl is released. The reservoir valve is provided with a divided disc to indicate the amount of opening, and consequently the rate at which the load is applied to the briquette. The reservoir itself slides up and down a guide pillar; on reaching the top it is held in position by a catch. The *modus operandi* is as follows: The briquette having been inserted in the clips and screwed up, the reservoir is raised and the valve opened, when the mercury gradually rises in the fixed tube, and applies the load on the specimen until fracture occurs. The mercury suddenly falls, but leaves the index in its highest position, thus indicating the breaking stress in kilogrammes per square centimetre on the fractured area.

By a slight modification the apparatus may be adapted for compression as well as tension tests.

*The Determination of the Specific Gravity of Viscous and Frothy Syrups, &c.* A. Genieser. *Zeits. f. angew. Chem.* 1890, 44—45.

THE removal of the air from frothy syrups in order to determine their specific gravity, is often effected by heating them on the water-bath in a funnel with its tube closed by a glass rod. Even after eight hours' heating at 100°, the complete removal of the air cannot be effected in this way, and further there is a considerable loss of moisture noticeable after half an hour's heating. To overcome these sources of error the following process is adopted:—The syrup is first warmed so as to thin it, filtered through a wire gauze and transferred to a specific gravity bottle, which is about two-thirds filled with it and weighed. The bottle is then heated in a suitable bath (oil-bath or bath of hydrochloric acid) until the syrup boils briskly and is kept for a few minutes at this temperature. The air collects above the liquid in bubbles and these are broken up and the air driven out by the addition of water after the bottle has cooled to 17.5°. The bottle is filled up with water and the whole weighed. The difference between the two weighings will give the weight of water added and thus the weight of syrup (free from water) is got; the weight of water contained by the bottle at 17.5° being known, the specific gravity of the syrup is obtained.

—C. A. K.

*Detection of Small Quantities of Nitrous Acid.* G. Lunge. *Zeits. f. angew. Chem.* 1889, 666—667.

SOLUTION of  $\alpha$ -naphthylamine, even when made from the pure white salt, very soon becomes coloured, and the original delicacy of the reagent is lost. Furthermore, in very dilute solutions (such as one to a thousand millions), the reaction is exceedingly slow, so that in some cases it is even doubtful whether the colouration may not be due to nitrous acid contained in the air. Warming hastens the reaction, but even then 15—20 minutes may be necessary. Høsvay has shown that colouration is more quickly effected if acetic acid be used instead of hydrochloric or sulphuric; and, besides this, the reaction is more marked. He points out also that by boiling solid naphthylamine with water, and pouring off the clear liquid, a solution is obtained which does not darken on standing. He carries out the test as follows:—(a.) 0.5 gram. of sulphanilic acid is dissolved in 150 cc. of dilute acetic acid. (b.) 0.1 gram. of solid naphthylamine is boiled with 20 cc. of water, and the colourless solution poured off; 150 grms. of dilute acetic acid are then added to this solution. About 20 cc. of the sample solution are taken, a few cc. of the sulphanilic acid solution added, and the whole warmed to 70°—80° when naphthylamine solution is added. Red colouration is produced very quickly if only small quantities of nitrous acid be present; if comparatively large quantities be present only a yellow solution is obtained, unless a more concentrated naphthylamine solution is used. The present author introduces an improvement by mixing together the sulphanilic acid and naphthylamine solutions, and keeping them so mixed for use. When the two solutions are kept separate, small quantities of nitrous acid are absorbed from the air by con-

tinual opening of the bottles; such absorption is not indicated by the solutions themselves, and whenever evidence of the presence of nitrous acid in a sample is obtained it is necessary to perform a separate experiment with only the reagents themselves. If the two be kept mixed, however, any absorption of nitrous acid from the air is at once indicated; the solution then only needs treating with a small quantity of zinc dust to fit it perfectly for use.

—T. L. B.

*Analysis of Tungsten, Ferro-Tungsten, Tungsten Steel, Ferro-Chromium, and Chromium Steel.* A. Ziegler. *Dingl. Polyt. J.* 274, 513—528.

TUNGSTEN for steel-making occurs in commerce as a brownish or black powder. Of two samples examined, one was of the former hue, and was partly oxidised, containing only 75 per cent. of tungsten (1.), while the other was a pure black, and in it 91 per cent. of the metal was present (11.). Various methods of attacking these were tried:—Roasting in air (compare *Chem. Zeit.* 13, 65) is slow and uncertain, though it may be aided by the use of ammonium nitrate; hydrofluoric acid only acts on the impurities present, such as iron, manganese, and silicon; aqua regia is effective only on prolonged and repeated digestion; alkaline carbonates act only on the oxide, and therefore require the aid of air to attack the metal; it is possible to approximately gauge the extent of oxidation of any given sample by determining the quantity of alkaline tungstate formed by dusting it into a bath of alkaline carbonate kept very fluid so as to allow the particles to sink promptly; 240 parts of sodium carbonate and 160 parts of nitre are a good flux, but attack platinum; sodium bisulphate dissolves the substance readily; nitric acid is almost without action; hydrochloric acid and potassium chlorate act but slightly. All the above reagents behave alike to the two varieties of commercial tungsten. Strong sulphuric acid formed a certain amount of the blue oxide ( $W_2O_3$ ) with the brownish sample, and had no action on the black; heated, it yielded the blue oxide in both cases; and on cooling gave a blue solution with Sample 11.; concentrated hydrochloric acid caused the evolution of gas with (1.), and the formation of a blue colouration becoming violet; with (11.) a blue solution was obtained, which finally became yellow; on heating, both samples gave a yellow liquid, from which a yellow solid ( $?WO_3$ ) eventually separated; hydrofluosilicic acid of sp. gr. 1.06 gave a permanent blue solution with (1.), and a turbid liquid, blue only by reflected light (on filtering which the liquid was found to be colourless), with (11.). On this a method for determining the proportion of oxide to metal might be based, though strict attention to conditions would be necessary, as, by the use of excess of acid, no perceptible reaction occurred with either specimen. Sodium nitrate was found to attack both samples thoroughly. The separation of tungstic acid from an alkaline tungstate is best effected by sulphuric or nitric acid, and not by hydrochloric acid, which retains a portion in solution.

*Analysis of Commercial Tungsten.*—On these facts the following method is based:—One-half to 1 gram. of the sample is finely powdered in an agate mortar and fused with sodium nitrate in a silver crucible for about half an hour, and the melt dissolved by digestion with water. Any insoluble matter is filtered off and treated with strong hydrochloric acid; should it dissolve slowly or leave a yellow residue, it contains unattacked tungstic acid, and the residue must be re-fused with sodium nitrate, and its aqueous solution dealt with just as the main quantity was. A portion of the tungstic acid present in the residue will have been dissolved by the hydrochloric acid and must be recovered by evaporating twice with nitric acid, the last traces being obtained, if thought necessary, by nearly neutralising the filtrate, acidified with nitric acid, by means of ammonium carbonate, and precipitating with mercurous nitrate. The aqueous solution of the main melt is evaporated to dryness with excess of nitric acid, heated to 120° C., an acid solution of ammonium nitrate added, filtered, and the tungstic acid washed with the same liquid. Traces of tungstic acid are separated by concentrating the filtrate,

nearly neutralising with ammonium carbonate, and precipitating with mercurous nitrate, allowing the mercurous tungstate to settle for 12 hours. At this stage one has (i.) the whole of the tungsten as tungstic acid or mercurous tungstate on the filter; and (ii.) the iron and manganese (together with a little silica) in the filtrate. (A trace of iron which was originally present in the melt as sodium ferrate may be found in the filtrate from the mercurous nitrate treatment; the author suggests the avoidance of this possibility by decomposing the ferrate by passing a stream of carbon dioxide through the aqueous solution of the melt.) The tungsten may be determined by dissolving the mixed tungstic acid and mercurous tungstate through the filter with ammonia, evaporation in a platinum dish, cautious ignition until the weight is constant, and treatment with hydrofluoric acid and re-ignition to volatilise traces of silica, and final weighing. The iron and manganese are best determined in another portion, so as to avoid the difficulty of dealing with the small quantities existing in the filtrates from the mercurous nitrate precipitations of the tungstic acid apart from the main portion dissolved in hydrochloric acid. For this purpose another fusion of the original sample with sodium nitrate is made, the residue insoluble in water dissolved in hydrochloric acid, the solution precipitated with sodium carbonate, the mixed oxides ignited and weighed as  $\text{Fe}_2\text{O}_3$  and  $\text{Mn}_2\text{O}_4$ , then dissolved in hydrochloric acid, evaporated with sulphuric acid (any silica filtered off), in excess of zinc oxide added, the solution made up to a known volume, a portion drawn off and the manganese in it determined by titration with potassium permanganate.

The silica in the sample is best determined on a fresh portion, evaporating the aqueous solution of the melt with sulphuric instead of nitric acid, thus getting the tungstic acid (containing all the silica) in a state less soluble in ammonia than that got by nitric acid, but more readily washed. The weight of the mixed silica and tungstic acid is taken, the former volatilised by hydrofluoric acid, and thus its weight determined.

The author recommends sodium bisulphate, as an alternative flux for attacking tungsten, only when speed is desired at the expense of rigid accuracy, the tungstic acid not separating as perfectly as with sodium nitrate; the mode of work is similar to that described above.

**Analysis of Ferro-Tungsten.**—This may be effected by the process given for commercial tungsten, but the following is a reliable alternative:—1 gm. of the finely powdered sample (obtained by crushing in a diamond mortar and sifting) is gently warmed for some time with concentrated hydrochloric acid, diluted and evaporated with nitric acid of sp. gr. 1.2; the use of dilute aqua regia may be substituted for this treatment. The solution is evaporated on the water-bath with excess of strong nitric acid, and when as nearly dry as possible treated with a solution of ammonium nitrate acid with nitric acid, and filtered. The residue consists of tungstic acid, silica, and unattacked substance. The filtrate contains iron, manganese, and traces of tungsten and silicon; the two latter can be precipitated by mercurous nitrate if it be not intended to determine the two former on the same portion. The residue is dissolved off the filter with hot ammonia solution (1:3) into a platinum dish, evaporated to dryness, ignited, and weighed. Any undissolved matter (original substance not fully attacked) may be fused with sodium nitrate as described under the head of "*Analysis of Commercial Tungsten*."

Silicon is best determined on a fresh portion by heating with sulphuric acid (it is noteworthy that the dilute acid (1:3) acts on ferro-tungsten with evolution of sulphuretted hydrogen), then adding dilute aqua regia to complete the reaction, evaporating until sulphuric acid begins to be driven off, adding water, filtering, and washing with 1 per cent. hydrochloric acid; as silicon, and not tungsten is being estimated, a turbid filtrate is harmless. The precipitate is ignited, weighed, treated with hydrofluoric acid, reweighed, and the silicon reckoned by difference.

**Analysis of Tungsten Steel.**—5 grms. of steel is warmed with 80 cc. of nitric acid of sp. gr. 1.2, 100 cc. of sulphuric acid (1:3) added as soon as the action is over, and

evaporated until sulphuric acid begins to escape. Water is added, the residue is collected, washed with 1 per cent. hydrochloric acid, and used for the determination of silicon and tungsten, separation being effected by hydrofluoric acid. The filtrate serves for the estimation of manganese. Carbon is determined by Eggert's method.

**Analysis of Ferro-Chromium.**—Ferro-chromium, unlike ferro-tungsten, which is almost indistinguishable from ferro-manganese, possesses a characteristic radiating crystalline appearance, and is notably brittle; a sample may be prepared for analysis by crushing in a diamond mortar and sifting the product.

The specimen used by the author contained 67 per cent. of chromium; it was little acted on by nitric or sulphuric acid or aqua regia; hydrochloric acid attacked it fairly, but left a refractory residue. In its tendency to form refractory compounds with iron and manganese, chromium resembles tungsten, and therein lies the chief analytical difficulty. The action of cupric ammonium chloride upon it was incomplete, and, indeed, no single treatment was found effective. 0.5 gm. of the ferro-chromium to be analysed is fused with either sodium nitrate or a mixture of 240 parts of sodium carbonate with 160 parts of potassium nitrate, or one of 4 parts of fused sodium chloride, 1 part of fused sodium carbonate, and 1 part of potassium chlorate.

Instead of this direct treatment it may be previously exhausted with hydrochloric acid, and the residue attacked with a fusion mixture. If the third fusion mixture be used, it is essential to continue the heating for a moderate time only, as prolonged heating decomposes the chromate first formed.

The melt is extracted with water, and the aqueous solution (containing chiefly the chromium) and the residue (containing chiefly the iron and manganese) analysed separately, or together. In the latter case the residue is dissolved in hydrochloric acid (any insoluble matter re-fused), and the solution added to the aqueous solution of the melt, care being taken to keep the whole acid. The mixture is evaporated to dryness, and heated to 120° C. for a quarter of an hour to separate silica, taken up by hydrochloric acid, filtered, and the residue ignited, weighed, and treated with hydrofluoric acid, giving the silica. Any tangible residue of oxides of iron and manganese is dissolved in hydrochloric acid precipitated by sodium carbonate, and re-fused with one of the above fluxes to make sure of the absence of chromium. The filtrate from the silica containing iron, manganese and chromium is treated by Reinhardt's process (Stahl und Eisen, 1889, 5; this Journal, 1889, 572), modified by the author, which is as follows:—

To the boiling solution is added sodium hypophosphite (200 gm.  $\text{NaH}_2\text{PO}_2$  in 400 cc. of water) in quantity sufficient to give a pure green solution without any shade of yellow, pure zinc oxide in suspension in water added while it is still hot, in slight excess, the precipitated chromium, together with the zinc oxide, filtered as quickly as possible, washed, re-dissolved, the reduction and precipitation repeated, the precipitate dissolved in hydrochloric acid, precipitated with ammonia in excess, the chromium hydrate (now nearly free from zinc) re-dissolved, and reprecipitated by a small excess of ammonia. The purity of the chromic oxide after ignition and weighing is proved by fusing it with the second fusion mixture mentioned above.

**Analysis of Chromium Steel.**—Chromium steel may be dissolved by cupric ammonium chloride, but such a mode of solution presents no advantages and contains many pitfalls. The author prefers to dissolve the steel in hydrochloric acid, separate the silica as usual, and the chromium from the iron and manganese by Reinhardt's method (see above). This has the incidental advantage that manganese may be determined in a portion of the same solution. Carbon is best estimated by burning the finely-divided metal in oxygen. If Eggert's test be used, a chromium steel must be taken as a standard, the chromium giving a more or less grey tone to the solution.

Some analytical hints for working these methods are collected at the end of the paper.

A small quantity of tungstic acid will sometimes remain undissolved on the filter through which the bulk has been

washed by ammonia; in this event the filter may be ignited, treated with hydrofluoric acid, and the residue weighed. Nitric acid does not separate silica perfectly, even by evaporation to dryness. Ignited tungstic acid is quite unaffected by heating with hydrofluoric acid, a practice that should be never omitted to eliminate silica. Hydrated tungstic acid is not quite insoluble in any acid, even nitric acid; hence excess should be avoided. Tungstic acid appears to be slightly soluble in silver nitrate. Seeing how refractory are the compounds of tungsten and chromium with iron and manganese (chrome iron ore will serve as a case in point), special watch should be kept over iron precipitates or residues obtained in analyses of alloys of these metals, and their freedom from the two former ascertained.

Ferrie tungstate appears to be somewhat soluble in ammonia under certain circumstances, for a little ferrie oxide often separates during the evaporation of an ammoniacal solution of tungstic acid obtained by the use of nitric acid. On drying commercial tungsten at 120° C. a loss of 0.37 per cent. was observed in one case. All work with solutions of chromium is best done by daylight. Ferro-silicon and ferro-aluminium are not attacked by fusing sodium nitrate sufficiently for analytical purposes.—B. B.

*Examination of a Method for the Determination of Tartar, Tartaric Acid and Malic Acid.* R. Gans. Zeits. f. angew. Chem. 1889, 669—670.

THE author has examined the working of the method given by Borgmann.

The method for determining the tartar is as follows:—50 cc. of wine are evaporated in a porcelain dish to a syrup with addition of a little quartz sand; after cooling about 70 cc. of 96 per cent. alcohol are added, the whole being kept well stirred. After standing in a cool place for 12 hours, filtration is performed, and the residue is washed with alcohol until the washings are no longer acid. The alcoholic filtrate serves for determination of the free tartaric acid. The filter is returned to the dish, hot water is added and the solution is run through a fresh filter paper. Extraction with hot water is repeated until the filtrate no longer shows an acid reaction. The solution is then titrated with decinormal caustic soda solution. For determination of the free tartaric acid, the alcoholic solution before mentioned is made up to a known volume and divided into two equal parts. One part is exactly neutralised with decinormal caustic potash solution; the two parts are then recombined and the alcohol is distilled off. The residue is evaporated to a syrup in a porcelain dish with a little quartz sand, and the determination carried out exactly as before.

The author states, with regard to the tartar determination, that in solutions containing large quantities of sugar part of the tartar is not precipitated by this method, whilst on the other hand part of the free tartaric acid refuses to be washed out from the syrupy mass. With regard to the tartaric acid determination, this is likewise not accurate, as a part of the tartar, not precipitated, and some of the potassium malate which has not dissolved, are estimated along with it; and again a small percentage of the tartaric acid remaining in the tartar residue is not determined in its proper place. If no sugar be present the results obtained are still worse, as almost the whole of the potassium malate separates and is determined as tartaric acid. From such considerations it is also evident that the method cannot be used for the determination of the malic acid present.—T. L. B.

*The Citrate Method of Determining Phosphoric Acid.* O. Reitmaier. Zeits. f. angew. Chem. 1889, 702—709.

FROM an exhaustive consideration of the various reactions which probably take place when phosphoric acid is precipitated from solutions containing oxides of manganese, calcium, iron, and aluminium, by means of magnesia mixture, the author draws the conclusion that a double citro-phosphate of ammonia and magnesia is invariably formed to a greater or lesser extent, and that the less the

excess of magnesia in the solution, the greater is the extent to which this salt is formed, and as a practical outcome he recommends that the more citric acid required to retain the oxides in solution the greater should be the excess of magnesia added above that required to unite with the phosphoric acid.—J. W. L.

*The Determination of Oxide of Iron and of Alumina in Crude Phosphates.* A. Stutzer. Zeits. f. angew. Chem. 1890, 43—44.

THE author points out that the method generally adopted of weighing the iron and alumina in crude phosphates in the form of a mixture of iron and aluminium phosphate, and taking the ferrie oxide and alumina as being equal to one-half of the total weight of the precipitate, is both unscientific and unsatisfactory. It is far better to separate the oxides from the phosphoric acid, and the following method has been found to work well:—The hydrochloric acid solution of the phosphate is made alkaline with ammonia, and then slightly acid with acetic acid. The residue, consisting of the phosphates of iron and alumina, is washed into a beaker, and treated with ammonium nitromolybdate. The precipitate is filtered off and the iron and alumina determined in the filtrate as hydrates. Any molybdic acid carried down by them is removed by redissolving in hydrochloric acid and reprecipitating.

—C. A. K.

*Rapid Methods for Testing Tallow and the Solid Fatty Substances offered as Substitutes for them.* H. Talfe. Bull. Soc. Chim. (3), 2, 209—210.

TALLOW contains its fatty acids combined as glycerides. Only minute quantities of these acids are to be discovered in the free state, owing to spontaneous decomposition or to the treatment of the crude fat whilst hot; the stearic acid serving for the manufacture of candles is on the contrary not in the combined state. The following method is used by the author for testing tallow and stearic acid. Five grms. of the fatty substance to be tested are treated in a capacious crucible with 10 cc. of pure olive oil and gently heated, a drop of turmeric solution added, and after cooling the mixture is titrated with caustic soda lye with continual shaking round. The free acid found is reckoned as stearic acid. In like manner, of course, the free acid in the oil employed for solution must be estimated, after mixing with double its volume of strong alcohol. The free acid in the oil is deducted in the estimation of the fat under examination. In olive oil 2.5 per cent., and in tallow candles 3.6 per cent. of free acid were found. In stearin candles of best quality 100 per cent. of stearic acid were found.

*Simple Process for Discovering Cotton-Seed Oil in Fats and in Olive Oil.* T. Leone. Gazz. Chim. 19, 1889, 355—357.

RECENTLY a process of adulteration has made its way into Italy by which fats of every kind are treated with cotton-seed oil and are made to acquire the taste and melting point of pig's fat. This sophistication can easily be recognised in the following way:—A few cc. of the suspected fat are introduced into a test tube, and an equal quantity of an acid alcoholic solution of silver nitrate added (1 per cent. of  $\text{AgNO}_3$  and 0.5 per cent.  $\text{HNO}_3$ ). The mixture is then warmed for 5 to 6 minutes on the water-bath. If the fat is adulterated there is formed at the point of contact of the two liquids a yellowish-brown ring, whereas in the opposite case, not the slightest colouration is perceptible. An addition of 5 per cent. of cotton-seed oil can thus be recognised with certainty. Similarly the presence of cotton-seed oil in olive oil may be detected.

*Optical Analysis of Oils and Butters.* E. H. Amagat and F. Jean. Compt. Rend. 109, 616—617.

THE oils are placed in a metallic cylinder with glass ends forming an angle of 107°. There is a suitable arrangement for maintaining the less fusible oils in a liquid condition.

The amount of refraction is observed in a special kind of spectrometer not essentially different from the ordinary form of this instrument.

It is found that there is considerable difference in the refractions of various kinds of oils, though the same kind of oil even when coming from different localities has practically the same refraction.

This method of examination is useful for the detection of certain adulterations, for instance, the addition of 10 per cent. oleomargarin to butter may be detected in this way.

—A. L. S.

*Analysis of Manures.* Zeits. f. angew. Chem. 1889, 690—691. (Compare this Journal, 1890, 115.)

THE following are the methods agreed on at a meeting of chemists from the various manure works of Germany:—

#### A. Preparation of the Samples in the Laboratory.—

1. Dry samples of phosphates and artificial manures require sieving and then mixing.

2. In cases of moist manures, a thorough mixing must suffice.

3. On receipt of the sample it must be weighed. Raw products must be sieved, then one-half finely powdered, and the remainder used, simply sieved, for determination of the moisture.

4. Samples should be kept for six months, for reference, stored in good bottles in a cool place.

5. The water of phosphates and bone charcoal must be determined at 105°—110°. Where samples give off ammonium carbonate, this must be determined also.

6. Samples sent for analysis should be fairly selected, should weigh about 500 grms., and should be packed in a bottle with good stopper.

#### B. Determination of Soluble Phosphoric Acid of Superphosphates.—

1. 10 or 20 grms. of the superphosphate are made into a paste with water, and introduced into a measuring flask. The flask is filled up to the containing mark with water, the whole well shaken, allowed to stand, and filtered.

2. The volume of the undissolved residue is here neglected.

3. Volumetric determination of the phosphoric acid is considered out of date.

4. Wagner's modification of the molybdenum method may be used.

5. The citrate method may be used, provided this agree with the results given by the molybdenum method. "Controls" may be performed by the last-named method only.

6. Moisture is to be determined in superphosphates by heating 10 grms. for three hours at 100°. Loss of weight is taken as due to moisture.

#### C. Determination of the Phosphoric Acid soluble in Citrate Solution.—

No conclusions arrived at.

#### D. Determination of the Insoluble Phosphoric Acid.—

1. For determination of the phosphoric acid in bone meal, fish-guano, flesh manures, phosphates, and of the total phosphoric acid in superphosphates, the sample is dissolved in *aqua regia* (3 parts of hydrochloric acid of sp. gr. 1.12 and 1 part of nitric acid of sp. gr. 1.25).

2. Slag powder is dissolved in hydrochloric acid, nitric acid being subsequently added.

#### E. Determination of Iron and Aluminium in Phosphates and Guano.—

The so-called "conventional method" is not to be relied on; the alcohol method (this Journal, 1890, 111) is recommended as worthy of trial.

#### F. Nitrogen Determinations.—

1. Nitrogen in blood, flesh, and similar organic substances is determined either by Kjeldahl's method or with soda lime.

2. Ammoniacal nitrogen is determined by Kjeldahl's method. If it is certain that no other nitrogenous compounds are present, distillation with 5 per cent. caustic soda, magnesia, or lime may be used.

3. The nitric nitrogen in mixtures is determined by the method of Schlösing and Grandean, Lunge, or Kjeldahl and Iodbauer. For determination of nitrogen in saltpetre a direct method is to be sought after; the methods of Kjeldahl and Iodbauer, Schlösing and Grandean, and Lunge (nitrometer method) are permissible.

#### G. Generalities.—

1. The value of phosphates, guano, bone-ash, and saltpetre must be reduced according to the moisture contained in the original sample.

2. Control analyses must never be performed with substance, part of which has been used in the previous analysis. Two reserve samples must be taken for the purpose.

—T. L. B.

*The Simultaneous Estimation of Carbon and Sulphur in Organic Sulphur Compounds.* L. Prunier. Compt. Rend. 109, 904—906.

THE substance to be analysed is mixed with 80—100 times its weight of pure powdered potassium permanganate. The mixture is introduced into a combustion tube and heated in a way similar to that when copper oxide is employed. The gases produced during combustion are allowed to bubble through a solution of potassium permanganate. The apparatus ends in a tube containing baryta water, which should not become turbid if the combustion be properly conducted. On the completion of the experiment, the contents of the tube are dissolved in water and filtered through asbestos to remove the insoluble manganese dioxide. The filtrate is mixed with the solution of permanganate from the bulbs. From half of the combined solutions the sulphuric acid is precipitated as barium sulphate in the usual way, the permanganate being previously destroyed by heating with hydrochloric acid. The other half of the solution is boiled with an excess of sulphuric acid and the carbon is completely converted into carbon dioxide, which may be estimated in the usual way by absorption in potash bulbs. The insoluble portion is treated in a similar manner, the quantity of carbon found being added to that found in the solution. Satisfactory results were obtained in the case of substances containing as much as 65 per cent. of sulphur. The analyses were made on bodies containing, besides carbon and sulphur, hydrogen and oxygen. It remains to be seen whether the method is applicable to nitrogenous bodies.

—H. T. P.

#### PATENT.

*Improvements in Thermostats.* H. Cortland, Toronto, Canada. Eng. Pat. 15,800, October 8, 1889. 6d.

See under XI., page 197.

## New Books.

A DICTIONARY OF APPLIED CHEMISTRY. By T. E. THORPE, B.Sc., Ph.D., F.R.S., Treas. U.S., Professor of Chemistry in the Normal School of Science and Royal School of Mines, South Kensington. Assisted by eminent contributors. In three volumes. VOL. I. 1890. London: Longmans, Green, and Co.; and New York: 15, East 16th Street.

LARGE octavo volume, handsomely bound, price 42s., containing Preface by the Editor, list of Abbreviations of Titles of Journals and Books referred to in the Text, list of Contributors to Vol. I., and 715 pages of Subject-Matter. With the Text are interspersed some 222 well executed wood engravings. The last subject treated of in Vol. I. is "Dysodil," this being immediately preceded by the valuable

contribution on "Dyeing," the volume thus comprises the treatment of subjects extending from A to DY.

In the Preface Professor Thorpe, in an interesting paragraph, refers to the relative positions of Pure and Applied Chemistry, represented, as regards Pure Chemistry, by the new edition of Watts' Dictionary of Chemistry by Morley and Muir, to which, as he says, "the present work may be said to be complementary," and as regards Applied Chemistry, by his own Dictionary now under notice. He adds, "Although the two works are in a broad sense complementary, it is practically impossible to avoid a certain amount of overlapping, and therefore a certain degree of independence. Hence in the present work the chemical history of a product of technical importance, so far as it is known, has often been completed, although its derivatives have, at present, no applications in the Arts. Moreover, such subjects as the Atmosphere, Water, Fermentation, the Chemistry of the Hydrocarbons, the Vegeto-Alkaloids, Glucosides, &c., &c., all of which are dealt with in the other work, find also a place in this Dictionary by reason of their relations to Technology or to Medicine and Sanitation. In all cases, however, these subjects are treated from the standpoint of practical application." The list of the Names of Contributors and the selection of their subjects inspires confidence at once. Without desiring to attempt any comparison of the methods of treatment of the various articles dealt with in the text, or even of referring to all the specially able articles written, a brief extract of some of more immediate interest may be given. Following then the list of Contributors, the subject of *Cellulose* by its very subdivision suggests all that need be said of it. It is first considered physiologically and theoretically, and then a brief account follows of the action of Hydrolytic and Dehydrating agents upon the typical *Cellulose*. After this follows "Technical Application." Next we have enumerated and briefly described the reactions with various reagents, such as Alkalis, Chloride of Zinc, and Sulphuric Acid, leading again to various other *Technical Applications*. "*Oxy-cellulose*" is then treated of, and its relation to and distinction from *β-Oxy-cellulose* explained, and lastly the "*Constitution of Cellulose*" is given and a formula advanced.

The "*Physiology of the Compound Celluloses (Vegetable Fibres)*" is now discussed, and after this their chemistry is considered. These are classified for treatment as follows:—"*Adipocelluloses*" (the protective tissues of plants), the "*Pectocelluloses*," and the "*Lignocelluloses*." In the course of this treatment technical methods are given and some patented processes quoted.

The reader is thus led on to "*Chemical Processes for Disintegrating Wood*," and the article closes with Bibliographic references.

Under the heading of Benzene and its Homologues, a technological feature of special interest, is the engraving and description of Aders' Benzene rectifying apparatus of latest type.

The subject of *Cements* is subdivided as follows:—1. Building Cements. 2. Resinous Cements and Adhesive Materials. Under *Building Cements* we have 1. Lime Burning. 2. Mortar. 3. Plaster of Paris. 4. Hydraulic Mortar. 5. Pozzolana. 6. Hydraulic Cements. 7. Oxchloride Cements. 8. Artificial Stone. 9. Concrete. Specially divided paragraphs are devoted to the consideration of the Causes of Setting of the above Cements. Under Group II., Resinous Cements and Adhesive Materials are considered: 1. Resinous Cements. 2. Oleaginous Cements; and 3. Gummy and Gelatinous Cements.

If the mode of treatment of the subject of *Ammonia* be finally sketched the general arrangement and system of the work will be rendered tolerably clear, though several other subjects, the development of which is just as skillfully conceived, though necessarily more detailed and complex, might be given in abstract.

*Ammonia*.—General Conditions of Formation; Properties; Solubility; and Table by Lunge and Wiernik of Densities of Aqueous Solutions at 15°. *Technical Sources*.—A. Natural Occurrence of Ammoniacal Compounds. B. Ammonia made from Hydrogen and Atmospheric Nitrogen. C. Ammonia from Cyanides, prepared by Means of Atmospheric Nitrogen. D. Ammonia from Urine, Sewage, and

Animal Excreta. E. Ammonia from Guano, &c. F. Bones, Horn, Leather, and other Animal Substances rich in Nitrogen. G. Ammonia formed in Inorganic Chemical Manufactures. H. Ammonia as a By-product in the Manufacture of Beet-root Sugar. I. Ammonia from Peat and similar Matters. K. Ammonia from Bituminous Shale. L. Ammonia from Coal. The "*Working-up of Ammonia Liquors*" is next considered, and then are described, with the aid of engravings, the Ammonia Stills of Coffey, Mallet, Grünberg, Grünberg and Blum, and Feldmann. The manufacturing processes for Ammonium Chloride, Commercial Carbonate of Ammonia, and Caustic Ammonia are also described.

These examples will serve to indicate the general method of treatment and subdivision followed in the case of most of the subjects.

DIE CHEMIE DES STEINKOHLENTHEERS MIT BESONDERER BERÜCKSICHTIGUNG DER KÜNSTLICHEN ORGANISCHEN FARBSTOFFE, VON DR. GUSTAV SCHULTZ. Zweite vollständig umgearbeitete Auflage. Zweiter Band DIE FARBSTOFFE. Sechste Lieferung. Braunschweig: Friedrich Vieweg und Sohn. London: H. Grevel and Co., 33, King Street, Covent Garden. 1889.

THIS, the sixth part of the second volume of Dr. G. Schultz's well-known work on the "*Chemistry of Coal Tar with Special Reference to the Artificial Organic Colouring Matters*," is a simple continuation of the fifth part, without any Preface or other introduction, commencing with page 961 and closing on page 1152, though apparently not completing the work on that page.

The subject first taken up and continued is that of *Colouring Matters of Unknown Constitution*, amongst which *Phthalimide Blue* and the *Thiamine Group* are treated of. We then come to an Appendix containing a series of additions to the Subject-Matter of the preceding volumes, with references to those volumes and to the particular places therein where the additions should come. The larger proportion of these additions are abstracts of German patents, but wherever it has been possible the data have been gathered together and tabulated. This Appendix commences on page 969, and is still under treatment on the concluding page of the sixth part.

## Trade Report.

### TARIFF CHANGES AND CUSTOMS REGULATIONS.

(From the Board of Trade Journal.)

#### SWITZERLAND.

##### Classification of Animal Oil.

Note.—Quintal = 220·4 lb. avoirdupois. Franc = 9  $\frac{9}{10}$  d.

The following decision was given by the Swiss Customs authorities in December last:—

Animal oil (oil of hart-horn, bone oil) raw.—Category 259. Duty, 1 franc per quintal.

#### TURKEY.

##### The Importation of Pharmaceutical Specialities.

The *Journal de la Chambre de Commerce de Constantinople* for the 18th January last states that Colonel Rassim Bey, Inspector-in-Chief of Customs, has just applied the article of the law on the sale of medicines in Turkey,

according to which pharmaceutical specialities, the composition of which is not declared at the Imperial School of Medicine and approved by the council of that school, are not to be admitted into the Empire. Also, pharmaceutical products manufactured in the country, and of which the formula is not deposited with the council of the said school, will be confiscated.

#### UNITED STATES.

##### Recent Customs Decision.

So-called "acid stibic" or white oxide of antimony is dutiable as chemical salt at the rate of 25 per cent. *ad valorem* under T. I. 92.

#### ARGENTINE REPUBLIC.

##### New Customs Law for 1890.

A despatch, dated the 30th December, has been received at the Foreign Office from the Hon. F. J. Pakenham, Her Majesty's Minister at Buenos Ayres, enclosing two copies of the *Boletín Mensual* for November, in which is given the law (a translation of which is also enclosed) regulating the Customs duties for 1890. The following is taken from the translation in question:—

Art. 1. All merchandise from foreign countries shall pay an import duty of 25 per cent. on its value in bond, except the following articles, which shall pay: 50 per cent.:—Gunpowder for sporting purposes, ammunition, and perfumery. 45 per cent.:—Matches, other than wax. 10 per cent.:—Paper for writing and printing. 5 per cent.:—Coarse salt, sulphuric acid, and sulphate of lime, tin and lead plates.

Specific duties as follows:—

Articles.		Rates of Duty.	
		Dols.	Cts.
Starch .....	Per kilo.	0	07
Unrefined sugar.....	"	0	07
Refined sugar.....	"	0	09
Candles.....	"	0	15
Stearin .....	"	0	12
Wax matches .....	"	0	50
Paper, other than for writing or printing	"	0	12

The following articles shall be free of duty:—

Quicksilver, crude sulphur, dynamite, and vegetable fibre for manufacture of paper.

All kinds of produce and manufactures of the country are free of export duties.

#### CANADA.

##### Addition to Customs Free List.

Sulphate of soda, crude only, and for manufacturing purposes only.

## EXTRACTS FROM DIPLOMATIC AND CONSULAR REPORTS.

#### JAPAN.

##### Oil Colours.

These articles are purchased exclusively in England, there being no German competition at present. The demand is extending from year to year, yellow, red, and white being the most popular colours.

#### Drugs and Chemicals.

The total import of drugs and chemicals in 1888 was valued at 1,645,875 yen, of which 956,567 yen were imported into Yokohama. The bulk of these articles are of German origin. All medicinal goods before reaching the Japanese public must be stamped with the words "Fit for medical use," and pass a special board of examiners. Since the beginning of 1888, however, only such preparations as have been prepared according to the instructions of the Pharmacopœia Japonica are admissible. This Japanese Pharmacopœia is issued in Latin and in the vernacular, and modelled very closely upon the German one. The Japanese pharmaceutical industry is hardly yet sufficiently advanced to offer any serious competition to foreign importers. The only Japanese pharmaceutical preparations which have almost completely ousted foreign-made goods from the market are phosphorus, sulphur, sulphuric acid, and nitrate of silver. For the rest no native competition need be feared for at least some years to come; in fact, European medicine being more and more popularised in Japan, the prospects are that this branch of trade will still develop considerably. The following observations refer to the principal drugs sold on the Japanese market:—The demand for boric acid has fallen off; in 1887 the consumption in Yokohama amounted to about 1,000 lb. per month; in 1888 to only about 800 lb. Carbolic acid continues to be bought exclusively in England, but salicylic preparations, another article of great importance, come mostly from Germany. Salicylic acid is principally used in Japan for preserving saké, the national beverage, prepared from rice. The best way to import salicylic acid is to pack it in tin or cardboard boxes, as the local dealers like to put it up in their own vials. The consumption of alcohol is increasing. It is bought in Germany and America, and is packed in kegs. The manufacture of sulphuric ether is about to be taken up in Japan. The trade in atropine has almost entirely gone into British hands during the last few years. German atropine has been frequently objected to by the Japanese authorities on account of its bad quality. Similar objections have been made to bi-muth, which was formerly also bought almost exclusively in Germany. Chloroform finds a regular market in parcels of 500 lb. at the time, but the demand has fallen off owing to the increase in the consumption of cocaine, which is generally sold in 1-grain vials. In quinine Pelletier's brand continues to be popular, but during 1888 German brands have for the first time made themselves seriously felt on the market. Antipyrin and antifebrin are becoming very popular. Iodine preparations come mostly from Germany, but nearly all the morphia used is English. The demand for santaline has declined, but that for pilocarpine in 1-grain vials is slightly on the increase. The use of tannin is increasing in medicine as well as for dyeing purposes. Crystals are almost exclusively used. Caustic soda is becoming an article of importance, owing to the growth of the local soap and paper-making industries. It has hitherto been imported exclusively from England in 500 lb. to 600 lb. iron drums. The following figures refer to the quantities of some of the principal drugs imported into Yokohama in 1888:—Alum, 549,233 cattie; bromide of potassium, 20,146 cattie; quinine, 64,765 oz.; morphia, 5,966 oz.; gum arabic, 40,376 cattie; glycerin, 61,649 cattie; iodide of potassium, 18,598 cattie; carbolic acid, 52,371 cattie; cinchona bark, 41,041 cattie; salicylic acid, 7,701 cattie; amorphous phosphorus, 8,236 cattie; santaline, 3,600 cattie; bicarbonate of soda, 1,397,577 cattie; caustic and crystal soda, 2,653,275 cattie; various drugs and pharmaceutical articles, 545,000 yen; dyestuffs, 816,000 yen; oils, 1,743,000 yen.

#### Japan Wax.

The preparation of vegetable wax is one of the principal industries of the island of Kiu Siu. The wax is gathered in the most primitive manner from the berries of the *Rhus succedanea* (Hazenoki tree). In some districts hydraulic presses have been introduced for its preparation, but the result has not been satisfactory. The best wax is produced in Kummamoto, but does not appear in commerce, as it is all sent to the Imperial Court. The best commercial



wax is made in the province of Iizen, and brought into commerce *via* Osaka. The traders in that place are better capable of purifying and bleaching the wax than the peasants in Kin Sin. In Japan the wax is largely used for candles and in the preparation of hair ointment.

#### PORTUGUESE COLONIES.

##### *Export Duties in Mozambique.*

An export duty is levied on nearly all goods shipped from Mozambique, and this duty, in the case of some articles, such as ivory, is so heavy, as to operate considerably against any expansion of trade. Calumba root and oil seeds, however, only pay 1 per cent.; orchella and gums, 2 per cent., and beeswax 5 per cent. *ad valorem*.

#### SPAIN.

##### *Cream of Tartar.*

In his report on agriculture in Catalonia and Valencia the British Consul at Barcelona states that the plague of phylloxera is increasing at an alarming rate in these provinces, and it is now reported that proprietors are almost decided to cut down their vine plants to the ground with the hope of saving them. This would also injuriously affect the interests of the cream-of-tartar manufacturers, as there will be no wine lees, which is used in its production; these manufacturers are, therefore, getting up a petition to the Government to remove, for the time being, the duty on the importation of French wine lees, otherwise they will have to close their factories.

##### *Olive Oil in Malaga.*

The consumption duties on olive oil are so heavy that Spanish olive oil is actually sold in England at more than 1*l.* a lb. cheaper than in Malaga. Various mercantile associations continue to petition the Government for the abolition or reduction of these duties, but with little or no success.

It is to be regretted that no satisfactory method for refining the oil has ever been put into operation, because, owing to the want of such a system, after the oil extracted from the first pressure the remaining large quantities are used for lubricating machinery or lighting purposes.

#### STRAITS SETTLEMENTS.

##### *Drug Cultivation.*

At the Botanic Gardens in Penang they are hard at work experimenting with the propagation of economic plants. Castor-oil seeds were obtained from the Botanic Gardens at Calcutta. The plants have grown vigorously and are now commencing to fruit. Croton oil grows freely and fruits abundantly. Amatto also grows vigorously and is deserving of a trial on a large scale. Cubebs promise well, and are being propagated as largely as possible.

#### VENEZUELA.

##### *Cocoa-nut Oil and Tonquin Beans.*

The manufacture of cocoa-nut oil has recently been commenced in Venezuela by a Frenchman, M. Boissellier, who has opened a factory at Cumana. Tonquin beans are principally collected in the forest region of the Upper Orinoco Valley, where the bulk of the divi-divi and india-rubber exported from the country are also gathered. These forests are exploited by the Compagnie Française du Haut Orénoque. The principal trade centre for these products is Ciudad Bolivar, to where the goods are carried by the Orinoco steamers.—*Chemist and Druggist*.

## MISCELLANEOUS TRADE NOTICES.

#### PRODUCTION OF SALT IN GERMANY.

The French Ambassador at Berlin says, in a recent report, that a statement has recently been issued by the German Government on the subject of the production of salt works in Germany.

From this statement it appears that in the year 1888-89 there were produced in the Empire, 15,934 tons of crystallised salt, 342,596 tons of rock-salt not crystallised, and 516,521 tons of salt produced by evaporation, the two last qualities showing an increase on the average of the last 10 years. Crystallised salt comes from the Prussian province of Saxony, rock-salt from Wurtemberg, from the province of Saxony, from Anhalt, and from Posen. The salt produced by distillation comes from the provinces of Saxony, Hanover, Alsace-Lorraine, and Thuringia.

In the course of the year 1888-89, 734,623 tons of native salt have been disposed of for the home trade, of which 349,715 tons intended for food have paid the tax of 12 marks per 100 kilos., and 384,908 tons, utilised by industry and agriculture, have been exempt from any duty.

Exports reached 134,171 tons, distributed as follows:—30,212 tons for Bremen, Hamburg, and the other parts of the Empire not included in the Zollverein; 24,567 tons to Austria-Hungary, 19,638 tons to Belgium, 19,107 tons to Holland, 10,514 tons to Russia, 6,534 tons to Denmark, 6,504 tons to Sweden and Norway, 6,444 tons to the British Indies, 5,100 tons to England.

Exported salt does not pay any duty.

Imports were 28,057 tons, of which 25,265 tons were of British origin.

Foreign salt introduced through the ports pays an import duty equal to the tax. That imported by land routes pays 12·80 marks per 100 kilos.

The average consumption is 7·8 kilos. per inhabitant in the whole Zollverein.—*Board of Trade Journal*.

#### SALES OF QUININE IN HOLLAND.

According to the official Dutch notices, the public sales of quinine bark will take place at Amsterdam, during the present year, on the following days:—February 20, March 27, May 1, June 5, July 10, August 28, October 2, November 6, and December 11. The comparison between the results of the sales which took place in 1888 and 1889 shows a considerable increase in favour of the latter year. In the course of 1889 there have been sold 2,073,921 kilos., as against 1,435,047 kilos. in 1888. The State cultivation during the two years was nearly identical, but there was an immense increase in the amount grown by private proprietors in Java. The average of sulphate of quinine in the bark manufactured was 4·12 per cent.—*Ibid*.

#### INDIGO PRODUCTION IN WEST AFRICA.\*

The following information is contained in a letter addressed by Sir Alfred Moloney, Governor of Lagos, to the Secretary of the Liverpool Chamber of Commerce:—

"In Western Africa the indigo industry is, I may say, almost entirely confined to women, and is co-extensive with the cotton manufactured by the natives. Its range may be gauged when we remember the millions of cotton cloths that are there turned out annually for home consumption and export from the crude hand-loom of the country. The cloth export in the direction of Brazil is of special interest and of growing importance; their use has become a fashion there, where such cloths are employed for decorative and other purposes.

"The fashionable colour of native cloths is blue, from the dye to which I now invite attention. The special commissioners who were sent into Yoruba in 1886 by the Government of Lagos saw repeatedly indigo under cultivation, and thus remarked on its use in the town of Ibadan, the estimated population of which is 150,000:—'Nearly all the people wearing blue cloths, the market-place was quite a study in blue.'

\* See this Journal, 1889, VII., 581.



"Within my own experience the industry proceeds extensively on the banks of the river Gambia, where indigo is called in Mandingo *karro*, and in Volof *n'ganyha*; in Yoruba, where *du* is given to it, and in Houssa, where it is known as *suini*, *suni*, or *zuni*, and the plant as *baba*. I give the following translation of a saying in Houssa in explanation of the interest taken in this industry. Schön's 'Dictionary of Houssa Language': 'The place where indigo is growing is good land; its leaves are small, and its seeds are small, but when it is ripe it turns red, and its mouth is pointed. My country is full of indigo and of cotton.'

"Along the valley of the Niger we come across the pure precipitate, the only form in which it is of marketable value.

"In the Gambia and in Yoruba we come across it in the form of balls of decomposed or fermented leaves of indigo plants, generally mixed with cow dung, and of no commercial value in foreign markets.

"The process of extraction of indigo is as follows:—In an earthenware pot of a capacity of 15 gallons, an extract by soaking the leaves is made, and fermentation allowed to follow when the liquid is poured off, and aerated so far as the rapid withdrawal and restoration from a height of 2 or 3 feet of a portion by means of a calabash or some other vessel will allow. Precipitation follows, and when all the dye matter (*fecula*) has sunk to the bottom of the pot the supernatant fluid is poured off. The precipitate, in the form of powder, is then mixed with a little gum and shaped into small balls, cones, &c.

"Dyeing process.—A cloth is soaked in the extract, unaerated, and then freely exposed to the air to dry; the process is repeated until the depth of colour desired is reached. When stripes or other shade designs are desired, patterns are secured by stitching up for process of dyeing portions on which colours are to be lighter. Such confined parts are less affected by the dye, and thus fancy is indulged in."—*Ibid*.

#### MINING INDUSTRY IN ITALY.

The Belgian Consul at Lucca in his last report says that according to the report of the Inspectorate of Mines, the number of mines in operation in Italy amounts to 662, and they employ 49,237 workmen; the total value of the ores extracted amounted to 53,591,771 frs. The provinces which have chiefly contributed to this industry are: Cagliari, where ores to the value of 17,824,000 frs. are extracted; Caltanissetta, where the production is valued at 13,501,985 frs.; Girgenti, which has produced 8,794,750 frs. worth; Tuscany, where the production has exceeded 5,500,000 frs.; and finally in each of the provinces of Arezzo, Grosseto, and Palermo, the value of the production amounted to nearly 2,500,000 frs. To these figures should be added a production of 4,255,372 frs. coming from some mines situated in other provinces.

The values of the principal ores extracted are as follows: iron ores, 2,292,454 frs.; copper ores, 1,100,065 frs.; zinc ores, 6,911,696 frs.; lead ores, 7,128,363 frs.; silver ores, 1,441,400 frs.; and gold ores, 5,327,651 frs.; mercury, 929,865 frs.; sulphur, 27,962,282 frs.; and boracic acid, 1,531,000 frs.—*Ibid*.

#### PRODUCTION OF ALANG-ALANG.

The *Bulletin du Musée Commercial* for January 25th says, that it has now been proved by various experiments conducted by experts, that the plant known as "alang-alang" (*Imperata arundinacea*) can be successfully used as material for the manufacture of paper. This plant grows in the Dutch Indies in boundless profusion, and requires no cultivation. Alang-alang, under advantageous circumstances, grows to the height of a metre and a half, and in Java the farmers are engaged in incessant efforts to keep it within bounds. It can be encouraged to grow anywhere in the humid atmosphere and rich soil of Java, and it is proposed to dedicate some waste lands in the suburbs of Batavia to the growth of alang-alang, a specimen of which has been sent to Brussels by the Belgian Consul-General at Batavia. —*Ibid*.

#### PAPERS INTERESTING TO TECHNOLOGISTS.

The following papers of interest will be found in the *Board of Trade Journal* for February:—

- "Production of Pine Apple Fibre in India," p. 190.
- "German Trade in the Straits Settlements," p. 199.
- "French and Foreign Competition in Senegal," p. 214.
- "Economic and Commercial Condition of Venezuela," p. 225.

#### FURTHER REPORT OF THE LABOUR CONCILIATION COMMITTEE TO THE COUNCIL OF THE LONDON CHAMBER OF COMMERCE.

*Adopted at a Special Meeting of the Council, and Council of Reference, of the London Chamber of Commerce, on Thursday, the 6th February 1890.*

Your committee has endeavoured to devise some practical methods for carrying out the scheme contained in the interim report provisionally adopted by the council on the 12th December.

The committee offers the following suggestions:—

- I. That a permanent body be constituted, to be called "The London Conciliation Board," which shall be affiliated to the London Chamber of Commerce, and that its composition shall be as follows, viz.:—
  - (a.) Twelve members representing capital or employers, to be elected by the council of the Chamber.
  - (b.) Twelve members representing labour, to be elected by the employed.
  - (c.) To these shall be added representatives from the separate trade conciliation committees as herein-after referred to.
  - (d.) The body thus constituted shall add to their number six other members, who from their position, authority, or experience, may be useful in promoting the objects of the London Conciliation Board, three to be appointed by the labour representatives on the Board, and three by the representatives of capital.
  - (e.) Four members, viz., the Lord Mayor of London, or some member of the corporation to be nominated by him, the Chairman of the London County Council, or some member of the council to be nominated by him, a representative of the London Trades Council, and a representative of a London labour organisation to be selected by the labour representatives on the Board.

The elections shall take place every three years, and the first election shall take place on the

II. The duties of the London Conciliation Board shall be as follows:—

- (a.) To promote amicable methods of settling labour disputes and the prevention of strikes and lock-outs generally, and also especially in the following methods:—
  1. They shall, in the first instance, invite both parties to the dispute to a friendly conference with each other; offering the rooms of the Chamber of Commerce as a convenient place of meeting. Members of the committee can be present at this conference, or otherwise, at the pleasure of the disputants.
  2. In the event of the disputants not being able to arrive at a settlement between themselves, they shall be invited to lay their respective cases before the committee, with a view to receiving their advice, mediation, or assistance. Or, should the disputants prefer it, the committee would assist them in selecting arbitrators, to whom the questions at issue might be submitted for decision.
  3. The utmost efforts of the committee shall, in the meantime, and in all cases, be exerted to prevent, if possible, the occurrence or continuance of a strike or lock-out until after all attempts at conciliation shall have been exhausted.

The London Conciliation Board shall not constitute itself a body of arbitrators except at the express desire of both parties to a dispute, to be signified in writing, but shall, in preference, should other methods of conciliation fail, offer to assist the disputants in the selection of arbitrators chosen either from its own body or otherwise. Any dispute coming before the Board shall, in the first instance, be referred to a conciliation committee of the particular trade to which the disputants belong, should such a committee have been formed and affiliated to the Chamber.

(b.) To collect information as to the wages paid and other conditions of labour prevailing in other places where trades or industries similar to those of London are carried on, and especially as regards localities either in the United Kingdom or abroad, where there is competition with the trade of London. Such information shall be especially placed at the disposal of any disputants who may seek the assistance of the London Conciliation Board.

III. The separate trade conciliation committees shall be composed of equal numbers of employers and of employed.

Each trade shall elect its own representatives, employers and employed voting separately for the election of their respective representatives. The number of members and the general rules of procedure shall be determined by each particular trade, subject to the approval of the London Conciliation Board.

The trade conciliation committees shall be affiliated to the London Chamber of Commerce, and shall be represented upon the London Conciliation Board. Any trade conciliation committee constituted as above, representing a body or trade in the metropolitan districts of more than 1,000 individuals, shall send two representatives to sit on the London Conciliation Board, one being an employer and the other an operative workman, each to be separately elected by employers and employed respectively. In the case of trade conciliation committees representing bodies or trades in the metropolitan districts smaller in number than 1,000 individuals, two or more such committees may unite together to elect joint representatives to the London Conciliation Board.

It shall be the duty of the trade conciliation committees to disown matters of contention in their respective trades; to endeavour amicably to arrange the same, and in general to promote the interests of their trade by discussion and mutual agreement. In the event of their not being able to arrange any particular dispute, they will refer the same to the London Conciliation Board, and in the meantime use their most strenuous endeavours to prevent any strike or lock-out until after the London Conciliation Board shall have exhausted all reasonable means of settlement.

They may, from time to time, consider and report to the London Conciliation Board upon any matter affecting the interests of their particular trade upon which it may be thought desirable to employ the action or influence of the London Chamber of Commerce as a body.

IV. The London Chamber of Commerce places its rooms at the disposition of the London Conciliation Board, and of the trade conciliation committees for holding their meetings. Any alterations in the rules and regulations of these bodies which may be from time to time proposed, shall be submitted for approval to the council of the Chamber.

V. The above regulations shall be subject to bye-laws, to be specially framed for the purpose, and which shall be open to amendment as required from time to time, on agreement between the council of the Chamber of Commerce and the London Conciliation Board.

Here follow the names of the Members of the Committee, January 31st, 1890.

#### *Explanatory Circular.*

The council has had particularly in view the necessity of giving capital and labour an equal representation, and equal voting power in the organisation of the proposed London Conciliation Board and committees.

Attention is specially called to the fact that this Board will not in itself be primarily, or of necessity, an arbitrating body, but its main object will be to bring the interested parties together for purposes of discussion and explanation at the earliest stage of any difficulty, with the view of preventing that difficulty reaching the acute stage of strike or lock-out, arbitration being only suggested in the last resort. It is hoped that the existence of a permanent board, composed of experienced and influential representatives of both capital and labour, to whom any difficulty arising in the labour market can be referred for discussion and consideration at an early period may, by intervention in a friendly and disinterested manner, enable points of difference to be settled by mutual consent, without any stoppage of trade. Should the difficulty, however, prove to be acute, or not one of a nature for settlement on general grounds, the Board will have powers to assist the disputants in nominating special arbitrators. One very important function of the Board will be to assist in promoting the formation of separate trade conciliation committees for the various industries carried on in the metropolis, to whom all disputes affecting those particular industries will in the first instance be referred.—*London Chamber of Commerce.*

#### THE CULTIVATION OF INDIGO IN CHINA.

*M. Jaues. Bull. de la Soc. d'Encour. pour l'Ind. Nat.*  
1889, 494.

Indigo is very largely cultivated in the province of Tchin-Kiang; each family requires the colour for dyeing their garments, the excess being sent to market. It is a plant which is very easily cultivated; it requires a special soil, as close as possible. When one month old the plants are replanted at distances of about 50 centimetres apart. Very little care is necessary, and in dry seasons the plants need not even be watered. When they attain a height of 60 centimetres they are cut down to the root and removed to pits dug in the earth or built of bricks of about 160 litres capacity. The roots throw out new shoots and yield a second crop. The plants are piled in the pits and covered with pure water and allowed to remain for some days, when a fermentation is set up. The operation may be hastened by the addition of a little chalk and by frequent agitation of the mass. At the end of the operation the indigo forms a thick blue layer at the bottom of the pit and it only requires drying to render it marketable.—*E. J. B.*

#### A NEW TIN ISLAND IN THE DUTCH INDIES.

The latest reports from Singkep, an island belonging to the Lingga group on the east coast of Sumatra, state that the prospects of finding permanent tin deposits there, similar to those in Banea and Biliton, are highly favourable. The concession for working these mines is in the hands of a private company.—*Industries.*

#### DYNAMITE IN SPAIN.

Although there is a large dynamite works at Bilbao, there is a good opening for another such establishment in the centre of Spain to supply the markets of Linares, the coal basin of Belnez, and the large and increasing mining districts in the provinces of Ciudad Real and Estremadura. The greatest inconvenience and delay are experienced in getting regular supplies in consequence of the difficulties of carriage. The dynamite from Bilbao must either pass through two systems of railways which are not on the best understanding with one another, or must go by sea to Seville, and thence a long distance overland. The Bilbao works are reported as having done a good business for many years.—*Ibid.*

#### WOOD-PULP MANUFACTURE IN SCANDINAVIA.

The erection of new wood-pulp manufactories continues, although the prospects cannot be said to be particularly bright. There will shortly be 54 wood-pulp manufactories at work in Norway, against the 50 working last year; and their probable production has been put at about 250,000 tons. The export during last year was about 190,000 tons, against

a production of 175,000 tons in the previous year. The above figures refer to "mechanical" wood-pulp. About 25,000 tons of "chemical" wood-pulp, of which rather more than half was wet, was also exported, being in excess of the figures for 1888. At the end of 1889 there were 15 manufactories of chemical pulp at work—11 sulphite and 4 soda cellulose works. Their production is estimated at 30,000 tons sulphite (wet) and 9,000 soda cellulose (dry). During last year chemical pulp maintained its price and commanded a ready sale—the exports being valued at about 200,000*l.* f.o.b. Norwegian port. The profits thereon were not equally satisfactory owing to the rise in raw materials. For mechanical wood-pulp the prices, which have been declining for the last 20 years, fell to 2*l.* 2*s.* 6*d.* per ton f.o.b. Hull. Attempts have been made to reduce and regulate production but without success. The value of mechanical pulp exported during 1889 amounted to about 450,000*l.* Increased production has given an impetus to the Scandinavian paper industry. In Sweden two or three paper manufactories have been formed of late, and in Norway existing ones have been materially extended. The effect of these extensions will be no doubt apparent before long.—*Ibid.*

#### PROGRESS OF THE BASIC OR THOMAS-GILCHRIST PROCESS DURING THE 12 MONTHS ENDING 31ST DECEMBER 1889.

The total make of steel and ingot iron from phosphoric pig during this period amounts to 2,274,552 tons, being an increase over the make for the previous 12 months of about 321,318 tons, and making the total production of basic steel to this date 10,845,000 tons.

It will be noticed that of the above-mentioned make of 2,274,552 tons, no fewer than 1,764,639 tons were ingot iron containing under 17 per cent. of carbon.

The makes of the various countries for the year ending 31st December 1888 and 31st December 1889, respectively, are as follows:—

	1889.		1888.	
	Total.	With under 17 per Cent. Carbon.	Total.	With under 17 per Cent. Carbon.
England .....	Tons. 493,919	Tons. 318,828	Tons. 408,594	Tons. 276,176
Germany, Luxemburg, and Austria .....	1,431,642	1,185,323	1,276,070	1,026,033
France .....	222,392	153,271	222,333	158,223
Belgium and other countries .....	76,599	71,217	46,237	32,360
	2,274,552	1,764,639	1,953,234	1,493,032

With this 2,274,552 tons of basic steel, were produced some 700,000 tons of slag (containing about 36 per cent. of phosphate of lime), most of which was used as a fertiliser.

#### THE STASSFURT INDUSTRY.

The Syndicate of the Alkali Works has issued its report of the sales during 1889. It appears that the deliveries of potassium chloride during the first half of 1889 amounted to 1,200,000 centners, and during the last half to 1,271,000 centners, as against 1,119,000 centners and 1,126,700 centners during the corresponding periods of 1888.—*Industries.*

#### WEIGHTS AND MEASURES ACT, COMMENCING 1ST JANUARY 1890.

The following clauses of the above Act are of importance:—

"Every weighing instrument used for trade shall be verified and stamped by an inspector of weights and measures, with a stamp of verification under this Act.

"Every person who, after the expiration of 12 months from the commencement of this Act, uses, or has in his possession for use for trade any weighing instrument not stamped as required by this Act, shall be liable to a fine not exceeding two pounds, or in the case of a second offence five pounds."

"In this Act, unless the context otherwise requires, weighing instruments includes scales, with the weights belonging thereto, scale-beams, balances, spring-balances, steelyards, weighing machines, and other instruments for weighing."

#### ALCOHOL IN THE INDUSTRIAL ARTS.

(Report for Year 1889 of United States Commissioner of Internal Revenue, p. 137.)

A Bill (H.R. 9051, Fiftieth Congress, First Session), having for its object the reduction of taxation and simplification of laws in relation to the collection of the revenue, has been printed and contains certain provisions relative to alcohol in the industrial arts. The Commissioner summarises its provisions as follows:—

Section 3 provides for bonded warehouses for storing the alcohol under internal revenue storekeepers and regulations.

Section 4 provides for the transfer of the alcohol from distillery warehouses to the bonded warehouses provided in section 3.

Section 5 provides for the removal of alcohol from the bonded warehouses to manufacturers' storerooms, to be there used in the industrial arts and in the manufacture of drugs and chemicals; proprietary articles and beverages being excepted. This section also provides certain regulations under which bonded warehouses and manufacturers' storerooms may be established.

Section 6 provides for the use of warehouse stamps.

Section 7 makes further provision for removals from warehouse to manufacturers' storerooms.

Section 8 gives revenue officers right of access to every part of the manufactory.

Section 9 provides for the methylation of alcohol in the bonded warehouses and for the use of methylated spirits in the arts and manufactures, for burning, and in the manufacture of compounds, except beverages. Bonds and returns may be exacted. Right of access to the revenue and penalties against fraud are provided.

Section 10 provides penalties for improper use of alcohol.

Section 11 provides for assessing taxes on spirits not used within three years, forbids use of methylated spirit within 600 ft. of a rectifier or distiller, and imposes penalties for breaches of the various provisions of the law and regulations.

The Commissioner in the main approves the Bill, but suggests that section 9 should be so modified as to provide that no methylated spirits shall be permitted on any premises in which distilling or rectifying is carried on, or where wines or spirits are kept for sale as beverages.

The Commissioner considers that since the object of demethylation is to secure a cheap alcoholic beverage, the importance of separating methylated spirits from all stocks of such beverages is very great; as is also that of keeping methylated spirit out of the hands of distillers and rectifiers who use stills.

The Commissioner, from data in his possession, estimates the quantity of alcohol likely to be used annually in the arts at about eight million gallons—and the additional expense required to carry these provisions into effect to be about 290,000 dols. per annum.—C. G. C.

#### COPPER ROASTING IN SPAIN.

About eighteen months ago the open-air calcination of copper ores in the province of Huelva was prohibited by a decree signed by Señor Albareda, then Home Secretary, now

Spanish ambassador in London. The order was to come into force on January 1, 1890. The Rio Tinto Company, as well as private copper-roasters, tried all they could to have the decree rescinded, urging, among other arguments, that the prohibition implied a breach of public faith, because calcination in the open air was practised in connexion with the copper works which had originally been made over by the Government to the various companies by whom the *explotacion* is now carried on. Having failed in their endeavour, the companies had no option but to discontinue the roasting of copper altogether, and dismiss the men who had hitherto been employed on this work. The number of those discharged in this way ranges, according to conflicting reports, from 500 to 1,500. Those are probably nearest the mark who set down at 2,000 the number of persons who have been deprived of their means of subsistence by the stoppage of the calcination. Some 250 of the discharged men proceeded to Huelva directly after New Year's Day, to lay their case before the Governor of the province, who was greatly surprised, for he had no idea that there were so many men wanted to make up the heaps. *La Epoca* relates that the spokesman of the roasters gave "the most excellent gentlemen" quite a lecture on the subject, pointing out to him that making up the heaps was only a small portion of the work; that the *teleros* must be minded and attended to, and that, before they are made up, the ores must be got out, picked, and worked up, all of which involves a numerous *personnel*; that there are, moreover, tools, machinery, and trains to which a number of men have to attend, and lots of other things to be done of which the Governor of Huelva was perfectly ignorant, although the copper mines are situated within his province. The latest intelligence shows that the Government have called upon the companies to employ the men in some other way, and that the companies have replied that they had no other work to offer to the men. Since the end of the ministerial crisis it has been expected that immediate means would be taken to relieve those whose living has been taken away from them by the ministerial decree.—*La Epoca*.

#### COMBINATION OF NITRATE MAKERS.

The following heads of agreement have been unanimously adopted by the nitrate companies in London, and telegraphed out to Iquique:—"It is agreed to form a combination of nitrate makers, representing not less than 90 per cent. of the whole, to restrict the production of nitrate to the estimated consumption, the amount of production to be fixed from time to time by the nitrate committee in London, in conjunction with the committee in Iquique. It is also agreed that, so far as the committee may be able to regulate the selling price of nitrate, such price shall not exceed 9*l.* 10*s.* per ton. A committee, consisting of seven members residing in Iquique, to be elected to ascertain the production and fix the quota to be assigned to each oficina. The quotas to be fixed on the following basis, and to be agreed to by all the members of the committee:—1. The basis of the calculation of the quotas for all oficinas completed on 1st January 1889 to be the actual quantity of nitrate produced by them during 1889. 2. Oficinas which were not completed by 1st January 1889, to have the option of selecting any three consecutive months' production, as shown by their books, as a basis of their annual production. 3. No new oficina to have any quota until actually producing. The new iodine combination formed on the coast to be approved so soon as the present nitrate combination be completed. The London committee to at once advise the iodine committee in Iquique that the late combination is to be continued until the new one can be put in force."—*Standard*.

#### THE PETROLEUM STATISTICS.

If figures can be believed, it would seem that the consumption of Pennsylvania oil in excess of production will soon bring us face to face with an oil famine, so far as the Pennsylvania product is concerned. It is not a question of the contraction of national transit certificates in existence, as that might arise from an artificial condition

of affairs, created by the issuers and manipulators of them; but the actual consumption at home and the amount exported largely exceeds the production. What the possibilities are for increasing the production is hard to say with certainty, but judging from the small results obtained by the energetic prospecting operations which have been carried on through a great portion of the past year, the probabilities in favour of great or steady increase are small. If this is the true situation, and everything points to it being so, we are fast approaching the time when the accumulated surplus of past years will be exhausted, and at the present rates of consumption and production the latter would be from 20,000 to 30,000 barrels a day behind the former. The energy with which prospecting for new territory is being carried on can be judged of from the enhanced prices paid for even possible oil lands, and the fact that in the past year there were 5,700 wells completed against 1,700 in 1888, and 1,800 in 1887. Prospecting was more active than in the three preceding years, 1884-86, but in the first of these, in which there were the greatest number of wells completed of recent years, the figure only reached 3,400.

The daily output for the year 1889 gave an average considerably in excess of that for 1888, viz., 60,256 barrels, but at the same time it was slightly inferior to that of 1887, which amounted to 61,875 barrels, and greatly below that of 1886, while all the time the consumption has been largely and steadily increasing. The reduction in net stocks by all pipe lines has gone on at about the rate of 650,000 barrels a month, as they amounted on December 31st, 1888, to 18,604,474 barrels, and on the corresponding date in the past year to about 10,800,000. It is unnecessary, however, to contemplate the time when, with stocks exhausted, we shall suddenly find ourselves with a demand that cannot be satisfied, as before that happens the price will surely adjust itself, and the demand will be curtailed in consequence of the article becoming, in certain cases, a luxury instead of a necessity.

The year has opened with a smaller daily production than what we have already recorded as the average of the past year, and the figures point to a large reduction of stocks during the current month, being 54,588 barrels a day, against deliveries, 79,597. Whether this state of affairs will continue can only be more or less a matter of guesswork, and dependent on the foreign markets, but it is interesting to note the decline of shipments of American petroleum to Great Britain during the four years ending 1888, and the increase of Russian petroleum imported into that country. In 1885 there were 1,367,720 barrels of American oil imported against 70,149 barrels Russian; in 1886, 1,363,801 against 46,814; in 1887, 1,444,350 against 188,461; and in 1888, 1,286,148 against 549,126.—*Eng. and Min. Journal, New York*.

#### MINERAL STATISTICS OF CANADA.

The official report of the mining and mineral statistics of Canada, which has just been issued by the Geological and Natural History Survey, gives a list of about twenty-six minerals which are obtained in Canada in quantities of commercial importance. The mineral of most value is, of course, coal, of which in 1888 over 2,500,000 tons were mined, the value at the pit's mouth being over 5,250,000 *dols.* Nova Scotia yields the greatest quantity, nearly 2,000,000 tons, valued at 3,000,000 *dols.*; and British Columbia is second with 500,000 tons, valued at nearly 2,000,000 *dols.*; the North-West territories, whose mines had just been opened, having yielded over 100,000 tons; and New Brunswick about 6,000 tons. Canada exported about 2,000,000 *dols.* worth of coal, and imported about 9,000,000 *dols.* worth, about 5,500,000 *dols.* of which was, however, anthracite, not found in Canada.

Canada's production of iron ore was small—only 79,000 tons, valued at about 150,000 *dols.*, having been mined during 1888. About 22,000 tons of pig iron, of the value of about 313,000 *dols.*, were manufactured in Canada in 1888, a decrease, in spite of the increased duties on pig. Sir Charles Tupper levied duties on pig iron in order to encourage smelting and stimulate mining enterprise.

The following table shows the growth of Canada's product in pig iron:—

*Pig Iron made.*

Year.	Tons.
1876 (no duty; no bounty) .....	4,000
1877 " " .....	13,500
1878 " " .....	16,600
1879 (import duty, 2 dols. per ton) .....	16,500
1880 " " .....	23,600
1881 (duty, 2 dols. per ton; bounty, 1'50 dols. per ton) .....	18,500
1882 " " .....	21,500
1883 " " .....	32,000
1884 " " .....	29,389
1887 (duty, 4 dols. per ton; bounty, 1'50 dols. per ton) .....	24,829
1888 (duty, 4 dols. per ton) .....	21,700

The increase was greater during the period in which there was no duty than during any other period. Canada imported over 68,000 tons of pig iron, handling over 500,000 tons of scrap iron and steel, and about 12,000,000 dols. worth of iron in one form or another, upon which duties amounting to nearly 3,000,000 dols. were paid. There is, therefore, plenty of scope for the development of Canada's iron resources. Of Canada's whole production of iron ore, Nova Scotia is credited with more than half, or over 42,000 tons, Quebec with 10,000, Ontario with 16,000, and British Columbia with 8,000.

The production of gold for the year amounted to over 61,000 oz., valued at over 1,098,000 dols. The rapid development of silver mining in the Lake Superior region is shown by the statistics of Ontario production, which has increased from 16,505 dols. in 1886 to 184,763 dols. in 1887, and to 208,064 dols. in 1888, and the total value of the Dominion's silver production for 1888 amounted to 395,377 dols. Quebec was the only province that mined copper in 1888, the value of which was placed at 667,543 dols., an increase over the production of the two preceding years. Canada is not rich in lead, the only production being that of British Columbia, worth 27,472 dols. The Kootenay and Field mines were the most productive. Platinum to the value of 6,000 dols. was produced in British Columbia, and manganese was produced by Nova Scotia and New Brunswick to the extent of 1,801 tons, of the value of 47,994 dols. The mica product is valued at 30,207 dols. A variety of other metals and mineral substances are alluded to, found in small quantities, but growing in importance. On the west shore of Lake Memphre Magog a rich galena vein has been discovered. An assay shows it contains 70 per cent. of lead and 15 per cent. of silver. The property, which contains over 200 acres, is controlled by McDonald Brothers, Sherbrooke.

**BOARD OF TRADE RETURNS.**

**SUMMARY OF IMPORTS.**

	Month ended 31st January	
	1889.	1890.
	£	£
Metals.....	2,157,316	2,085,663
Chemicals and dyestuffs.....	912,914	810,293
Oils.....	499,769	601,519
Raw materials for non-textile industries.....	2,769,978	2,651,310
Total value of all imports ....	38,958,318	38,143,850

**SUMMARY OF EXPORTS.**

	Month ended 31st January	
	1889.	1890.
Metals (other than machinery) ....	£ 3,056,923	£ 3,577,155
Chemicals and medicines .....	668,719	689,818
Miscellaneous articles.....	2,491,125	2,573,383
Total value of all exports.....	20,527,057	21,860,752

**IMPORTS OF METALS FOR MONTH ENDED 31ST JANUARY.**

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Copper:—				
Ore..... Tons	18,358	5,448	£ 163,874	£ 44,945
Regulus..... "	6,918	8,141	263,401	219,966
Unwrought .... "	3,917	4,839	301,373	252,210
Iron and steel:—				
Iron ore..... "	323,945	355,957	237,625	288,193
Iron bolt, bar, &c. "	5,390	5,743	47,904	50,250
Steel, unwrought.. "	608	485	6,534	5,225
Lead, pig and sheet "	12,997	16,461	164,822	223,721
Pyrites..... "	89,264	61,524	180,212	116,440
Quicksilver..... Lb.	543,925	841,194	65,372	118,618
Tin..... Cwt.	58,917	54,445	291,916	257,519
Zinc..... Tons	4,369	4,737	73,436	106,238
Other articles ... Value £	..	..	360,547	402,638
Total value of metals	..	..	2,157,316	2,085,663

**IMPORTS OF CHEMICALS AND DYESTUFFS FOR MONTH ENDED 31ST JANUARY.**

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Alkali..... Cwt.	6,637	2,888	£ 1,771	£ 1,955
Bark (for tanners, &c.)..... "	18,663	32,311	7,419	12,878
Brimstone..... "	29,666	41,330	6,582	8,845
Chemicals..... Value £	..	..	108,584	109,190
Cochineal..... Cwt.	486	1,205	2,951	7,648
Cutch and gambier Tons	1,846	2,529	47,303	64,442
Dyes:—				
Aniline..... Value £	..	..	18,484	19,745
Alizarine..... "	..	..	29,964	29,777
Other..... "	..	..	1,800	1,930
Indigo..... Cwt.	18,812	15,902	411,494	316,676
Madder..... "	1,078	740	1,449	1,905

IMPORTS OF CHEMICALS AND DYE-STUFFS FOR MONTH ENDED 31ST JANUARY—*continued*.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Nitrate of soda.... Cwt.	179,812	141,276	£ 96,526	£ 68,385
Nitrate of potash. „	37,833	33,145	32,675	29,729
Valonia ..... Tons	3,336	2,121	47,443	35,851
Other articles... Value £	..	..	128,460	108,237
Total value of chemicals	..	..	942,014	810,293

## IMPORTS OF OILS FOR MONTH ENDED 31ST JANUARY.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Cocoa-nut..... Cwt.	15,397	24,885	£ 19,929	£ 30,693
Olive ..... Tons	965	1,415	32,943	53,394
Palm ..... Cwt.	75,122	74,266	86,216	79,077
Petroleum ..... Gall.	6,227,762	8,185,795	181,784	230,103
Seed ..... Tons	1,628	1,338	45,906	38,564
Train, &c..... Tons	1,188	1,710	23,983	35,300
Turpentine ..... Cwt.	26,761	37,800	45,953	62,660
Other articles .. Value £	..	..	63,055	81,726
Total value of oils ...	..	..	490,769	601,519

## IMPORTS OF RAW MATERIALS FOR NON-TEXTILE INDUSTRIES FOR MONTH ENDED 31ST JANUARY.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Bark, Peruvian .. Cwt.	19,196	11,661	£ 71,606	£ 36,947
Bristles..... Lb.	309,520	195,362	43,065	25,116
Caoutchouc..... Cwt.	22,507	22,104	273,580	274,241
Gum:—				
Arabic..... „	9,592	3,300	43,884	11,980
Lac, &c..... „	4,835	4,452	14,911	18,672
Gutta-percha .... Cwt.	2,399	5,158	23,370	55,038
Hides, raw:—				
Dry..... „	54,229	46,591	151,433	122,028
Wet..... „	50,931	47,723	119,203	105,019
Ivory..... „	1,481	870	72,312	47,100
Manures:—				
Guano..... Tons	1,182	5,782	7,412	40,695
Bones..... „	6,972	5,352	34,090	29,230
Paraffin..... Cwt.	19,277	28,536	24,133	32,954
Linen rags..... Tons	4,655	2,811	38,044	29,388
Esparto, &c. .... „	31,475	31,163	166,744	148,676
Pulp of wood .... „	10,280	8,348	53,168	51,806
Rosin..... Cwt.	94,814	200,417	21,504	41,655
Tallow and stearin „	96,682	138,790	149,003	173,036
Tar ..... Barrels	2,321	3,867	1,346	2,453

IMPORTS OF RAW MATERIALS FOR NON-TEXTILE INDUSTRIES FOR YEAR ENDED 31ST JANUARY—*cont.*

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Wood:—			£	£
Hewn ..... Loads	137,012	103,832	268,854	230,566
Sawn ..... „	97,868	63,775	225,427	185,517
Staves ..... „	10,226	5,699	59,306	38,080
Mahogany ..... Tons	1,078	2,850	8,592	22,411
Other articles... Value £	..	..	889,082	928,292
Total value .....	..	..	2,760,078	2,651,310

Besides the above, drugs to the value of 83,810*l.* were imported as against 57,958*l.* in 1889.

## EXPORTS OF METALS OTHER THAN MACHINERY FOR MONTH ENDED 31ST JANUARY.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Brass..... Cwt.	6,307	8,783	£ 33,600	£ 40,236
Copper:—				
Unwrought ..... „	13,298	69,541	53,224	165,118
Wrought..... „	8,721	38,298	40,309	115,007
Mixed metal .... „	18,763	36,781	65,157	99,847
Hardware..... Value £	..	..	289,142	231,538
Implements..... „	..	..	84,290	115,734
Iron and steel.... Tons	300,324	302,751	2,158,679	2,531,563
Lead ..... „	5,901	3,358	86,980	54,989
Plated wares... Value £	..	..	27,061	29,658
Telegraph wires... „	..	..	113,230	71,686
Tin ..... Cwt.	7,713	5,866	39,975	29,520
Zinc ..... „	6,159	11,905	6,613	11,254
Other articles .. Value £	..	..	59,563	81,625
Total value .....	..	..	3,056,923	3,577,135

## EXPORTS OF DRUGS AND CHEMICALS FOR MONTH ENDED 31ST JANUARY.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Alkali..... Cwt.	515,059	522,455	£ 133,605	£ 115,510
Bleaching materials „	139,872	134,536	54,228	39,525
Chemical manures. Tons	27,736	20,427	189,784	167,866
Medicines..... Value £	..	..	79,938	£ 83,852
Other articles ... „	..	..	201,164	253,125
Total value .....	..	..	638,719	689,818

EXPORTS OF MISCELLANEOUS ARTICLES FOR MONTH  
ENDED 31ST JANUARY.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Gunpowder..... Lb.	939,700	495,400	22,333	13,228
Military stores.. Value £	..	..	92,526	76,296
Candles..... Lb.	980,500	1,082,800	18,742	20,206
Caoutchouc ..... Value £	..	..	82,875	95,251
Cement..... Tons	49,177	47,038	94,934	94,519
Products of coal Value £	..	..	169,256	135,986
Earthenware ... "	..	..	156,621	154,964
Stoneware ..... "	..	..	12,251	18,816
Glass:—				
Plate..... Sq. Ft.	366,582	346,466	21,188	19,078
Flint..... Cwt.	9,612	8,516	22,298	21,318
Bottles..... "	88,969	76,107	42,146	34,876
Other kinds.... "	17,173	17,483	13,210	15,110
Leather:—				
Unwrought .... "	9,994	12,508	91,568	113,810
Wrought ..... Value £	..	..	35,716	27,412
Seed oil..... Tons	5,505	4,814	116,597	107,598
Floor cloth ..... Sq. Yds.	1,631,300	1,493,490	78,724	65,461
Painters' mate- rials..... Value £	..	..	122,771	114,180
Paper ..... Cwt.	95,645	80,282	165,667	140,023
Rags..... Tons	5,764	4,281	47,697	31,246
Soap..... Cwt.	39,960	39,519	39,085	41,103
Total value .....	..	..	2,491,125	2,573,383

## QUICKSILVER.

—	Imports.	Exports.	Price.	
			Highest.	Lowest.
1889	Bottles. 67,664	Bottles. 57,658	£ s. d. 9 12 0	£ s. d. 7 7 0
1888	73,770	47,132	11 0 0	6 15 0
1887	61,212	62,476	11 5 0	6 10 0
1886	58,966	66,080	7 10 0	5 13 0
1885	55,153	48,823	6 15 0	5 10 0
1884	59,970	52,402	6 15 0	5 2 0
1883	51,520	48,997	5 17 6	5 2 0
1882	45,921	40,424	6 5 0	5 14 0
1881	56,261	24,842	7 0 0	6 2 6
1880	49,541	16,070	7 15 0	6 7 6

Estimated Consumption, United Kingdom, 12,000 bottles per annum.

—Alex. S. Pickering.

## Monthly Patent List.

\* The dates given are the dates of the Official Journals in which acceptances of the Complete Specifications are advertised. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the said dates.

## I.—GENERAL PLANT, APPARATUS, AND MACHINERY.

## APPLICATIONS.

606. J. Brock and T. Minton. Filter presses. January 13.  
668. J. S. Rigby and J. R. Wyld. Apparatus for separating from each other the solid and liquid constituents of mud and other semi-fluid mixtures, or for washing said solid constituents. January 14.

828. A. Smith. Filters for the purification of water and other liquids. January 16.

1122. T. E. Wilson. The concentration, evaporation, and distillation of liquids. January 22.

1177. W. Sinclair. A composition suitable for lining casks and like vessels, and for making screw stoppers for bottles. January 22.

1234. R. Mannesmann. Regenerative gas furnaces. January 23.

1235. R. Mannesmann. Compressing air or other gaseous fluid, and apparatus therefor. January 23.

1236. R. Mannesmann. Compressing air or other gaseous fluid, and apparatus therefor. January 23.

1239. D. Bethmont. Apparatus for the instantaneous production of vapour from water or other liquids. January 23.

1242. R. A. Kemp. Apparatus for containing material for absorbing moisture or humidity. January 23.

1307. A. Capillary. Apparatus for filtering liquids. January 24.

1383. R. Johnson. Rotary apparatus for blowing, exhausting, or pumping air, water, and other gases or liquids. January 27.

1476. A. J. Boulton.—From G. Schweickhart, United States. Improvements in tyères. January 28.

1515. H. E. Newton.—From W. J. Maedonagh, R. Rock, W. Wood, C. S. Wilkinson, J. Plummer, and R. B. Wilkinson, New S. Wales. Appliance for injecting liquid and gaseous fuel into furnaces. January 28.

1823. P. S. Conron. Improvements in thermometers for converters, mash tuns, and other vessels used in brewing and other industrial operations. February 3.

1877. A. Riedler. Apparatus for compressing air or gases. Complete Specification. February 4.

1910. J. F. H. Gronwald. Improved sterilising apparatus. Complete Specification. February 5.

1948. H. W. Deacon and V. C. Driffield. Apparatus for separating liquids from solids. February 5.

2055. B. J. B. Mills.—From A. Seigle, Gonjon, France. Apparatus for heating liquid and gaseous bodies. February 7.

2070. C. A. Sahlström and A. F. Hill. Furnaces for burning liquid fuel. February 8.

2139. O. Bellot. Means for regulating the generation of heat in ovens, furnaces, and the like. Complete Specification. February 10.

2207. W. Scollay. Apparatus with means for regulating the admission of air and of gas to Bunsen burners. February 11.

2218. R. Marshall and E. FitzGerald. Surface condensers. February 11.

2343. T. Wrigley. Mechanical stokers. February 13.



2427. A. Fryer. Treatment of solutions or liquids for the purpose of concentration or evaporation, and apparatus therefor. February 14.

2502. W. R. Renshaw. Apparatus for heating, cooling, and condensing fluids. February 15.

#### COMPLETE SPECIFICATIONS ACCEPTED.\*

1889.

3065. W. S. Squire. Machinery for separation of solid matters from liquids, and for washing out and exhausting the said matters. February 5.

3753. O. M. Row. Apparatus for condensing steam and vapours, and for heating or cooling fluids. February 12.

4354. E. B. Parnell. Calcining furnaces. January 22.

5326. T. F. Shillington and J. A. Hanna. Appliances for heating and circulating liquids by steam. February 5.

5373. P. W. Turner. Wooden vessels for corrosive substances. February 5.

6242. R. Haig. Mechanical retorts for treatment of animal, vegetable, and mineral matters. February 19.

8596. J. G. Chapman. See Class XVI.

10,361. H. H. Lake.—From La Société Anonyme des Parfums Naturels de Caunes. Process and apparatus for the lixiviation of various materials. January 29.

13,598. H. McPhail, E. Simpson, and C. H. Simpson. Method and apparatus for generating steam and evaporating various liquids. February 5.

16,478. A. de Kunwald. Distillatory apparatus. February 19.

19,002. J. E. Searles, jun. Method and apparatus for extracting the liquid or soluble constituents from disintegrated vegetable materials. February 5.

1890.

206. J. F. H. Gronwald and E. H. C. Oehlmann. Sterilising apparatus. February 12.

266. A. Brock and H. A. Romero. An improved air pump. February 12.

## H.—FUEL, GAS, AND LIGHT.

### APPLICATIONS.

788. W. W. Horn.—From C. W. Jarrell, United States. An improved apparatus for washing gas. January 15.

794. H. S. Maxim and G. S. Sedgwick. Improvements relating to the carburetting or enrichment of coal and other gas, and to apparatus therefor. January 15.

910. E. Freund. Improvements in the process of and apparatus for distilling, roasting, or carbonising coal for the manufacture of gas, applicable also to the like treatment of other materials. January 17.

1001. W. H. Nevill. Improvements in the manufacture of artificial fuel, and the treatment of coal, coke, and other similar products with a view to their better combustion. January 20.

1022. T. Walrond-Smith. Improvements in the manufacture of water gases. January 20.

1237. R. Mannesmann. Improvements in the manufacture of illuminating gas, and apparatus for that purpose. January 23.

1238. R. Mannesmann. Improvements in the production of water and illuminating gas, and apparatus for that purpose. January 23.

1283. O. Imray.—From R. Mannesmann and M. Mannesmann, Germany. Improvements in the production of coke, and apparatus for that purpose. January 24.

1285. O. Imray.—From R. Mannesmann and M. Mannesmann. Improvements in gas-producers. January 24.

1349. W. L. Wise.—From E. Solvay and L. Semet, Belgium. Improved method or process for the gasification of fuels, and apparatus therefor. January 25.

1556. W. C. Owston. Coal-saving composition. January 29.

1698. C. Heyer. Improvements in carburetting gases, and in the purification of hydrocarbons for that purpose. Complete Specification. January 31.

1801. P. Scharf. Improvements in heating by means of gas. February 3.

2283. W. Hughes and E. Grippiech. Improvements in or appertaining to apparatus for the manufacture of oil gas. February 12.

2341. F. Scudder and H. G. Colman. Improvements in the manufacture or treatment of gases containing carbonic oxide, such as water-gas and producer-gas, to render them odorons. February 13.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

2093. J. F. M. Pollock. Machinery for manufacturing blocks of compressed coal dust and pitch or other agglomerate; also clay, cement, &c. February 12.

2561. J. Pring. Plant for the manufacture of patent fuel. February 19.

3498. W. R. Hutton. Making briquettes for fuel. January 22.

4288. A. Beeson. Manufacture of fuel. January 29.

4644. F. Siemens. Regenerative gas furnaces and producers. January 22.

4726. R. A. Gilson. Utilising waste heat of gas flame for warming purposes generally, and for increasing luminosity of the flame. January 29.

4869. D. Irving and A. Cochey. Apparatus for extracting ammonia, sulphur, and other impurities from coal gas. January 22.

4880. J. Broad and G. C. Fowler. Improved nightlights, and method of manufacturing same. January 29.

4955. Brin's Oxygen Co. and K. S. Murray. Means for producing oxygen and the like. February 5.

5145. J. H. R. Dinsmore. Manufacture of gas for illuminating and heating, and apparatus therefor. February 5.

5635. G. Bamberg. Automatically maintaining the internal temperature of retorts at full red heat, and preventing the use of temperature beyond full red heat. January 29.

5697. B. E. Kelly, J. H. Spencer, and H. Spencer. Fuel blocks. February 5.

5974. D. S. Hawks.—From E. L. Brady. Anti-corrosive lubricant and lubricating vapour for carbon disulphide and other vapour motors. February 12.

6131. S. Fox and E. Blass. Manufacture of water-gas, and apparatus therefor. February 12.

6325. R. B. Main, A. P. Main, and H. Darwin. Refractory lumps for incandescent gas fires. February 19.

16,489. F. Morris and L. van Vestrant. Apparatus for charging inclined gas retorts. January 22.

20,980. H. E. Newton.—From The Fuel, Gas, and Light Improvement Company, Incorporated. Process and apparatus for the manufacture of gas. February 5.

20,981. H. E. Newton.—From The Fuel, Gas, and Light Improvement Company, Incorporated. Apparatus for the manufacture of gas. February 5.

\* See previous page.

### III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

#### APPLICATIONS.

1107. M. Lange. A method of separating the isomers contained in crude nitrotoluol. January 27.

2242. F. Ilawaty. Improvements in the process of preparing benzol and its homologues naphthalene and anthracene from petroleum, petroleum scales, tar, tar oils, paraffin, vaselin, ozokerit, and the like, and from natural illuminating gas. February 11.

#### COMPLETE SPECIFICATION ACCEPTED.

1889.

4597. W. Young and G. T. Beilby. Distillation of mineral oils, and apparatus therefor. February 5.

### IV.—COLOURING MATTERS AND DYES.

#### APPLICATIONS.

724. J. J. Hummel. See Class XIII.

735. A. Bang.—From Messrs. Dahl and Co., Prussia. The production of dioxynaphthalene-mono-sulpho acid and diazo-dyestuffs from the same. January 15.

1579. J. Imray. — From La Société Anonyme des Matières Colorantes et Produits Chimiques de St. Denis, A. F. Poirrier, and D. A. Rosenstiehl, France. Manufacture of azo-amines by the reduction of azo-colouring matters derived from nitramines. January 29.

1688. T. R. Shillito.—From J. R. Geigy and Co., Switzerland. Producing brown dyes or colouring matters. January 31.

1699. O. Imray.—From The Farlowwerke vorm. Meister, Lucius, und Brüning, Germany. Improved manufacture of colouring matter of the induline series, soluble in water. January 31.

1771. T. R. Shillito.—From J. R. Geigy and Co., Switzerland. Production of yellow dye or colouring matter. February 1.

1808. O. Imray.—From the Society of Chemical Industry in Basle, Switzerland. Production of yellow basic colouring matter. February 3.

1811. R. Holliday. Improvements in the manufacture of colouring matters. February 3.

1812. R. Holliday. Improvements in the manufacture of colouring matters, and in dyeing or printing wool and other fibres therewith. February 3.

1828. B. Willecox.—From The Farbenfabriken vorm. F. Bayer and Co., Germany. Improvements in the manufacture of azo-colouring matters for dyeing and printing. February 3.

1874. J. Imray.—From La Société Anonyme des Matières Colorantes et Produits Chimiques de St. Denis, and Dr. Chapuis, France. Production of soluble blue colouring matters of the induline class. February 4.

1962. A. Bang.—From Messrs. Dahl and Co., Prussia. Improvements in the production of yellow dyestuffs. February 6.

2011. W. S. Conrad and H. T. Lilley. An improved copying ink. Complete Specification. February 7.

2499. H. H. Lake.—From K. Ochler, Germany. Improvement in the manufacture of soluble blue cotton dyestuff or colouring matter. February 15.

### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

1046. B. Willecox.—From The Farbenfabriken vorm. F. Bayer and Co. Production of unsymmetrical acid green. January 22.

2360. E. Brasier and J. H. Knowles. A new extract, and its utilisation as a dye, ink, stain, tanning liquor, and the like. January 29.

3048. I. Levinstein. Manufacture of red colouring matters for dyeing and printing. January 22.

3441. C. Dreyfus. Production of colouring matter from phenol-phtalein. January 22.

5549. O. Imray.—From The Farbenfabriken vorm. Meister Lucius und Brüning. Manufacture of green and bluish-green colouring matters. January 29.

5785. J. Y. Johnson.—From F. von Heyden. Production of oxymetho-oxybenzoic acids. February 12.

### V.—TEXTILES, COTTON, WOOL, SILK, Etc.

#### APPLICATIONS.

682. O. Imray.—From the Celluloid Manufacturing Co., United States. Waterproof collar, cuff, and bosom, composed partly of vegetable parchment. Complete Specification. January 14.

683. O. Imray.—From the Celluloid Manufacturing Co., United States. Collar, cuff, and bosom, with waterproof body, and coverings having a coating of pyroxyline varnish. Complete Specification. January 14.

725. G. Broadbridge.—Partly communicated by M. J. de Sant Anna, Lagos. Improved bast or piassava substitute, and manufacture of same. January 15.

1461. J. Smith, I. Smith, and J. Smith. Improvements in the method of and apparatus for treating or scouring and washing wool and other fibrous substances. January 28.

1462. J. B. Whiteley and E. Whiteley. A new or improved method and means for drying or extracting moisture from wool, cotton, and other like fibres or fibrous materials. January 28.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

4991. J. Yates and G. Kay. Open soapers for soaping and dunging fabrics. January 29.

20,936. L. Pinagel. Process and apparatus for freeing wool from grease. February 12.

### VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

#### APPLICATIONS.

703. R. Haddan.—From B. Leplat, France. Apparatus for use in treating and dyeing cotton slivers, rovings, or caps, and other textile materials. Complete Specification. January 14.

903. E. Charles. Improvements in vats employed in dyeing. January 17.

1062. S. Knowles and J. Knowles. Improvements in the production of azo-colours upon cotton or other vegetable fibre in the piece, and in apparatus therefor. January 21.

1157. G. Young and F. Pearn. Improvements in mechanism or apparatus for dyeing and bleaching cotton, wool, silk and other fibrous materials in the raw and manufactured, or partly manufactured state. January 22.

1607. J. Smith, I. Smith, and J. Smith. Improvements in the method of and apparatus for mordanting and dyeing wool, cotton, and other fibre, and also for extracting or separating animal from vegetable fibre. January 30.

1702. G. H. Craven. Improvements in dyeing machines. Complete Specification. Filed January 31. Date applied for July 22, 1889, being date of application in United States.

1812. R. Holliday. See Class IV.

1813. R. Holliday. Improvements in dyeing textile fibres. February 3.

2195. I. Braithwaite. Improvements in or appertaining to dyeing with indigo. February 11.

2338. J. Boardman and J. Higginson. An improved means of treating or dyeing hats, caps and other head coverings, whereby they change colour with alteration in atmospheric conditions. February 13.

2381. J. Königswerther. See Class XIV.

2411. T. Ingham. Improvements in dyeing cotton. February 14.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

4602. H. A. Seegall. See Class XIII.

4991. J. Yates and G. Kay. See Class V.

5995. D. G. FitzGerald. Bleaching by the agency of chlorine and certain of its compounds. February 19.

1890.

703. R. Haddan.—From B. Leplat, France. Apparatus for use in treating and dyeing cotton slivers, rovings, or caps, and other textile materials. February 19.

### VII.—ACIDS, ALKALIS, AND SALTS.

#### APPLICATIONS.

670. A. Domeier.—From E. K. Mitting, United States. Improvements in purifying salt. Complete Specification. January 14.

779. H. H. Leigh.—From H. P. Lorenzen, H. Stahr, and R. Heinz, Germany. Obtaining alumina and phosphoric acid from phosphates of alumina. January 15.

858. C. Roth. Improvements in the manufacture or separation of ammonium nitrate, and sulphate, or chloride of sodium, and of potassium. January 16.

933. W. Feld. Improvements in the manufacture of pure caustic soda from sodium sulphate, sulphur being recovered as sulphuretted hydrogen. January 18.

984. H. Grimsnaw. An improved compound for the generation of carbon dioxide, and suitable for use in gazogenes, fire extinguishers, and similar apparatus. January 20.

1015. F. Ellershausen. Improvements in the manufacture of soda and potash. January 20.

1016. F. Ellershausen. Improvements in the manufacture of soda and potash. January 20.

1117. J. Massignon and E. Watel. Improvements in the manufacture of chromates, bichromates, and other chromium compounds. January 21.

1259. G. Dames. A machine or appliance for use in manufacturing for making solution or wash of dried Fuller's earth, alkalis, or other substances, and at the same time screening and separating therefrom all hard and insoluble substances. January 24.

1300. H. H. Leigh.—From T. de Brochowski, France. Improvements in the manufacture of anhydrous barium oxides. Complete Specification. January 24.

1524. G. Wischin. Improvements in the method of vaporising solutions containing nitrates of manganese, and in the means used for decomposing the said nitrates. January 28.

1584. F. Ellershausen. Improvements in the manufacture of soda and potash. January 29.

1653. G. Kennedy. Improvements in and connected with saturators for sulphate of ammonia plant. Complete Specification. January 30.

1848. A. R. Davis. Improvements in apparatus for the distillation of liquors containing ammonia. February 4.

1914. F. M. Spence and D. D. Spence. Improvements in the manufacture of alum. February 5.

1964. W. O. Kibble. Improved apparatus for generation of sulphuretted hydrogen and other gases. February 6.

2296. J. C. Richardson and T. J. Holland. See Class XI.

2432. A. M. Chance and H. W. Crowther. Improvements in the manufacture of nitrate of ammonia. February 14.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

4095. F. Mühlenbrüch. Producing monocarbonate of calcium. January 22.

4417. W. L. Wise.—From E. Solvay and Co. Manufacture of anhydrous chloride of magnesium, and of chlorine therefrom. January 29.

4701. S. Daw. Treating waste or spent sulphuric acid pickle from the manufacture of tin, terne, and black plate. February 12.

5225. R. Dormer. Obtaining chlorine. February 12.

5340. G. A. H. Gabet. Manufacture of ammoniacal calcic phosphate. January 29.

5341. G. A. H. Gabet. Manufacture of potassic and sodic alkalis. February 12.

5779. J. W. Kynaston and J. Sutherland. Treating recovered ash from paper mills. February 12.

6668. M. Netto. Manufacture of artificial cryolite. February 19.

17,272. P. de Wilde and A. Reyehler. Processes for the production of chlorine and for obtaining concentrated gaseous hydrochloric acid for that purpose. January 29.

20,135. J. E. J. Johnson. Manufacture of acetic acid and soluble acetates. January 22.

20,722. J. C. Fell.—From The American Ammonia Manufacturing Company. Method and apparatus for obtaining ammonia from atmospheric nitrogen and the hydrogen of decomposed steam. February 12.

20,908. M. B. Mason, S. D. Warren, and F. Warren. Method and apparatus for recovering chemicals from solutions or when mixed with substances which are volatilisable or destructible by heat, especially adapted for recovering soda from waste liquors of pulp digesters. February 12.

### VIII.—GLASS, POTTERY, AND EARTHENWARE.

#### APPLICATIONS.

612. W. C. Bennett. Improvements in the manufacture of glass bottles, and apparatus therefor. January 13.

738. C. F. Bailey. Improvements in the method or means for supporting pottery-ware whilst being fired. January 15.

830. H. L. Phillips. Improvements in or connected with the manufacture of glass bottles, jars, and similar vessels, and in apparatus employed therefor. January 16.

927. W. W. Pilkington. Improvements in plate and rolled plate glass annealing kilns and machinery. January 18.

1011. H. Worthington. Improvements in garden tiles. January 20.

1013. J. P. Guy. Improvements in machines for pressing and making hollow earthenware articles. January 20.

1215. H. Walker. Improvements in annealing and other heating furnaces. January 23.

1588. J. Bates and R. Stanley. Improvements in the manufacture of burnt-clay or earthenware goods. January 29.

1666. T. Bellamy. An improvement in glass melting pots. January 31.

1803. L. Appert. New or improved method of moulding glass, and apparatus therefor. Filed, February 3. Date applied for, August 29, 1889, being date of application in France.

1815. A. Bevington. Improvements in decorating china, earthenware, and the like. February 3.

1820. H. Gurney. An improved method of burning bricks and tiles, and an improved kiln therefor. Complete Specification. February 3.

2487. J. McMillan. Apparatus for making bottles or other articles having a narrow orifice in glass, crystal, or similar molten metals, with a certain modification of same for making similar articles in clay, stone, flint, or china ware. February 15.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

5048. D. Rylands. Moulds for forming bottles by the pressing and blowing process. January 29.

5205. H. L. Doulton and S. H. Leech. Manufacture of blocks of terra-cotta, &c., and apparatus therefor. February 12.

5691. J. A. Edwards. Method and apparatus for supporting earthenware and porcelain whilst being fired. February 5.

5992. J. Rhodes. Manufacture of china and earthenware saucers. February 12.

7387. G. Falin. Improvement of fireclay goods by adding sugar to the compound known as silica and alumina. February 5.

13,051. J. Peirce and T. F. Luther. Enamelled metallic tessellated plates for hearths and floors. February 5.

19,744. G. A. Marsden. Manufacture of tiles, bricks, &c. having ornamented surfaces. January 22.

#### IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

##### APPLICATIONS.

617. F. T. P. Wells. An improved treatment of wood for the purpose of rendering it practically indestructible. January 13.

1097. H. H. Lake.—From J. F. Gesner, United States. Manufacture of an artificial solid material, and apparatus therefor. Complete Specification. January 21.

1176. W. Sinclair. A compound for cementing iron rails to stone kerbing, joints of stone, drain pipes, ships' seams, and the like. January 22.

1248. W. W. Horn.—From J. S. Goodwin, United States. An improved building block. January 23.

1351. J. E. H. Gordon. Improvements in pavements. January 25.

1356. A. J. Boulton.—From W. Deissner, Germany. Improvements in the manufacture of rough, glazed, and coloured facing stones. Complete Specification. January 25.

1525. I. Button, E. Peters, and J. W. Goodsell. Improvements in kilns or ovens for burning bricks, earthenware, limestone, cement, and other substances. Complete Specification. January 28.

1654. M. B. Parrington. Improvements in the manufacture of lime, and in apparatus therefor. January 30.

1934. C. Eifield. An improved silicious paving and building stone. February 5.

2371. C. H. Edwards. Improvements in or appertaining to the manufacture of certain kinds of bricks and fireclay substitutes. February 13.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

2093. J. F. M. Pollock. See Class II., February 12.

4946. R. R. Clunis. Improvements in bricks. January 22.

5157. J. W. H. James. Manufacture of cement. February 5.

18,487. H. H. Lake.—From G. M. Fiske. Improvements relating to the construction of brickwork. February 12.

18,493. H. H. Lake.—From G. M. Fiske. Improvements in bricks. February 12.

18,911. P. de Kristoffovitch. Manufacture of artificial granite. January 29.

19,744. G. A. Marsden. See Class VIII.

#### X.—METALLURGY, MINING, ETC.

##### APPLICATIONS.

840. R. Manuesmann. Improved means of mixing molten metal in furnaces or converters. January 16.

859. W. White. Improvements in or connected with the manufacture or production of aluminium. January 16.

877. D. Elliott. Improvements in furnaces for heating iron or steel. January 17.

1053. H. Grimshaw. Improvements in and connected with the recovery of zinc from galvanised iron and steel. January 21.

1215. H. Walker. See Class VIII.

1227. E. C. Miller.—From The Magnolia Anti-Friction Metal Co., United States. Method of preparing or treating metals for the manufacture of compositions or alloys for anti-friction purposes. January 23.

1355. A. E. Haswell and A. G. Haswell. Improvements in the coating of iron, steel, nickel, zinc, or tin, for the prevention of corrosion. January 25.

1369. G. C. Fricker. Improvements in cleaning and annealing iron, and apparatus therefor. January 25.

1427. M. Netto. Improvements in the manufacture of metallic compounds or alloys. January 27.

1443. J. T. King.—From C. Adams, United States. Improvements in the reduction of metallic ores, and in apparatus therefor. January 28.

1522. T. H. Johns. Improved construction or arrangement of apparatus for coating hoop iron with tin or terne metal. January 28.

1624. A. H. Holdich. An improved form of stopper or valve for controlling the flow of steel from the ladle or reservoir used in steel works. January 30.

1625. F. K. S. Lowndes and J. C. Kaller. A machine for the extraction of gold, silver, and other metals from quartz and ailings, blanketings, &c., by a continuous process of chlorination and amalgamation. January 30.

1634. G. D. Burton. Improvements in the manufacture of forgings. Complete Specification. January 30.

1641. F. W. Dähne. Improvements in calcining and refining copper ore, copper regulus, and copper precipitate, and in the construction of furnaces therefor. Complete Specification. January 30.

1647. — Hopkinson. Hardening metallic alloys. January 30.

1682. R. Heathfield. Improvements in apparatus or machinery for coating metal sheets with metals or alloys. Complete Specification. January 31.

1691. D. Ormerod. Improvements in metal alloy for use in pipe-jointing, also applicable for moulding and casting purposes. January 31.

1733. J. F. Spencer. Improvements in machinery or apparatus for charging pig iron and scrap metal into open-hearth furnaces. February 1.

1754. G. Höper. Improved method of and apparatus for the manufacture of dense-metal castings, and of sheet metal and wire therefrom. February 1.

1794. C. Pfeiffer. An improved amalgamator for extraction of gold and silver from the ore by the action of mercury. February 3.

1806. M. R. Conley and J. H. Lancaster. An improved process for making steel, and apparatus therefor. February 3.

1842. E. L. Mayer and J. G. Lorrain. Improvements in or connected with the treatment and recovery of float-gold. February 4.

1888. R. C. Totten. Improvements in and relating to castings, such as dies, ingots, and other castings. Complete Specification. February 4.

1913. W. P. Thompson.—From E. Carez, Belgium. Improvements in or relating to the treatment or utilisation of scrap tin orterne plates, or the like. February 5.

1987. S. Wohle. A process for electro-plating metals with aluminium, or its alloy with nickel. February 6.

2002. G. Shenton.—From M. J. Berg, France. Improvements in the manufacture or production of aluminium. February 6.

2057. A. J. Campion. Improvements in processes for smelting and treating tin ores, slags, slimes, and furnace debris containing tin and other products. February 7.

2226. N. Lebedeff. Improvements in extracting iron, copper, nickel, or other metals or alloys from combinations thereof with other substances. February 11.

2248. F. W. Harbord. Improvements in the utilisation of tin-plate scrap. February 12.

2312. J. Teague. An improved process of extracting tin and other metals associated therewith from tin slags and other tin products or refuse. February 12.

2390. J. Buchanan, jun. Improvements in the method of precipitating gold or silver in the process of its extraction. February 14.

2477. T. Teague. An improved process for the extraction of tin from slags or tin debris. February 15.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1888.

18,211. G. E. Vaughan.—From L. Weiller. Manufacture of metallic alloys or compounds. January 22.

1889.

2350. T. Bell. Method of lining barrels, &c., to resist acids, for use in extracting metals by chlorination, &c. February 12.

2377. A. C. A. Holzapfel. Machine for flanging and shaping steel, iron, and other metal plates. February 5.

2937. E. B. Parnell. Treatment of ores for recovery of metals therefrom, and apparatus therefor. January 22.

4003. E. Lones, R. Davies, J. Davies, and B. A. Webb. Coating metal plates with tin and other metals, and apparatus therefor. January 22.

4169. J. L. E. Daniel. Manufacture of aluminium, and apparatus therefor. February 19.

4537. A. Hodgkinson, jun. Treatment of the calcined products of ironstone mines and shale, and manufacture of same into blocks for the extraction of iron therefrom. February 5.

4719. T. R. Jordan. Apparatus for extraction of gold and silver from their ores. January 29.

4882. P. C. Gilchrist. Manufacture of copper. February 5.

5037. D. Owen. Apparatus for the purification of molten tin or its alloys. January 29.

5078. A. E. Barthel. Production of fixed or detachable deposits of homogeneous iron in any form from iron ores, or iron or steel. February 5.

5195. J. Lones, C. Vernon, E. Holden, and R. Bennett. Manufacture of buffer iron or steel. January 22.

5914. E. Green. Production of aluminium. January 29.

6026. F. W. Rose.—From T. K. Rose and D. Dennes. Method and apparatus for treating ores. February 19.

7311. R. Heathfield. Apparatus used in coating metal sheets with metal or alloys. January 22.

13,659. W. L. Wise.—From M. Kramer. Hardening steel tubes internally. February 5.

13,718. A. M. Clark.—From La Société en participation Bourbonze et Cie. The production of a new metal or alloy. February 12.

16,649. G. G. Mullins. Treating copper and its alloys. January 29.

19,890. H. Aiken. Casting ingots, blooms, billets, slabs, or the like, and apparatus therefor. February 5.

19,944. J. A. H. T. Ranft. Process and apparatus for the continuous extraction of gold and silver by the agency of chlorine. February 12.

20,180. R. F. Neuninger. Combined smelting and reverberatory furnace and apparatus for catching the dust and condensing the fumes from the furnace. January 29.

#### XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

##### APPLICATIONS.

761. O. Dahl. Process of generating electricity by gas batteries. Date applied for June 17, 1889, being date of application in United States. Complete Specification.

907. W. A. Thoms. Improvements in the electro-deposition of metallic alloys. January 17.

1094. H. H. Lake.—From T. Ewing, United States. Improvements relating to secondary or storage batteries. Complete Specification. January 21.

1110. C. A. Hitchcock.—From W. Barnley, United States. An improvement in galvanic batteries. Complete Specification. January 21.

1246. D. G. FitzGerald and A. C. Falconer. Improvements in means for the electrolytic generation of chlorine and compounds of chlorine with earthy bases, more especially intended for bleaching purposes. January 23.

1281. V. H. Ernst. Improvements in secondary batteries. Filed January 24. Date applied for, September 28, 1889, being date of application in United States. Complete Specification.

1306. C. Smith.—From W. L. F. Hellesen, Denmark. Improvements in galvanic dry elements. January 24.

1347. T. Coad. An improved fluid for primary batteries. January 25.

1495. H. H. Lake.—From G. A. Johnson, United States. Improvements relating to storage batteries or accumulators. Complete Specification. January 28.

1784. T. A. Garrett. Improvements in and connected with the production of ozone by means of electricity. February 3.

1878. J. B. Williams. Improvements in electric insulating compounds. Complete Specification. February 4.

1897. E. Fabrig. An improved composition applicable for electrical insulating and other purposes, and the process for manufacturing the said composition. January 4.

2091. W. P. Thompson.—From the Gibson Electric Co. of Europe, France. Improvements in secondary batteries and plates or elements therefor. February 8.

2113. G. A. Schoth. Improvements in the manufacture of plates of electrodes as used in secondary batteries or accumulators. February 10.

2273. G. R. Postlethwaite. Improvements in voltaic cells or primary batteries. February 12.

2296. J. C. Richardson and T. J. Holland. Improvements in the electrolytic production of caustic soda, caustic potash, and other products from their salts. February 12.

2297. J. C. Richardson and T. J. Holland. Improvements in apparatus to be used for electrolytic purposes. February 12.

2302. H. Weymerseh and R. McKenzie. Improvements in electric batteries working with two liquids or electrolytes. February 12.

2347. Sir C. S. Forbes, Bart. Improvements in primary batteries. February 13.

2439. F. S. Roberts. Improvements in or connected with electrical accumulators or storage batteries. February 14.

2606. E. C. F. Verschave and A. Baron. Improvements relating to electric batteries. February 15.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

813. W. Diehl. Separation of aluminium and its alloys from its fluorides by electric or by chemical metallurgical process. January 22.

2472. W. L. Wise.—From the Gibson Electric Co. of Europe. Secondary battery plates. February 19.

2544. A. Douglass and F. Smith. Improvements in the form and manufacture of secondary battery frames and plates. January 22.

3533. C. D. Abel.—From Siemens and Halske. Extraction of copper and zinc from their ores, as also the precious metals contained therein, by electrolysis, and apparatus therefor. January 22.

7719. P. Schoop. Electric or galvanic elements or cells. February 19.

11,661. C. Huelser.—From H. Mestern. Thermo-electric and furnace batteries. January 22.

14,677. R. R. Moffatt. The utilisation of electromagnetism for the separation of metals, and apparatus therefor. January 29.

19,892. H. E. Fowler. Apparatus for welding metal by means of electricity. January 22.

20,911. A. E. Woolf. Plates for storage batteries. February 12.

1890.

302. H. H. Lake.—From P. H. Alexander. Secondary batteries and plates therefor. February 12.

303. H. H. Lake.—From P. H. Alexander. Secondary batteries. February 12.

#### XII.—FATS, OILS, AND SOAP MANUFACTURE.

##### APPLICATIONS.

656. J. Templeman. Improvements in the manufacture of soaps and saponaceous compounds. January 14.

983. H. Grimshaw. Improvements in soap and soap powder for use with hard and sea waters. January 20.

1047. E. Ireland. An improvement in soap called "Aurora herbal cosmetic soap." January 21.

1069. A. F. St. George. Decolourising and oxidising or solidifying drying oils for manufacturing purposes. January 21.

1109. H. Noerdlinger. Treatment of press and extraction residues of oil manufacture for obtaining valuable products therefrom. January 21.

1126. C. W. Hazlehurst and S. Pope. Improvements in the treatment or utilisation of salt obtained from spent soap lyes in the recovery of glycerin therefrom. January 22.

1343. T. H. Gray and S. S. Bromhead. Improvements in refining linsced and other oils. January 25.

1650. M. Perls. Improvements in the manufacture of soap. January 30.

1652. B. D. Barnett. Improvements in the process of cleaning and extracting oil and grease from cotton waste and other fibrous materials. January 30.

1659. P. Plisson. Improvements in the manufacture of lubricating grease, and in the apparatus employed therefor. Complete Specification. January 30.

2104. P. Wild. Process and apparatus for melting margarine tallow by means of hot air. February 8.

2264. J. Green and G. Macdonald. An improved lubricant or grease. February 12.

2336. H. Harford. An improved method of making lubricating grease. February 13.

2446. A. des Cressonnières and E. des Cressonnières. Improved process and apparatus for the manufacture of kneaded or agglomerated soaps. February 14.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

4367. W. G. Reid. Treatment of used soapy liquors. January 29.

5622. J. Pearson. Treating cotton-seed oil mangle to obtain soap. February 5.

20,237. W. Rödiger. Manufacture of soap. January 22.

#### XIII.—PAINTS, PIGMENTS, VARNISHES, AND RESINS.

##### APPLICATIONS.

724. J. J. Hummel. New or improved red dye or pigment, and process for making and applying the same. January 15.

764. W. H. Barnes. Improvements in compositions to be used as paints. January 15.

812. W. H. Ness and W. C. Folkard. An improved preservative composition or solution for coating surfaces of articles or structures where great adhesiveness is required. January 16.

893. A. N. Ford. An improved composition for insulating and waterproofing purposes. January 17.

985. H. Grimshaw. A new or improved white pigment. January 20.

1178. W. Sinclair. Waterproofing and preserving composition for stone, bricks, plaster, and cement surfaces. January 22.

1489. J. J. E. Mayall. Improvements connected with colouring photographic impressions, and in the mixing and preparing the colours, and in a varnish or enamel for same. January 28.

1684. A. J. Boulton.—From N. A. Bibikof, New Mexico. Improvements in paints. Complete Specification. January 31.

1821. C. B. Phillips and H. R. Lewis. Improvements relating to the application of metallic copper to the hulls of iron or steel and other sea-going ships or vessels and other submerged structures to prevent fouling thereof, and to the manufacture of compositions or pigments to be used for this purpose. February 3.

2308. H. Beck.—From E. Schaal, Germany. Process for the production of resin acid esters applicable as substitutes for the lacquer-resins or gums generally used in the manufacture of lacquers and varnishes. Complete Specification. February 12.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

4179. S. Kenyon. Preparation of a substance applicable as a substitute for india-rubber, and for waterproofing or dressing fabrics, &c. January 22.

4602. H. A. Seegall. Production of chrome preparations. February 19.

5344. F. Greening. Production of a substitute for ivory, horn, whalebone, india-rubber, &c., useable as a coating or varnish for waterproofing, &c. February 12.

5349. J. Freemont. Manufacture of coloured sealing-wax. January 22.

20,010. J. H. Bath. A hoot-blackening powder. January 29.

## XIV.—TANNING, LEATHER, GLUE, AND SIZE.

## APPLICATIONS.

981. H. Grimshaw. Improvements in and connected with the recovery of tannin from waste leather. January 20.

1482. W. P. Thompson.—From F. G. Hermann, France. Improvements in apparatus for removing the hair from skins. Complete Specification. January 28.

1576. S. Wohle. Improvements in the treatment of leather to render it impervious, strong, and durable. January 29.

2366. A. Leturque. Improved indicating apparatus for use in connexion with tanning and similar vats. February 13.

2381. J. Koenigwerther. A new process for the dyeing of skins, hairy skins, or fabrics. February 13.

## XV.—AGRICULTURE AND MANURES.

## APPLICATIONS.

575. M. C. Ginster. Improvements in the process of manufacturing a manure or fertiliser from organic matter. January 13.

2238. C. W. Doughty. An improvement in the means and proportions of materials used for the deodorisation, evaporation, and, to a certain extent, decomposition of human excrement and its transformation into a nearly inodorous and valuable fertiliser. Complete Specification. February 11.

2265. J. Pipe and H. D. Andross. An improved compound for treating vines and other plants for the prevention and destruction of phylloxera, mildew, and the like, and the method of producing the same. February 12.

2450. F. Dorr. Improvements in the process of treating cereals. Complete Specification. February 14.

## COMPLETE SPECIFICATION ACCEPTED.

1889.

5703. H. J. Kirkman. Treatment and utilisation, for agricultural and other purposes, of bisulphate of soda or nitre cake. February 12.

## XVI.—SUGARS, STARCHES, GUMS, ETC.

## APPLICATIONS.

1247. A. Fairgrieve. Improvements in the manufacture or production of raw or refined sugar, and apparatus to be used therein. January 23.

1282. C. D. Abel.—From Messrs. Pfeifer and Langen, Germany. Improved treatment of sugar crystals or grains for facilitating the purifying or washing thereof. January 24.

1536. E. Woolley and W. H. Evans. An improved adhesive medium. January 29.

1809. H. T. Brown, G. H. Morris, and E. R. Moritz. Manufacture of amyloins. February 3.

2197. W. P. Thompson.—From K. Korenar and F. Zwicker, Austria. A new or improved adhesive cement and process of manufacturing the same. February 11.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

3526. A. G. Salamon. Preparation of cane and beet sugar. February 12.

20,515. C. Steffen. Process for obtaining the entire quantity of crystallisable sugar contained in sugar mass. February 12.

## XVII.—BREWING, WINES, SPIRITS, ETC.

## APPLICATIONS.

667. W. P. Thompson.—From J. A. H. Hasbrouck, United States. New or improved process and apparatus for ageing liquors. January 14.

678. E. Edwards.—From F. Hofmeister, Germany. Improved method of and apparatus for obtaining accelerated fermentation and producing sparkling drinks. January 14.

792. G. Guignard and A. Hédonin. Improvements in and relating to the manufacture of alcohol, and in apparatus therefor. January 15.

980. H. Grimshaw. Improvements in the process of ageing or artificially maturing whisky and other spirituous liquors. January 20.

1573. A. Rock. Improvements in and relating to malt kilns. January 29.

1823. P. S. Conron. See Class I.

1921. C. Gibbs. A method of and apparatus for condensing steam in brewing coppers. February 5.

1996. J. F. Littleton. Improvements in rousing beer and mixing or blending wort or gyle, and in apparatus employed therein. February 6.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

3574. M. P. Halschek. Improved system of "pneumatic" malting. January 29.

4549. E. A. Barbet. Rectifying and distilling alcohol, and apparatus therefor. January 22.

5647. C. Schmitt. Purification of raw spirit to produce highly rectified spirits of wine, and apparatus therefor. February 5.

8596. J. G. Chapman. Apparatus for evaporating and concentrating saccharine and other solutions. February 12.

19,996. W. Spilker, H. Bendel, M. Hahn, and C. Löwe. Killing minute organisms, such as yeast cells, bacilli, and the like, contained in wines, alcoholic and other similar liquors. January 22.



# XVIII.—CHEMISTRY OF FOODS, SANITARY CHEMISTRY, AND DISINFECTANTS.

## APPLICATIONS.

### A.—Chemistry of Foods.

982. H. Grimshaw. Improvements in the method of producing infusions of tea and similar solutions free from tannin. January 20.

1326. A. Schmidt-Mülheim. Improvements in the method of and apparatus for the production and preservation of sterilised milk. Complete Specification. January 25.

1328. J. T. Appleberg. Improved apparatus for preserving and purifying milk. Complete Specification. January 25.

2060. H. A. Snelling. A new or improved method of treating and preparing hops, and blending the same for infusion together with tea, coffee, cocoa, and other substances for which such prepared hops are applicable. February 7.

2300. D. Browne. A new process for preserving eggs. February 12.

### B.—Sanitary Chemistry.

1170. C. A. Brown. Improvements in furnaces for burning tan and other refuse material. January 22.

1279. A. Moseley. Preparing and compressing quick lime or gypsum so that its use may be more generally adapted for the more rapid decomposition of human bodies. January 24.

1623. W. Warner. Improvements in apparatus employed in treating sewage. January 30.

1816. G. W. Ewens. Improvements in purifying the sewage of cities and towns, and foul waters discharged from breweries, tan yards, glue works, and other description of manufactory. February 3.

2238. C. W. Doughty. See Class XV.

### C.—Disinfectants.

1017. W. Dammann. An improved process for rendering tar oils soluble in water, and the production of disinfectants and other useful products therefrom. January 20.

1289. A. Arnott. See Class XIX.

1589. C. T. Kingzett. Improvements in the manufacture of disinfectants, antiseptics, deodorants, insecticides, and sanitary reagents. January 29.

2047. H. Oppenheim and G. H. Elliott. Candles for disinfecting and diffusing ozone in sick and close rooms. February 7.

## COMPLETE SPECIFICATIONS ACCEPTED.

### A.—Chemistry of Foods.

1889.

4715. J. F. Pensgood. Preparation of tomato vinegar. February 5.

4966. J. Gray and T. Clement, jun. Means of estimating the acidity in milk, cream, whey, and curds in the process of making butter or cheese. February 5.

20,614. W. Douglas and J. Donald. Preserving fish, birds, and other analogous articles of food. February 12.

### B.—Sanitary Chemistry.

1889.

6219. W. Astrop. Process for deodorising, disinfecting, drying, and pulverising the solid portions of sewage. February 19.

16,388. G. W. Bremner. Lumps for destroying sewage gas and noxious vapours. January 22.

1890.

206. J. F. H. Gronwald and E. H. C. Oehlmann. See Class I.

### C.—Disinfectants.

1889.

2537. A. J. Shilten. A new disinfectant and deodoriser. February 5.

6411. F. W. A. Hillé. Manufacture and use of disinfectants. February 12.

# XIX.—PAPER, PASTEBOARD, ETC.

## APPLICATIONS.

795. R. Stone. Improvements in the treatment and utilisation of peat, saw-dust, and other fibrous and vegetable materials and substances. January 15.

1087. T. Goodall. An improved arrangement of roasting apparatus for the incineration of black liquor and spent soda lyes used in the boiling of esparto grass or other fibrous matter. January 21.

1289. A. Arnott. A new disinfecting paper. January 24.

1514. F. C. Alkier. Improvements in extracting substances dissolved in the waste-water of paper manufacture. Complete Specification. January 28.

1651. M. Perls. Improvements in the manufacture of paper. January 30.

2375. H. Colley. Improvements in or relating to the manufacture of paper, and in apparatus therefor. February 13.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

4264. T. Phoenix and G. Kirk. Preparation of tissue transfer paper, and of printed transfers for transferring designs to pottery ware. January 22.

8509. F. Favier, jun. Machinery for refining and finishing half-stuff in paper-making; and hydrostatic balances for changing the contents of the pulp reservoirs. January 29.

# XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES, AND EXTRACTS.

## APPLICATIONS.

1380. R. J. Downes. A solution of iodine for medical purposes. January 27.

1422. A. A. Vale.—From The Chemische Fabriks-Aetien Gesellschaft, Germany. Process of manufacturing eumaron and its homologues. Complete Specification. January 27.

1583. S. Radlauer. Improvements in the preparation of ethyl chloral urethane. January 29.

1988. F. L. Johnstone. Improvements in apparatus for filtering and derisining perfumes, tinctures, essences, cordials, and other liquids. Complete Specification. February 6.

2129. G. S. Dobrashian. Compound syrup of Sarracenia purpurea. February 10.

## XXI.—PHOTOGRAPHIC PROCESSES AND MATERIALS.

### APPLICATIONS.

1489. J. J. E. Mayall. *See* Class XIII.

1956. E. Ireland and C. Ireland. A new or improved method of producing coloured photographs. February 5.

## XXII.—EXPLOSIVES, MATCHES, ETC.

### APPLICATIONS.

1884. P. Jensen.—From H. G. Underwood, United States. Improvements in solidified pyrotechnic powders, and in the method of converting the same into solid form for convenience in transportation, storage, and combustion. Complete Specification. February 4.

1995. W. P. Thompson.—From J. Hess, Belgium. Improvements in or relating to lucifer matches. February 6.

### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

4477. H. S. Maxim. Manufacture of explosive compounds. January 22.

4479. A. V. Newton.—From A. Nobel. Preparation of explosive compounds. February 19.

5031. P. de Montravel.—Partly by O. Mauvant. Manufacture of explosives. January 22.

5503. E. Liebert. Manufacture of explosive compounds. February 5.

6399. C. O. Lundholm and J. Sayers. Manufacture of explosives. February 19.

## XXIII.—ANALYTICAL CHEMISTRY.

### APPLICATIONS.

1082. G. C. Topp. Improvements to prevent hydrometers, saccharometers, and lactometers rotating, and to strengthen the same. January 21.

1209. J. H. R. Paterson. Raising and adjusting the position of the flame of a Bunsen's and Argand-Bunsen's, or other gas burner used for heating chemical or physical apparatus. January 23.

### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

6083. L. Mathieu and J. Morfaux. Process and apparatus for testing the presence of colouring matter in wines. February 12.

20,859. I. K. Lindström. Method and apparatus for determining the quantity of fat in milk. February 19.

# THE JOURNAL

OF THE

# Society of Chemical Industry:

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Notice is hereby given, for the information of members and advertisers, that the advertisement columns of this Journal have been contracted for by Messrs. EYRE and SPOTTISWOODE, the Society's printers and publishers, to whom all communications respecting them should be addressed.

The Secretary is instructed to negotiate for the purchase of copies of the Society's Journal for January 1883, and January, February, and April 1886. Members possessing odd copies of these numbers are particularly requested to communicate at once, stating price required, with Mr. Cresswell. The stock of all other numbers is at present sufficient for the Council's requirements.

Notice is hereby given that the Annual General Meeting will be held in Nottingham in the month of July next. Full particulars will appear in a subsequent issue.

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Bennett, Wm., Belmont House, Grimsby, chemical manure manufacturer.

Bloxam, W. Popplewell, Royal Naval College, Greenwich, S.E., chemist.

Burn-Murdoch, J. V., c/o Van Ryn Gold Mining Co., Boksburg, S.A.R., assayer.

Barringtons, Silas M., Snow Hill Buildings, E.C., manufacturing chemist.

Cassal, C. E., Brenne House, Wandsworth Common, S.W., public analyst.

Cooksey, Dr. Thos., 21, Brownwood Park, London, N., consulting chemist.

Dunn, John, Morgan Academy, Dundee, science teacher.

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Holloway, G. T., 17, Bridge Road West, Battersea, S.W., analytical chemist.

Jones, Wm., 61, Chadwick Road, Peckham, S.E., analytical chemist.

Keys, W. H., Hall End Chemical Works, West Bromwich, chemical manufacturer.

Lampray, R. H., 24, Bughley Road, Highgate Road, N.W.

Matheson, W. J., 178, Front Street, New York, U.S.A., chemical and dye merchant.

Mitchell, Jas. Vernon, 12, Woodstock Street, Oxford Street, London, W., chemist.

Pennoek, J. D., Solvay Process Co., Syracuse, N.Y., U.S.A., chemist.

Platts, Jno. C., 25, Harecourt Road, Crookesmoor, Sheffield, metallurgical chemist.

Roberts, Chas. F., 48, Vicar Lane, Bradford, Yorks, chemical merchant.

Robey, Chas. M., Collingwood, Ontario, Canada, tanning chemist.

Rogerson, W. J., 38, Southwark Street, London, S.E., wholesale druggist.

Sayers, Jos. J., Nobel's Explosives Co., Ardeer, Steveston, N.B., chemist.

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### Death.

Muter, Jas., at Rangoon, Burmah, September 19, 1889.

## London Section.

### CHEMICAL SOCIETY'S ROOMS, BURLINGTON HOUSE.

Chairman: David Howard.

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S. Hall.	C. R. Alder Wright.
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#### SESSION 1889-90.

1890.

April 21st (extra meeting):—

Dr. E. R. Moritz. "Note on the Expulsion of Ammoniacal Compounds from Sulphuric Acid used in Kjeldahl determinations."

Mr. Boverton Redwood. "The Petroleum Fields of India."

May 5th:—Mr. Wm. Webster. "The Electrical Treatment of Sewage."

June 2nd:—Dr. W. S. Squire. "The Artificial Production of Cold theoretically and practically considered."

July:—Annual General Meeting in Nottingham.

Meeting held Monday, 3rd March, 1890.

MR. DAVID HOWARD IN THE CHAIR.

### THE EXAMINATION OF TEXTILE FIBRES AND FABRICS.

BY H. SCHLICHTER, D. SC. NAT.

In the first place my paper does not pretend to be an account of the constitution of textile fibres. This branch of the subject has already been dealt with by Chevreul, Grothe, Cramer, Schulze, Hugo Müller, Mulder, Schützenberger, Bourgeois, and others; but this paper is intended to describe methods by which chemists may identify and distinguish various textile fibres, and estimate them quantitatively in textile fabrics. Moreover, the paper is limited to such textile fibres as find practical application in the European manufacture of textile fabrics, among which I do not include paper.

The technical examination of fibres and fabrics has hitherto been dealt with systematically only to a very limited degree, although there exist a number of more or less elaborate monographs, mostly based on microscopical studies, in which may be found at intervals valuable material in the shape of descriptions of reactions, properties, and measurements. Many of these, however, are merely colour tests, applicable only to undyed or raw fibres.<sup>(1)</sup>

Among the authors who may be regarded as having contributed to the literature of this subject are the following, to whose names we append the titles of their works:—

Schacht: Die Prüfung der im Handel vorkommenden Gewebe, 1853.

Schlesinger: Mikroskopische Untersuchungen der Gespinnstfasern, 1873.

Höhnel: Die Mikroskopie der technisch verwendeten Faserstoffe, 1887.

Kölliker: Mikroskopische Anatomie, 1850.

Kölliker: Gewebelehre, 1852.

Leydig: Histologie, 1857.

Nathusius: Wollhaar des Schafes, 1866.

(1) See note (25)

- Wiesner: Technische Mikroskopie, 1867.  
 Wiesner: Mikroskopische Untersuchungen, 1872.  
 Wiesner: Rohstoffe des Pflanzenreiches, 1873.  
 Bolley: Technologie der Spinnfasern.  
 Bohm: Wollkunde, 1873.  
 Grothe: Technologie der Gespinnstfasern, 1875.  
 Grothe: Die Gespinnstfasern aus dem Pflanzenreiche, 1879.  
 Vétillart: Études sur les fibres végétales textiles, 1876.  
 Bowman: The Structure of the Cotton Fibre, 1881.  
 Bowman: The Structure of the Wool Fibre, 1885.  
 Christy: New Commercial Plants and Drugs, No. 6, 1882.

In many of these works we find little or no trace of a systematic treatment of the subject, but here, as well as in other publications not cited, we find notices on the properties of various fibres. When we realise the practical importance of the textile industries of this and other European countries, it is very difficult to understand why up to this time no *general scheme* of analysis in this branch of science has ever been worked out.

It would lead me too far now from my actual subject to deal with that question in a comprehensive way, and I will therefore mention only one important reason, viz., the fact that studies and inquiries about the proximate investigation of textile fibres and fabrics up to the present day are regarded as belonging much more to the province of botany, histology, or zoology, than to that of analytical chemistry.<sup>(2)</sup> From the practical point of view of textile industry, I hold quite an opposite opinion, and I have now to describe a system for the examination of textile fibres and fabrics, so as to ascertain their proximate constituents, which I think is convenient for all practical purposes.

A laboratory to be used for the examination of textile fibres and fabrics must be somewhat differently fitted up from that which is required for general chemical analysis; but although the microscope is a most important instrument for our purpose, it must not be regarded as the only means of making observations of this kind, as many have thought. It is the continuous working into one another of microscopy and chemistry, or, better expressed, of microscopical and macroscopical chemistry, that is the special characteristic of all investigations upon textile fibres and fabrics.<sup>(3)</sup> Any good microscope can be used for the purpose. I, however, especially recommend the use of one having revolving nose-pieces for rapidly changing the objectives, an arrangement which is coming more and more into general use. Furthermore, it is of importance, in making certain special observations, to use the well-known illuminating apparatus by Abbé<sup>(4)</sup> in conjunction with the microscope. It is self-evident that a micrometer is absolutely necessary for measuring the dimensions of the fibres. As I proceed I shall describe the reagents which have to be used, partly in connexion with the microscope and partly without it. Besides, there is especially one other kind of instrument which must be a chief feature in a laboratory for the examination of textile materials, i.e., an apparatus for determining the moisture of the fibres.

(2) The most important works of Wiesner, Vétillart, Nathusius Bowman, &c. tend in the direction of botany, histology, or zoology. See Wiesner, "Rohstoffe," p. 298, and Hugo Müller, "Pflanzenfaser" (in the Vienna Exhibition Reports), pp. 6 and 23.

(3) At any rate this is my own experience, after a long time of practical work on this subject. I was glad to find a similar opinion expressed by Cross, Bevan, and King in their "Report on Indian Fibres and Fibrous Substances," 1887, in the following passage, p. 2: "These authors (viz., Hugo Müller and Vétillart) have kept in the main to a distinct line of investigation, chemical or microscopical, only incidentally introducing the other, its essential complement. A method compounded of the two lines of inquiry must necessarily give a more complete account of the subject-matter, and this we think will be found from the method about to be described." Now, on Hugo Müller's negative view on *chemistry alone* for the distinction of vegetable fibres from one another, see his "Pflanzenfaser," p. 23; therefore it might be expected that microscopy would have an essential part in all investigations, but with reference to the most important vegetable fibres, this is by no means the case in the above-named Report, *cotton* and *flax* being treated quite insufficiently, and *hemp* not at all. This is much to be regretted when we consider what difficulty may arise in distinguishing hemp and flax fibres from each other. (See Schacht, Wiesner, Vétillart, Cramer, and Hohnel.)

(4) The instrument used by me is constructed by Zeiss, in Jena.

All textile fibres are hygroscopic,<sup>(5)</sup> some of them in a very high degree, and it is not sufficient to apply to them the methods which are used in general chemical analysis, because it is for practical purposes necessary to work with larger quantities (as regards weight and volume), in order to get the exact average of moisture.<sup>(6)</sup>

The apparatus<sup>(7)</sup> for such determinations is constructed in such a way that the substance to be dried is suspended in hot air of about 108° C., the air passing through the apparatus in a continuous current. Above the hot air chamber a chemical balance is fixed, an arrangement by which it is possible to weigh the substance while it is in the interior of the apparatus.<sup>(8)</sup> This offers two advantages; the precise moment can be ascertained when perfect dryness is attained, and, secondly, the prevention of re-absorption of moisture. It is of course understood that during the process of weighing the current of air has to be stopped.

All textile fibres are divided into three chief classes:

1. Those of vegetable origin;
2. All animal wools and hair; and
3. The different kinds of silks.

Now, if we get any textile material of unknown composition, the first fundamental experiment which we have to make is to examine some simple microscopical water objects of the material. If the substance is a raw product, a loose material, or a yarn, these objects are taken from different parts of the said material, but if we have a ready-made article, then the different parts of it have to be examined separately, e.g., in rectangular textures, the warp and the woof.<sup>(9)</sup>

The simple microscopical properties of the textile fibres as they present themselves to the eye, moderately magnified, are well known, as follow:

The fibroine fibres of all silks are uniform, and either entirely of a homogeneous structure, or show simple longitudinal lines.

The fibres of animal wool and hair have either characteristic scales on their surface, or a central medulla, or both of these properties; and as regards vegetable fibres, which exist in a great number of different forms, the most important of them are characterised by their *lumen* (central cavity of these fibres), and in a negative sense they are marked by the absence of the qualities which we have seen to be characteristic of wool, hair, and silk.

But a knowledge of these simple characteristics is not sufficient, for fibres are inclined to vary. Natural causes as well as changes produced by artificial influences can affect the appearance and the qualities of the fibres in various ways,<sup>(10)</sup> the artificial influences being produced by the different kinds and modes of manufacturing. Hence it follows that it is necessary to assist microscopical examinations by chemical reagents.

First, let us observe the influence which cold concentrated sulphuric acid has upon fibres. If they are immediately dissolved by the acid, and little or nothing remains, then the substances are either silk or vegetable, or a mixture of both. But if the fibres are not rapidly dissolved, and epidermal scales appear standing out from the surface, we have wool or hair before us.

Secondly, let further microscopical objects be subjected to the action of cold concentrated hydrochloric acid. The fibres will be dissolved if real silk is the object acted upon, but should vegetable fibres, animal wool or hair, and Tussah silks<sup>(11)</sup> be the objects, the fibres will remain unchanged by the action of the said acid.

(5) See Persoz, Sur le Conditionnement, 1878, pp. 104 and 391; Bolley, "Spinnfasern," pp. 24 and 34; Hugo Müller, "Pflanzenfaser," p. 30, and others.

(6) See Persoz, Conditionnement, p. 163; Storhay, Renseignements pratiques sur les Conditions publiques, 1888, p. 7.

(7) See drawing.

(8) See Talabot, Un Procédé nouveau sur la Condition; Bolley, "Spinnfasern," pp. 36-37; Persoz, Conditionnement, p. 116.

(9) See Hohnel, Mikroskopie, p. 106.

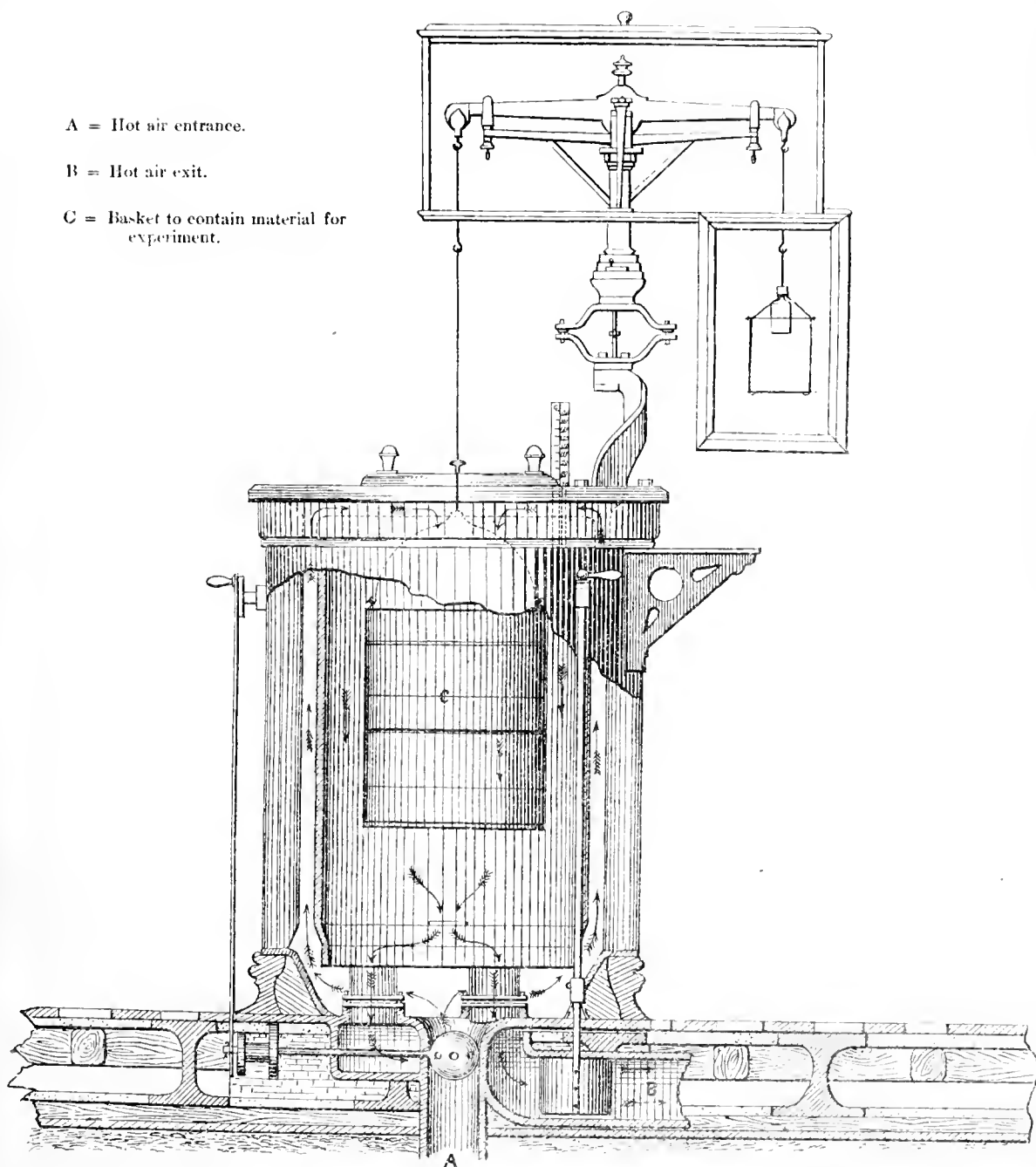
(10) See Hugo Müller, "Pflanzenfaser," pp. 23-24; Hohnel, Mikroskopie, p. 111.

(11) See Wiesner and Praseh, über Seiden in Mikroskop. Untersuchungen, pp. 45-54; Hohnel, Mikroskopie, p. 149; and Jour. Chem. Soc. 1888 (II.), p. 857.

A = Hot air entrance.

B = Hot air exit.

C = Basket to contain material for experiment.



This apparatus, the invention of M. Storhay, has been modified in various particulars by Mr. Hermann Schlichter and myself. By using Perret's heating system, it can be maintained at a constant temperature for hours without danger of injury to the fibres.





Thirdly, let a piece of the material to be experimented on be boiled for 10 minutes in a solution of 10 per cent. of caustic potash or soda, and then carefully rinsed and washed several times. The fibres of all kinds of silks, of wool, and hair are by this treatment dissolved,<sup>(12)</sup> but should anything remain, then the fibres can consist only of vegetable matter, the nature of which has to be ascertained by the microscope.

Finally, we have to ascertain the presence or absence of Tussah silks. For this purpose boil a piece of material for a short time in concentrated hydrochloric acid, and examine it microscopically. If a quantity of small but clearly defined portions of fibres manifest themselves with sharp and straight terminals, which very often show longitudinal lines, the presence of Tussah silks is proved.<sup>(13)</sup> Of course, other fibres may also be present, but this does not interfere with the reaction.

I now proceed to the quantitative distinction<sup>(14)</sup> of the several classes. For this purpose we take a piece of material, weigh it and put it into the drying apparatus, and as soon as it is entirely desiccated, weigh it a second time. By this means we find the hygroscopic moisture of the fibres. Then by boiling dyed and ready-made materials in highly diluted hydrochloric acid, we ascertain the amount of colouring matter, finish, dressing, &c.

Thirdly, we have to examine separately the various constituents found to be present by the preceding qualitative tests. If the substance is a mixture of *silk and wool*, we can dissolve the silk fibres in it with concentrated hydrochloric acid, or with dilute sulphuric acid, and the wool will remain unchanged. After thoroughly washing them, we dry and weigh the remaining fibres, which will give us the amount of wool, that of silk being ascertained by subtraction. But if we have a combination of *silk and vegetable fibres*, the silk can be dissolved only in hydrochloric acid, and the result is obtained by proceeding as in the last experiment. Finally, *wool and plant fibre* combined are separated in a similar way, viz., by boiling the material in a weak solution of caustic potash or soda, wool being dissolved.

But should we have a compound of the three substances, wool, silk, and plant fibre together, we must proceed as follows:—Wool and silk are simultaneously dissolved by caustic potash or soda, and the unaffected remainder will, of course, be vegetable fibres. Again, another piece of the same material being taken, we dissolve the silk and vegetable fibres by a weak solution of sulphuric acid, leaving the wool unaffected. The amount of silk is ascertained by subtraction. Of course, all weighings have to be made when the fibres are in a perfectly dry state.

Having now solved the preceding problems we come to more detailed ones of the several classes before us. And this is the especial province of the microscope, for there are differences which can never be discovered by macroscopical means, owing to the similarities<sup>(15)</sup> existing in a great variety of substances. Let us begin with the **VEGETABLE TEXTILE FIBRES**. Only a limited number of them find at the present time extensive application in the European manufacture of textile fabrics,<sup>(16)</sup> and I therefore confine my remarks to the most important representatives of this class. Of course the most important is *Cotton*. As a general rule the different kinds of cotton fibres are flat (band or ribbon-like), with a plainly discernible lumen; and they have a peculiar way of being twisted corkscrew-fashion.<sup>(17)</sup> Whilst the cotton fibre is a kind of vegetable hair, *Flax* is a

bast fibre of the linum, and is distinguished from cotton by its round or polyedric cross-section. The lumen of flax is narrow, and the fibre, as in cotton, consists for the most part of pure cellulose. Again, flax fibre frequently shows joints transversely to the fibre, giving it an irregular and chain-like appearance.

The bast of *Hemp* is of similar construction to flax, but its cells are more irregular, its cell walls more resisting, and the lumen is sometimes of varying dimensions. But these peculiarities are not sufficient to show with accuracy and promptness the true differences between flax and hemp, for there are many flax fibres, especially those taken from the lower part of the stem, which are so very similar to hemp fibre that the one cannot even microscopically be distinguished from the other.

While pure flax fibre consists mostly of cellulose, and—in case of undyed fibres—with iodine and sulphuric acid gives a blue reaction, hemp in part contains woody substances, and therefore with the just named reagents may give green or yellow colours.<sup>(18)</sup> But this reaction is by no means general and conclusive, for there are many hemp fibres which show exactly the same result, as cotton and flax, in blue.<sup>(19)</sup> Therefore attempts have been made to find the difference between the fibres by micrometric observations of their diameters, but unfortunately without any really satisfactory result.<sup>(20)</sup> A further distinguishing feature was supposed to exist in the appearance of the ends of the fibres in their natural state:<sup>(21)</sup> those of flax being assumed to be single, while those of hemp were thought to be double pointed. But then this difference is only partially correct, for many hemp fibres have been found terminating like flax in a single point.<sup>(22)</sup>

Finding all this insufficient to establish a true proximate analytical difference between flax and hemp, I tried another method which I found to give good and accurate results. The lumen of flax fibre always contains a certain amount of protoplasm<sup>(23)</sup> which is not destroyed by sulphuric acid, as cellulose is, but remains unchanged. Now the lumen of hemp very rarely shows the presence of protoplasm,<sup>(24)</sup> and hence hemp and flax fibres are very easily distinguished in this way, the one from the other. Some microscopical objects subjected to the action of sulphuric acid of 75 per cent. will solve the question without difficulty. In both cases the cellulose of the fibre is quickly destroyed by the action of the acid. In the case of flax there remains the protoplasm of the lumen as a most distinctly marked line or narrow ribbon; whilst hemp shows nothing of this, but is quickly reduced to a more or less shapeless mass.

I find this method to be still easier and give better results if iodine is used previous to the application of sulphuric acid. Of course this is possible only when the material to be tested is *without dye*. I have referred to the reaction of iodine and sulphuric acid, which is well known as being highly characteristic for vegetable fibres.<sup>(25)</sup> It is for one reason only not of *general* importance for the proximate analysis of fibres and fabrics, as it can be applied only to undyed fibres.<sup>(26)</sup>

(12) See Schlesinger, *Gespinnstfasern*, pp. 13, 15, and 26; Hohnel, *Mikroskopie*, p. 36.

(13) See Schacht, *Gewebe*, p. 25.

(14) See the micrometric measurements in Vétillard, *Études*.

(15) See Schacht, *Gewebe*, pp. 21, 23, and 26, and compare Hohnel, *Mikroskopie*, pp. 37–39.

(16) See Schacht, *Gewebe*, Table VI., Fig. 4, a; Vétillard, p. 77 and Table 7; Wiesner, *Techn. Mikroskopie*, p. 110, and Rohstoffe, p. 376; Schlesinger, *Gespinnstfasern*, p. 26; Cramer, *Programm d. Zurich Polyt.*, 1881.

(17) See Vétillard, *Études*, Table 7; Hohnel, *Mikroskopie*, pp. 35 and 36; Hugo Müller, "Pflanzenfaser," p. 38.

(18) See Schlesinger, *Gespinnstfasern*, Table, pp. 14–15; Wiesner, *Rohstoffe*, p. 390.

(19) Colour reactions—such as reactions with iodine, phenol, phloroglucin and indol, nitric and picric acid, plumbite of soda, stannic chloride, sulphate of aniline, &c., &c.—have hitherto been used to a great extent in distinguishing fibres. This is correct as long as the fibres are in a raw state, or are found in undyed materials. But as soon as the fibres are dyed, bleached, &c., as is well known to be the case in a vast number of textile fabrics, then the colour reactions vary considerably. Hugo Müller and Hohnel share this opinion. It is for these reasons that I have avoided colour reactions as much as possible in my general analytical treatment of fibres, and substituted acids and alkalis as reagents. The above-named reaction of iodine and sulphuric acid refers to a special case, as is stated expressly.

(12) See Schacht, *Gewebe*, pp. 18 and 30.

(13) At any rate, this is my own experience. About the collective name "Tussah Silks" see Hohnel, *Mikroskopie*, p. 150.

(14) See Remont, *Jour. Pharm. Chim.*, 1880; *Chem. News*, No. 1294; *Jour. Soc. Chem. Ind.* 1882 and 1884; and Persoz, *Compt. Rend.*, 55.

(15) See Bowman, *Structure of the Wool Fibre*, p. 127; *Grothe*, *Gespinnstfasern*, p. 1; Wiesner and Praseh, *Seiden*, *Micr. Unt.*, p. 54; and others.

(16) See Vétillard, *Études*, p. 252; Cross, Bevan, and King, *Report on Indian Fibres*, p. 1.

(17) Cotton may always be recognised very easily by these generally known features, no difficult investigation being necessary. See Bowman, *Structure of the Cotton Fibre* (plates); Hohnel, *Mikroskopie*, pp. 26 and 27; Vétillard, p. 141; Schacht, *Gewebe*, pp. 23 and 24.

I find that it is, as a rule, insufficient and unsatisfactory to use solutions of one degree of strength in all experiments as has been hitherto the rule exclusively,<sup>(26)</sup> for the best results are often lost owing to the improper strength of the reagents. I therefore recommend the following:—The solution of iodine to be always the same, and consist, e.g., of 1 part of potassium iodide in 100 parts of water, to which solution iodine is freely added.<sup>(27)</sup> But the sulphuric acid must be used in different degrees of strength. I use, for instance, a series of 25, 30, 40, &c., up to 90 per cent. of pure concentrated sulphuric acid.

I will now mention very briefly some other vegetable textile fibres<sup>(28)</sup> which are of more or less practical importance in addition to cotton, flax and hemp<sup>(29)</sup>.

*China grass* consists of bast fibres, the form of which is rather irregular, partly ribbon-like, partly round and with a lumen varying in size. It never appears under the microscope rolled up in a corkscrew form, as is the case with cotton.

*Jute* is another kind of bast fibre material. The lumen often varies in width, a sign by which the fibre is easily distinguished from others.

*New Zealand flax* is a fibre obtained from the leaves of the plant producing it. It consists of cells, sharply pointed at the terminals, and circular in cross-sections, the lumen and cell-walls being uniform throughout.

We now proceed to the second of our classes, viz., the different kinds of SILKS. Ordinary raw silk consists of double fibres which are coated with the so-called silk gelatine,<sup>(30)</sup> which is not a part of the fibre itself, and must be subsequently removed by boiling either in water or soap. The fibre throughout is a homogeneous and cylindrical body of fibroine. Besides genuine silk, other kinds of fibres of similar origin have been introduced into the silk trade, known by the collective name of *Tussah silks*.<sup>(31)</sup> They may be distinguished from genuine silk by various features.

First, it must be stated that their diameters are totally different,<sup>(32)</sup> that of real silk being much smaller and more uniform than that of Tussah silks.

Secondly, these last silks show distinctly longitudinal lines in their fibroine fibres,<sup>(33)</sup> and also often colouring matter in the same,<sup>(34)</sup> these properties being absent in real silk.

Thirdly, a special property of Tussah silks is the uneven thickness of the single fibres.<sup>(35)</sup> Genuine silk is quite uniform in this respect.

And lastly, in certain Tussah silks characteristic bands cross the fibres in an oblique direction,<sup>(34)</sup> the fibres appearing of a lighter colour in these bands. They originate in two fibres crossing each other in the cocoon before they have become hard. If the contact takes place after the fibres have hardened, no bands are of course produced, and either the frequent appearance or the absence

of the same is characteristic of many though not of all these silks, and forms another feature by which they may be distinguished from genuine silk. We see there is no difficulty in distinguishing real silk from other kinds, but, if we wish to classify the silks, the best method is the micrometric measurement of their diameters. But until now this was possible only in a very incomplete manner, for the diameters of Tussah silks are very variable, as I have remarked before. Attempts have therefore been made to find the greatest diameter of any single fibre.<sup>(35)</sup> This may be done in a few instances, but to continue the operation each time in 20 or 30 cases with accuracy, and strike an average of the whole, is a practical impossibility. I have, however, found that genuine silk as well as all other kinds, if treated with caustic potash (Tussah silks may also be treated with hydrochloric acid), fall into a great number of very small particles which completely retain their original structure, and show sharp and straight terminals. These small particles are admirably adapted for an easy and perfectly accurate determining of the diameters, and besides this they show the narrower parts of the fibres in a marked manner.

I found a peculiar passage in the second volume of the Journal of this Society, pp. 172—173. Reference is made there to certain colour appearances of the different kinds of silk. This passage is wholly unintelligible, for there can only be a question of polarised light, but this is not stated there. I do not know whether this inaccuracy has since been rectified, and I only mention the matter in case no such correction has been made.

The third of the classes of textile fibres is composed of all sorts of ANIMAL HAIR AND WOOL, the most important of which is the wool of the sheep. Hair and wool may be generally described as compounded of three different parts:<sup>(36)</sup> the innermost is a cylinder containing the medulla, next comes a horny stratum of fibre, and externally is a scaly layer of epidermal cells. According to the nature of the wool one or the other of these parts may be wanting, or may be of less importance. In many, especially the fine sheep wools, the innermost stratum—that containing the medulla—is absent, and the fibre gains thereby in uniformity and firmness.<sup>(37)</sup>

Amongst all animal fibre *sheep's wool*, of course, takes the first rank in practical importance. There are a great many varieties of sheep's wool,<sup>(38)</sup> as is well known, but all being absolutely equal in substance, the analytical investigation must be of an almost exclusively microscopic nature, chemical reagents finding only a subordinate place in their examination.

1. *Coarse wools*, in which many thick hairs are found in conjunction with the fine wool. The former are characterised by a thick central medulla, which constitutes the greater part of the fibre. In this class of wool the finer fibres differ materially from one another, and show no uniform features.

2. The so-called "*landwollen*,"<sup>(39)</sup> in which the finer fibres more predominate as in coarse wools, though thick hairs are always present. The finer fibres of this wool are frequently bent in an abrupt and irregular manner, and the diameters of single fibres are unequal.

3. The type of the *crossbred wools* has very long and strong fibres with very little inclination to curl. The fibre scales are very conspicuous, and the character of the wool itself is beautifully uniform, though the fibres are coarser than that of fine merino wools. A further characteristic feature is the absence of the medulla.

The so-called Lincoln and Leicester wools are relatives of the crossbred wools, to which they correspond in many respects. The medulla, although frequently absent, is not entirely so.

4. The most important and finest quality of all kinds of sheep's wool are the *merino wools*. The thinness, fineness, and uniformity of their fibres (coarse and stiff hair

<sup>(26)</sup> See Schacht, Gewebe, p. 8; Schlesinger, Gespinnstfasern, pp. 6 and 12; Wiesner, Rohstoffe, p. 300; Vétillart, Études, pp. 28—29; each observer giving his own recipe.

<sup>(27)</sup> See Vétillart, Études, p. 28.

<sup>(28)</sup> Cross, Beran, and King (Report on Indian Fibres) seem to attach particular analytical importance to the lengths of the ultimate fibres. But although this is of high morphological importance, it is not of the slightest value analytically, because it is a practical impossibility to measure the lengths of fibres after they are spun into yarns and woven into textiles; especially when such fibres are many hundred times longer than broad (See Wiesner, Vétillart and Bowman). Wiesner, e.g., has made the most minute investigations upon the lengths of fibres, and places particular morphological importance on it, but he does not use it in the analytical distinction of fibres. See Wiesner, Rohstoffe, p. 238 "Kennzeichen der Fasern;" p. 343, on distinction of cotton and flax; p. 375, of flax and hemp; p. 395, of jute, flax and hemp.

<sup>(29)</sup> Of the few following vegetable fibres only the chief characteristic features are given, because they are not of the same general importance as cotton, flax and hemp. Next to these comes jute, of which however Vétillart says (Études, p. 252): "Le jute ne se recommande par aucun autre avantage que son bas prix et la facilité avec laquelle on est arrivé à le filer."

<sup>(30)</sup> See Bowman, Wool Fibre, Plate III.

<sup>(31)</sup> See Hohnel, Mikroskopie, p. 150.

<sup>(32)</sup> See Schlesinger, Gespinnstfasern, p. 42, and Hohnel, Mikroskopie, p. 152.

<sup>(33)</sup> See Schlesinger, Gespinnstfasern, pp. 42—44; Hohnel, Mikroskopie, pp. 114 and 145.

<sup>(34)</sup> Wiesner and Prinsch, über Seiden, Mikr. Unt. pp. 45—54; Hohnel, Mikroskopie, p. 152.

<sup>(35)</sup> Schlesinger, Gespinnstfasern, p. 42.

<sup>(36)</sup> See Bowman, Wool Fibre, Plates V. and VI.

<sup>(37)</sup> See Grothe, Technologie der Gespinnstfasern, p. 18.

<sup>(38)</sup> See the admirable treatment of this subject in Bowman, Structure of the Wool Fibre.

<sup>(39)</sup> There seems to be no special expression for this type of wools in the English language, at least I could not get a collective name from practical people in the wool trade.

being entirely wanting) may be mentioned first. The total absence of the medulla is another feature connected herewith. Furthermore, the manner in which the epidermal scales are arranged is also characteristic. For, these cup-like scales fit one into the other, foot into mouth, and consist of one piece.<sup>(40)</sup> In other wools this is not the case, for the fibres are not encircled by one single scale, but always by a combination of several scales.

As I have previously stated, hair and wool of all other kinds of animals are of much less importance in the manufacture of textiles than sheep's wool. But I might briefly state the ways in which some of the said wools and hairs differ from each other. The wools of the *Angora goat* (41) consist of a mixture of fine wool and coarse hair; they are comparatively stiff, uncurled, uniform, and covered with flat scales. The hair is characterised by the thick medulla in the middle, which gets gradually and uniformly thinner towards the top of the fibre.

Similarly constituted is *alpaca wool*, which is not curled, and is found usually in dark or black varieties. When black, the fibres are too opaque to allow anything of their structure to be discerned. But gently warmed with sulphuric acid of 60 per cent. strength, the scales separate from the body of the fibre; the fibres become partly lighter and semi-transparent, and it can easily be observed that the original colour which, prior to the application of the acid, seemed to be uniformly black, is really a thick layer of natural brown colouring matter.

Very different from these are the thin, fine, and uniform fibres of *vicunna wool*, which are similar to camel wool and appear without scales. They are likewise not curled. In addition to fine fibres the vicunna wool contains thick hair which shows a strong medullar development. The fibres throughout manifest much natural brown colouring matter.

What is called *camel hair* is also a mixture of coarse hair and fine wool. The fine wool fibres are characterised in part by their having medullar cells or tubes. The difference of the development of the medulla in the coarser hair is furthermore characteristic, as it usually appears in all degrees and varieties. Camel hair always contains natural brown colouring matter in different degrees of intensity, varying from almost white to dark brown.

I have mentioned several times that textile fibres may contain certain quantities of natural colouring matter in their cells. In vegetable fibres and silks such colouring matters are practically only of subordinate importance, but in many cases it is essential to discover whether the brown colour of certain hairs or wools is natural, or has been given them by dyeing. It is not possible, however, to ascertain this by an ordinary microscopical observation,<sup>(42)</sup> and although the natural colour will offer a greater resistance to reagents than artificial ones do, this property is by no means a general one. Moreover, as brown is the predominant shade of all nature-coloured animal fibres, and this tint has few microscopical varieties artificially, this fact tends to make the matter still more difficult. But I may say I have found a practical and very simple method by which the natural and artificial colours of hair and wool can be distinguished the one from the other. For this purpose a microscope with Abbé's illuminator attached must be used. For a general examination of textile fibres diaphragms with the smallest apertures must be employed with this instrument, in order to observe the structures of the fibres. But if animal fibres have to be examined, whether they are of a natural colour or not, the objects must first be treated with a cold solution of caustic potash or soda, in order to produce swelling, by which the different parts and strata of the fibres become clearly visible.

Now, if such objects be brought under the microscope and the diaphragms of the illuminator entirely removed, so that the whole aperture of the objective is filled by rays of light, then we get a view of the fibre which does not show much of its structure, but presents the colour reactions so much the better. For, if the colour is an artificial one, the fibre

appears to be a homogeneous and uniformly dyed body; but if the colour is natural, we perceive the colouring matter arranged in a peculiar way in certain parts of the fibre, forming there longitudinal stripes or lines, consisting of small points or grains, and this arrangement is so characteristic that a mistake is absolutely out of the question.

Another material which needs a peculiar method of examination is *shoddy*, which is used in conjunction with good wool. It is well known that large quantities of old woollen articles and cuttings, rags, &c., are worked up again, spun and woven a second time, and reappear in the market. Enormous quantities of shoddy are now annually produced in Great Britain and on the Continent, and because it is generally four or five times cheaper than new wools are, we can form an idea of the practical importance of the shoddy trade. But this introduction of shoddy into textiles is not always done "in good faith," but often the said material is palmed off as good wool, and the public pays for it a much higher price than it is worth. In fact, a great many materials, especially those made of short wools, are more or less adulterated with shoddy. From this fact we may learn the great importance of having correct analytical methods of proving the amount of shoddy in textiles. But to accomplish this many different things have to be observed, in order to test fibres whose structure and chemical properties differ from good wool only to a certain degree.

None of the methods (43) known hitherto gave correct results, and therefore I apply my own test, which is as follows:—

All kinds of shoddy contain more or less vegetable fibres which are always to be found in old woollen articles, rags, &c., and which cannot be totally removed chemically from shoddy without damage to the shoddy. Frequently these vegetable fibres are dyed with different colours, according to their different origin. Now, after having made the tests to discover vegetable fibres in general, a piece of the material, which should be as large as practically possible, is weighed, then cut into pieces, and each of these pieces beaten in a mortar arranged for the purpose. Then the pieces are brushed on a specially prepared board, and what is brushed off is carefully collected. This having been done, the pieces are weighed again.

By this process a part of the shoddy is removed from the material, of course partly mixed with fibres of good wool, but these are brought into account, as we will see at once. The next step is, to examine the brushed off fibres most carefully under the microscope. Here shoddy always shows a great variety of features which are absent in good wool.

The most striking of these are:—

1. Wool fibres of differently dyed colours.
2. Damaged wool fibres.
3. Vegetable fibres, partly dyed.
4. Small particles of wool fibres with brush-like ends.
5. Absence of epidermis of the wool fibres.

To these features others may come according to circumstances. I repeat, a most minute and careful microscopic examination is necessary, in order to decide all these points. This examination also shows us the proportion of the shoddy fibres to the good wool fibres in these brushed off parts. Then these parts, as well as the pieces of the material from which they were brushed, are dissolved in caustic potash or soda, each separately, the solutions filtered through fine platinum filters, and the vegetable fibres collected in both cases from the filters. The amount of these vegetable fibres is then ascertained in both cases either by weighing, or by microscopical measurements.

Now experiments show that such vegetable fibres are always found uniformly divided in shoddy, and therefore we have the following calculation:—

1. Original weight,  $a$ .
2. Weight of brushed pieces,  $b$ .
3. Weight of brushed off fibres,  $a - b$ .
4. Amount of shoddy found by microscopic estimate in the brushed off fibres  $\frac{1}{x}$  of the total amount of these.

(40) Watson Smith, this Journal, 1889, 20, Figs. 8 and 9.

(41) Bowman, Wool Fibre, Plate 28.

(42) See Höhnel, Mikroskopie, pp. 90 and 101.

(43) See Schlesinger, Gespinnstfasern, p. 62; Höhnel, Mikroskopie, p. 107; Focke, Arch. d. Pharm., Vol. 24, p. 619. Höhnel's method is the best of these, but it cannot be applied for quantitative determinations of shoddy.

5. Proportion of vegetable fibres in brushed off parts to vegetable fibres in brushed pieces 1 :  $m$ .

Then it is evident that  $m$  times as much shoddy is contained in the brushed pieces as in the brushed off fibres, and therefore the total weight of shoddy in the material is  $\frac{a-b}{x} + m \frac{a-b}{x} = \frac{a-b}{x} (1+m)$ ; and the percentage of shoddy can easily be calculated from this.

I point out again that the factor  $\frac{1}{x}$  is based on a microscopical estimate, the more exact this is, the more exact will be the final result.

It would remain for me to deal with the most important *hygroscopic properties* (<sup>44</sup>) of textile fibres and fabrics, and their analysis, as well as the analytical treatment of *raw products*, especially *raw wool* (<sup>45</sup>) which contains, as you are well aware, besides moisture, a large amount of accessory substances, such as grease, sand, dust, burrs, &c. But this subject is too important to be treated only briefly at the end of this paper. Perhaps I may have an opportunity of dealing with it another time.

In conclusion I should like to mention that I am indebted to my brother, Mr. Hermann Schlichter, for the illustrations which appear with the paper. The drawings of the fibres have been made from microscopic observations, unless otherwise noted.

#### DISCUSSION.

The CHAIRMAN said that the very perfection of the gravimetric and thermometrical methods of analysis, as also the perfection of the microscope, was apt to constitute a source of danger. The chemist was accustomed to think that everything that could be known in chemistry was to be learnt by means of the balance and the burette; while the microscopist hardly considered that knowledge which could not be demonstrated by the microscope. Dr. Schlichter's paper dealt with one of those wise combinations of purely chemical and purely physiological methods which indicated the great possibilities of the progress of scientific investigation. It was by such mixed methods that immense progress could be made in arriving at approximate analyses. Of the commercial value of accurate analyses it was impossible to speak too highly. The triumphs of shoddy were wonderful; and to be able to say promptly and definitely what any particular fabric was made of was a triumph, not of pure chemistry, but of such mixed methods as Dr. Schlichter had explained. The value of these accurate methods of research to technical work was at present only dimly appreciated by those who had most to learn from them.

### A REVIEW OF THE METHODS OF PRODUCING OXYGEN COMMERCIALY.

BY E. T. THORNE, PH.D.

SOME time ago, when reading a paper before this Section on some industrial applications of oxygen (this Journal, 1889, 82—88) I promised a description of the process for producing oxygen on the industrial scale developed by the Brin's Oxygen Company. In redeeming my promise I think it may be of interest to our members if I preface this description with a brief sketch of the various methods which have been used or proposed for obtaining oxygen on the large scale.

(<sup>44</sup>) See *Musin*: Observations sur le Conditionnement Hygrométrique des Matières Textiles, 2<sup>e</sup> edit., 1877.

*Persoz*: Essai sur le Conditionnement, 1878.

*Storhay*: Renseignements Pratiques sur les Conditions Publiques, 1888.

(<sup>45</sup>) See *Grothe*. Technologie der Gespinnstfasern, p. 156. Zeitsch. d. Ver. d. Wollinteressenten, 1871.

The interest attaching to this problem from a practical point of view is shown by the fact that our Patent Office alone has, since 1849 (the date of the earliest patent I have found on this subject), granted no less than 53 patents for improvements in obtaining oxygen on what was intended by the patentees to be the commercial scale. I shall not, of course, attempt to notice all these specifications, many of which are manifestly absurd, contenting myself with referring briefly to the most important or remarkable ones; but at the end of my paper I give a complete list of the English patents on this subject.

The methods which have been employed for obtaining oxygen may be broadly classified under four heads:—

1. The decomposition by heat of compounds containing oxygen.
2. The decomposition by chemical means of compounds containing oxygen.
3. The decomposition by electrical means of compounds containing oxygen.
4. The extraction of oxygen from the atmosphere.

It would be going beyond the limits of this paper to review those methods which are only of theoretical or historical interest, and I shall therefore refer only to such processes as are of interest from a commercial or practical point of view.

1.—*Production of Oxygen by the Decomposition by Heat of Compounds containing Oxygen.*—The historically interesting method of preparing oxygen by the ignition of nitre—



by which Priestly first obtained oxygen in 1771, is also of interest from the point of view of this paper, as not only was it long one of the chief means employed for preparing oxygen in the laboratory, but was also the basis of many attempts to prepare this gas economically on the large scale. The first English oxygen patent recorded was taken out by S. White in 1849 (No. 12,536) for "Improvements in the manufacture of gases," wherein he proposed heating potassium or sodium nitrate in detached or moveable crucibles within retorts. Many subsequent patents were taken out (the last as late as 1865) for modifications of this process—nitrates or mixtures of nitrates with oxides or other non-fusible substances being employed. But the oxygen thus obtained is generally much contaminated with nitrogen and nitrogen oxides owing to the further decomposition of the nitrite when the heat is allowed to become too high, and no satisfactory results were obtained.

Priestly also described in 1779 the preparation of oxygen by heating manganese peroxide, when a third of the oxygen contained therein is evolved:— $3 \text{MnO}_2 = \text{Mn}_3\text{O}_4 + \text{O}_2$ . This peroxide occurring largely in nature as pyrolusite formed a cheap and convenient source for oxygen where considerable quantities were required, and its use has continued till the present day. But the high temperature needed has militated strongly against its general use.

But it is to Berthollet, who in 1785 first obtained oxygen by heating potassium chlorate above its fusing point, that we owe the method which until recently has probably been more used than any other. The heating of the pure chlorate is, however, attended with considerable danger, especially where large quantities are being used, as owing to decomposition commencing very little above the fusing point of the salt, and to the small amount of heat absorbed, or rendered latent, during the decomposition, the reaction when once it has started often becomes explosive in its violence, ruptures the containing vessel and scatters the molten chlorate. But the discovery that the admixture with the chlorate of small quantities of some non-fusible oxides (such as manganese peroxide, ferric oxide, &c.) causes the evolution of the oxygen to take place at a much lower temperature, far below the melting point of the chlorate, converted this into a very convenient means for producing oxygen. And the cheapening of the cost of potassium chlorate in late years rendered this so far an economical process that it almost superseded the rather more economical, but also much more cumbersome manganese peroxide method.

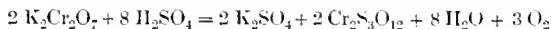
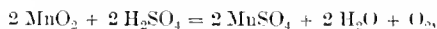
Another method which has found favour to some extent where considerable quantities of oxygen were required, is the decomposition of sulphuric acid by allowing it to drop on to red-hot surfaces. The acid is decomposed into sulphurous anhydride, water and oxygen—



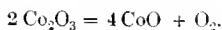
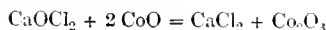
and when oxygen alone is required, the water and anhydride are removed by passing the products of the reaction over suitable absorptive media. This method, however, has often been employed in the manufacture of sulphuric anhydride, when only the water has been removed by a desiccating agent, a mixture of sulphurous anhydride and oxygen in the proportions requisite for the formation of sulphuric anhydride being thus obtained. This process is probably cheaper than any of those just described, but the greater complexity in manipulation has made its use restricted mainly, I believe, to the manufacture of sulphuric anhydride. It forms the subject-matter of several patents, and is the process suggested by Squire in his patent for the manufacture of sulphuric anhydride (Eng. Pat. 3278, 1875) as the best for that purpose.

With regard to the relative cost of these methods it is difficult to speak with certainty, as authentic data are wanting. But even when prepared on the large scale, the cost per 1,000 cub. ft. will certainly, I believe, not be less than 8*l.*—10*l.* from chlorate, 4*l.*—6*l.* from pyrolusite, and 2*l.* 10*s.*—3*l.* from sulphuric acid, and probably, in the latter case, where deterioration of plant, &c. is taken into account, the cost will be much higher.

II.—*Production of Oxygen by the Decomposition by Chemical Means of Compounds containing Oxygen.*—In this class there are, as far as I know, no methods which have been employed on a really large scale, but there are several which are of interest to the practical man, where a small quantity of oxygen is required to be prepared without much trouble or expense, or where a slow but steady evolution of oxygen is required. Of these the oldest are the decomposition of peroxidised compounds, such as manganese dioxide, potassic bichromate, &c., by sulphuric acid. Taking these two as examples, the reactions are expressed by the equations—



A very steady stream of oxygen may be obtained by adding a small quantity of a cobalt or nickel salt to a concentrated solution of bleaching powder kept at a temperature of about 85° (Mitscherlich, 1843). The cobalt or nickel seems to act by being alternately oxidised to a higher and reduced to a lower state of oxidation, in this way acting as a carrier of oxygen, and liberating that contained in the bleaching powder. The reactions may be expressed by the equations—



Really only a modification of this method, and a modification hardly likely to find much practical application, is the process wherein a stream of chlorine is passed into hot milk of lime, whereto a little cobalt or nickel salt or oxide has been previously added. The proposal to obtain oxygen from water by passing a mixture of superheated steam and chlorine over very strongly heated surfaces was also too cumbersome and costly to find much practical use.

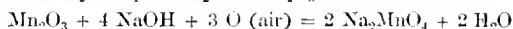
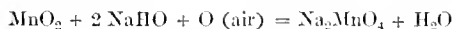
III.—*Production of Oxygen by the Electrolysis of Compounds containing Oxygen.*—In the preceding divisions we have been dealing with processes where a great part of the cost lies in the raw material which is destroyed in the process. We now come to a class where the cost of the raw material may be almost neglected, the expense lying in the means of obtaining the decomposition. For although the electrolysis of various other substances has been proposed, and even patented (*Gr.*, C. Watt, Eng. Pat. 13,755, 1851) for this purpose, we need only consider that of water as of practical importance. The first patent referring to

the use of the electrolysis of water for the production of oxygen is that of Madame Lefebvre (Eng. Pat. 1045, 1859), but, of course, the process had been known long before. The electrolysis of water has undoubtedly been used practically for the preparation of oxygen on the large scale, but the large and inevitable loss attending the conversion of heat into power and power into electrical force, and the need for skilled labour in working the electrical plant, must always militate very strongly against the economy of this method. It is difficult to obtain data of the actual cost of producing oxygen electrolytically, but in an elaborate and detailed specification of a recent patent (Eng. Pat. 15,935, 1888), the patentee, D. Latchinoff, estimates the cost of producing 23,000 ft. hydrogen (to be used for ballooning purposes) and 11,500 ft. oxygen at 36*l.*, and there is little doubt that the cost in practice would be found to be considerably higher.

IV.—*The Extraction of Oxygen from the Atmosphere.*—When Boussingault in 1851 made his important discovery that if barium oxide be exposed at or a little below a dull red-heat to a current of air it becomes converted into barium peroxide, and that this new compound is, at a somewhat higher temperature, reconverted into the monoxide with evolution of oxygen, he at once recognised the importance of this reaction as a means of extracting oxygen from the atmosphere, a problem which had long occupied the thoughts of chemists. That others also appreciated the value of Boussingault's discovery is evidenced by the fact that we find an English patent taken out in the following year (Eng. Pat. 390, 1852) by J. Swindells and W. Nicholson to utilise this reaction. Further patents on similar lines followed, and Boussingault and others devoted much work to the endeavour to bring the process to a successful practical issue. Large experimental works were erected in Glasgow, and much money spent thereon in a similar effort. But all these attempts failed to overcome the difficulty of the gradual loss by the barium oxide, after repeated oxidations and deoxidations, of its power of re-oxidation, and the reaction remained practically unavailable.

In 1865, J. T. A. Mallet took out a patent for extracting oxygen from the air by means of cuprous chloride. Cuprous chloride, when exposed to air, and especially to moist air, becomes converted into cupric oxychloride, and this, when heated to a dull red heat, yields up its oxygen, becoming reconverted into cuprous chloride, which may be again oxidised as before. The reoxidation may be partially effected in the retorts in which the oxychloride is heated, by passing air through them when partially cooled, and this reoxidation may be very much accelerated by the admission of a small jet of steam with the air current.

In 1866, Tessié du Motay and C. R. Marechal took out a patent for the extraction of oxygen from the air through the intermediation of alkaline manganates (Eng. Pat. 85, 1866). When an oxide of manganese is mixed with a strong base, and is subjected to the action of a current of air at a moderately high temperature, a manganate of the base is formed. If this manganate is then heated to a higher temperature and brought in contact with superheated steam, it is decomposed, oxygen is evolved, and a mixture of the alkali and manganese oxide re-formed. The reactions may be expressed by the following equations, though of course the actual mixtures re-formed will vary according to the quantity of steam and the temperature employed—



The mixture was said to undergo no deterioration (though this was not found to be the case in practice), and the process seemed thus a continuous one of great promise. Companies were formed and works erected at Paris, Lille, Brussels, Vienna, and New York. For a time some of the streets of Paris were lighted by oxyhydrogen lights, a double system of piping being laid down for the purpose, and the New York Company undertook and carried out the lighting of the work during the building of Brooklyn Bridge. It is difficult to obtain authentic information of the cost of

oxygen as produced by these companies, but it appears to have been about 3*l.* to 4*l.* per 1,000 cub. ft. In the Journal of the German Chemical Society (Ber. 1870, 901) there is a glowing account of the then state of the New York Company, which was said to be producing 3,000 cub. ft. per day, some of which at least it was selling at the handsome price of 18*s.* for 60 gallons, or 1*s.* 10*d.* per cub. ft. A curious commentary, however, on the flourishing state of the company is the fact that it failed in 1871, and all the other companies formed for working these patents seem to have suffered a similar fate.

Probably both this and the cuprous chloride methods produced oxygen, when prepared in very large quantities, at a far lower cost than that of any of those previously described, and their failure was most likely largely due to the cost still being too high for the application of oxygen to industrial pursuits and to the quantity required for retail trade capable of standing the high cost, being too small to make such large works profitable.

The same may probably be said for the next process of extracting oxygen from the air by taking advantage of the varying solubility of oxygen and nitrogen in water or other solvents, for which a patent was taken out by Mallet in 1869 (Eng. Pat. 2137, 1869). Taking water as the most suitable solvent, the coefficient of solubility of oxygen is .046, that of nitrogen .025. The air was compressed over water, the excess of nitrogen allowed to escape, and the dissolved gases extracted from the water by the help of a vacuum pump. In this way a mixture containing 33.3 per cent. of oxygen was obtained, and if this mixture was subjected to a repetition of the treatment the percentage of oxygen was increased. After four treatments, a mixture containing 75 per cent. of oxygen was said to be obtained, and after eight treatments oxygen of 97.3 per cent. purity resulted. This process seems to have been adopted by Phillips in his experimental lighting of part of Cologne about 1871-72 by special oxygen-fed oil lamps.

Attempts have been made to utilise the law of diffusion of gases, and of the fact established by Graham that caoutchouc membranes allow the passage of oxygen more readily than that of nitrogen, but hitherto with no practical success, and these phenomena do not seem likely to lend themselves to the production of oxygen on the large scale.

It was thus that the matter stood when, in 1880, the MM. Brin, frères, took out their patent (Eng. Pat. 1416, 1880), in which they claimed to have overcome the deterioration of the baryta, which had hitherto been the stumbling-block to the realisation of Boussingault's expectations. The permanency of the baryta was attained by a combination of great care in the preparation of the baryta in a suitable physical condition, of the careful purification of the air, and of the employment of reduced pressure during deoxygenation, by which means a lower temperature suffices for such deoxygenation. Further improvements were patented by the MM. Brin in 1885. The specially prepared baryta was heated in horizontal steel tubes or retorts to a dull red heat (about 600°) and a current of air, carefully dried and purified, forced over it under a pressure of about 10 lb. on the square inch. When the oxidation was completed, or carried as far as was found practically desirable, the pumps were stopped and the temperature of the retorts raised to a full red heat (about 850°) and connected with the pumps working as vacuum pumps. The previously absorbed oxygen was then evolved and passed from the pumps into the gas-holder. The process was thus made workable and continuous, but practical working still showed defects, and the cost of the oxygen obtained still remained too high for its application to industrial purposes. One of the difficulties lay in the formation of channels above the baryta owing to the subsidence of the latter; another, the difficulty of keeping the joints of the connecting pipes perfectly tight, owing to the irregular expansion of the retorts horizontally embedded in a brick furnace. Both of these difficulties were overcome by the adoption of vertical retorts. These are supported at the upper end, the lower end passing loosely through asbestos packing, and so allowing all expansion and contraction to take place freely. All the pipes connecting with the pumps, &c. are fixed to the upper ends of the retorts, and are thus not

affected by the expansion or contraction of the retorts. But the greatest defect in this process, when considered as one to be put in the hands of manufacturers, was the great changes of temperature required. Not only did these changes increase very markedly the wear and tear of the plant, but they caused such a great waste of time whilst they were being effected, that whereas what we may term the *active* or pumping period of an operation occupied from an hour and a half to two hours, the whole operation could not be effected much under four hours.

It has long been known that where a solid and a gas combine to form a solid, increase of pressure aids such combination, and conversely that when a solid decomposes into a solid and a gas, reduction of pressure aids such decomposition. It was hardly anticipated, however, that this effect of change of pressure would be sufficient to remove the need of change of temperature in a reaction such as that under discussion, but experiment showed that such was the case. It was found that if the temperature of the baryta was maintained at about 700° (or a little below the mean of the temperature previously used), a current of air under a pressure of about 10 lb. on the square inch above atmospheric caused considerable peroxidation, and that when the air current was stopped and the pressure reduced to about 13-14 lb. below atmospheric a considerable evolution of oxygen and deoxygenation of the peroxide occurred. Theoretically 1 lb. of baryta should absorb during peroxidation 1.25 cub. ft. of barium, and of course this same amount should be evolved on subsequent deoxygenation. In practice, where two temperatures were employed, about half the theoretical amount was obtained per operation. When a constant temperature of about 700° is maintained about .1 cub. ft. per operation is the result. But whilst an operation under the old conditions occupied about four hours, an operation under the constant temperature method can be completed in from 10 to 15 minutes. So that although the yield per operation under the new conditions is much smaller than heretofore, the yield per day is much larger. The maintenance of one temperature effects, of course, a considerable economy in fuel and wear and tear, and what is perhaps more important still from a practical point of view, it economises both in the quantity and quality of labour required. The multiplied reversals of pumps, cocks, &c. required by the new method is effected automatically by reversing gear designed by K. S. Murray, so that the labour required is reduced to that needed for stoking the furnaces and oiling and supervising the pumps.

For the barium oxide, the main desiderata are porosity and hardness. These are found to be best obtained by preparing the oxide from the nitrate. The nitrate is gradually filled into fireclay crucibles already heated in a coke fire to about 880°. The nitrate rapidly fuses, and decomposition soon commences, with evolution of oxygen and oxides of nitrogen. This decomposition continues for about three hours, during which time the contents of the crucible remain in violent ebullition, owing to the evolved gases. A porous mass is thus left, which is ignited for another hour to complete, as far as possible, the decomposition. In this way a very porous, but also hard baryta is obtained, the yield being about half the nitrate taken. The cost of the baryta is about 7*s.* per cwt. This is higher than the quoted price of many manufacturers, but hitherto samples that have been obtained elsewhere have failed to combine the requisite hardness and porosity.

The purification of the air is an important item in the success of the process, and this is effected by passing it through one or more drying chambers filled with quicklime, and finally through one containing lumps of caustic soda. In this way almost the whole of the carbonic anhydride and moisture is removed, the main work being done by the lime, the soda really acting as a catch purifier. Taking 1.25 per cent. by volume as a very rough average of the moisture present in the atmosphere in Great Britain (and this is certainly above rather than below the actual figure), one ton of lime should purify 3,000,000 cubic feet of air from moisture and carbonic anhydride, and in practice about 70-80 per cent. of this efficiency is attainable. Very little soda is used up, and both the spent lime and spent soda are



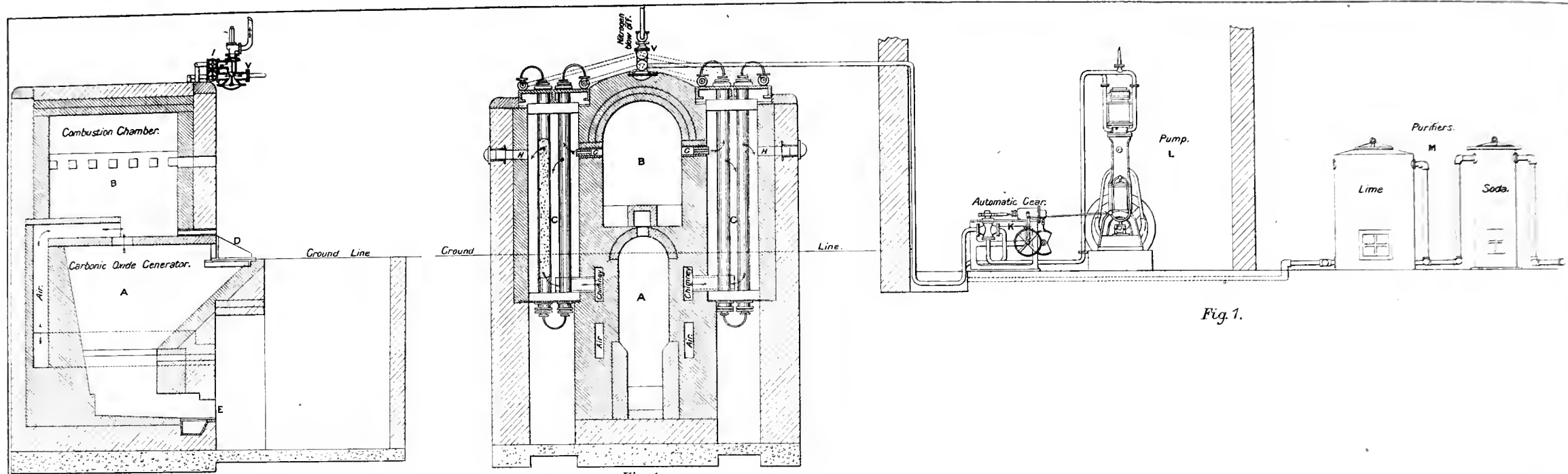


Fig. 3.

Fig. 1.

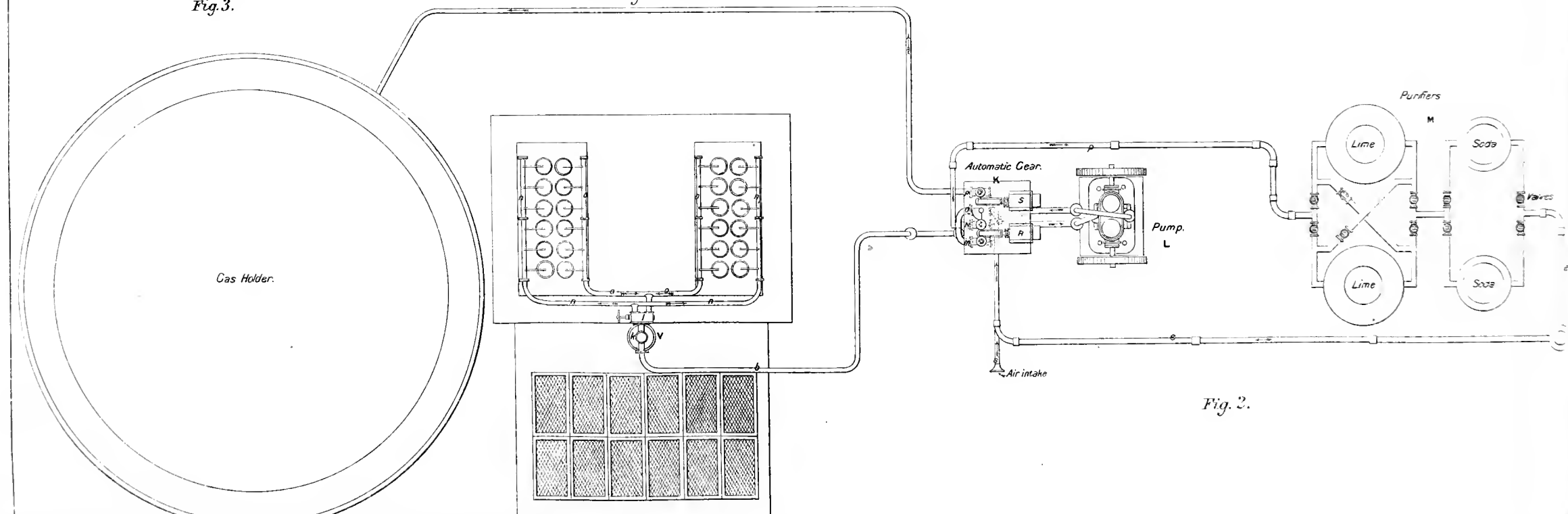


Fig. 2.

Fig. 1.

Fig. 2.

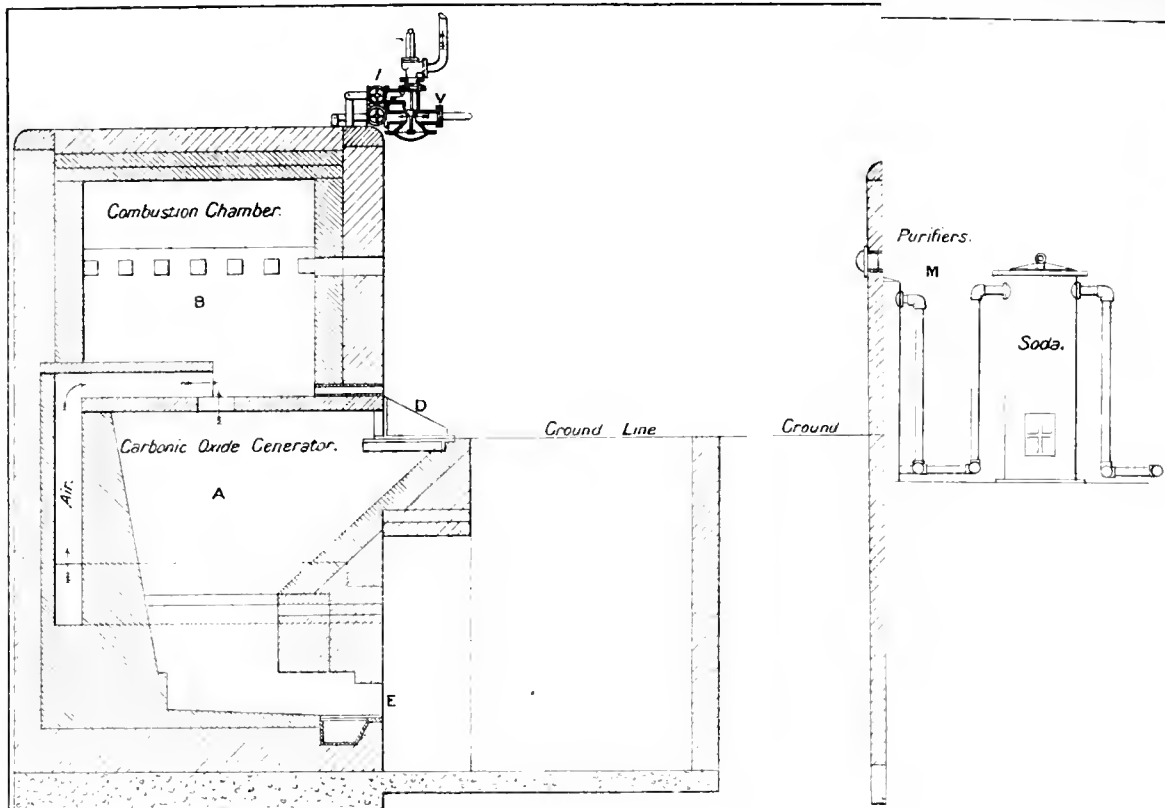
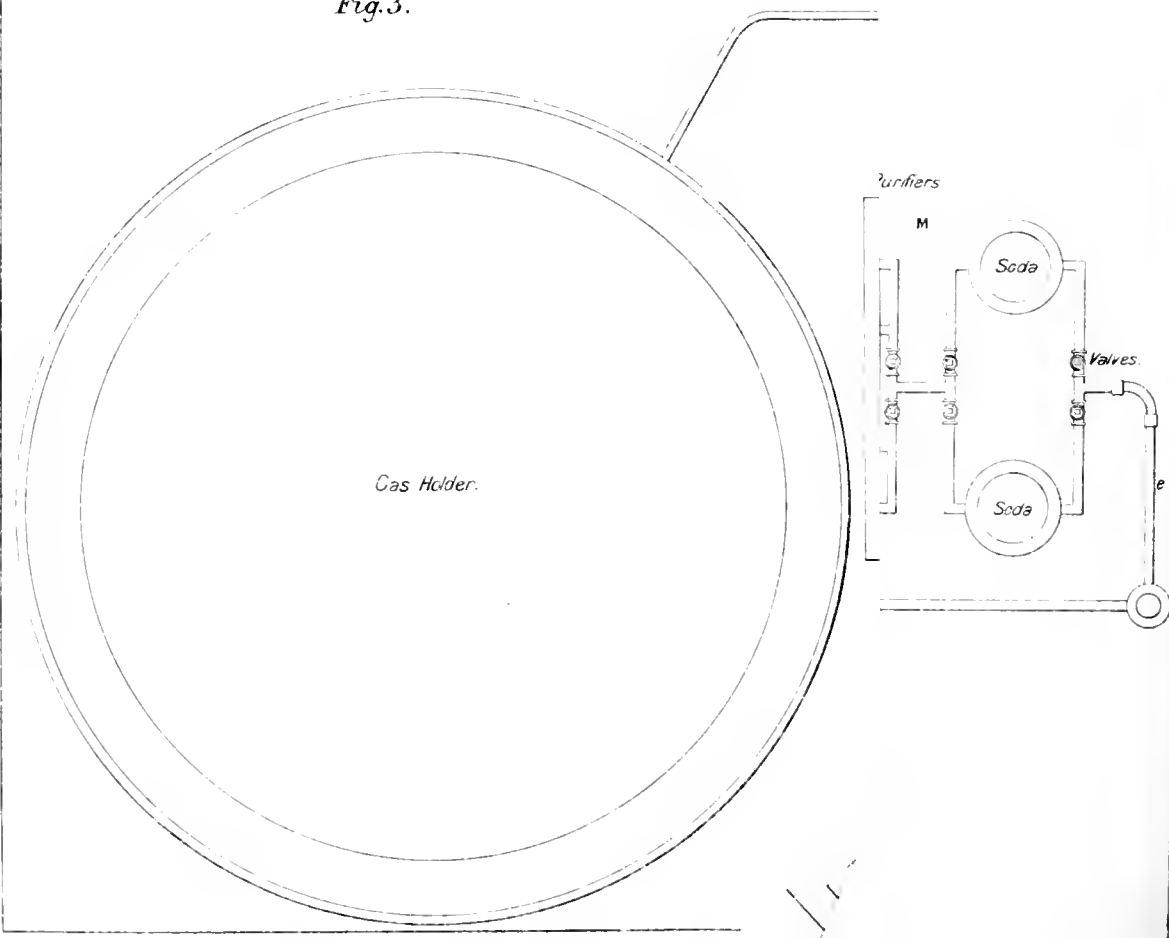


Fig. 3.



saleable, so that even their prime cost, which is small, is further reduced.

With regard to the purification of the air, there is a point of interest to which I would briefly refer. Of course, the purification of the air on a large scale cannot practically be made as perfect as on the small scale. Boussingault purified his air very thoroughly, and found the baryta gradually lose its absorptive power for oxygen. The admission of steam to the baryta restored its absorptive power, but at the same time had a deleterious effect, by causing disintegration. Now, all my experience in connexion with this process tends to confirm my belief that the trace of moisture left in practice in the air aids in keeping the surface of the baryta in a good physical condition, without being sufficient to cause disintegration. And though to the causes mentioned earlier in this paper the success of the process is largely due, I believe that the almost inevitable imperfection of an industrial process plays, in this one case, a not altogether negligible rôle in its success.

A detailed description of the plant used in this process is given by K. S. Murray, in a paper "On the Mechanical Appliances employed in the Manufacture and Storage of Oxygen" (Proc. Inst. Mech. E. Jan. 1890), and it is, therefore, unnecessary to repeat it here. The accompanying diagrams will, however, help to make the process clear. Fig. 1 shows the arrangement of purifiers, pumps, and producer, the latter being given in section parallel to its front. Fig. 3 shows a section of the producer taken at right angles to the front, through the centre of the carbonic oxide generator and combustion chamber. Fig. 2 shows a plan of the whole plant.

The carbonic oxide is generated in A (Figs. 2 and 3), which is fed with coke through D, the primary air-supply entering by restricted openings at E (Fig. 3). The carbonic oxide thus generated passes through the opening at the top of chamber A into the combustion chamber B (Figs. 2 and 3), being met at its entrance by the secondary air-supply, which, being admitted by the opening shown some little distance above E, has been heated by passing through the hot walls of the furnace, as shown by the channels marked "air." Complete combustion takes place in the combustion chamber B, and the hot products of combustion then pass by the outlet holes shown in plan in Fig. 3 and in section at G, Fig. 2, into the heating chambers C (Fig. 2) (placed on each side of the combustion chamber), in which the retorts are hung, finally passing away at the bottom to the chimney.

When the process is in operation, air is drawn in by the "air intake" (Fig. 2) through cock *m* by the pump L, by which it is forced, under a pressure of 10 lb. per square inch, through cock N, to the purifiers M. These are so

arranged that either one or both lime purifiers and one soda purifier are in action. The air then passes again through the automatic gear by the two-way cock O to pipe *b*, which takes it to the distributing valves V and I at the top of the furnace. Thence it passes, by pipes N, to the outside row of retorts in each heating chamber, through which it passes downwards, and thence by the connecting pipes shown in Fig. 2 upwards through the inner rows of the retorts, the impoverished air finally escaping by a relief valve on V. Provision is made in the distributing valves V and I whereby the direction of the air current may be altered from time to time, so as sometimes to pass downwards through the inner rows of retorts and upwards through the outer. When the oxidation has been carried on for the suitable time the automatic reversing gear shifts over the cocks, the purifiers being thus cut off under pressure. The exhaust cylinders of the pump being thus brought into direct communication with the retorts through the distributing box, soon cause a reduction of pressure therein, whereby the relief valve is automatically closed, and the pipes *n* and *o* both brought into communication with pipe *b*. As soon as the reduction of pressure in the retorts reaches about 26—28 in. mercury (13—14 lb. per square inch) below atmospheric the barium peroxide begins to give off its oxygen. This is then drawn to the pump, which delivers it into the holder. The first portions of oxygen so drawn into the pump still containing much nitrogen, the automatic gear is arranged not to open the cock to the holder till some few seconds after the reversing of the other cocks, the mixed oxygen and nitrogen meanwhile escaping by a snifting-valve opened by the gear for the purpose.

As now arranged the plant is one that can be readily erected in works where large quantities of oxygen are required, and needs no further skilled supervision than is always available in such works. For a producer capable of producing 10,000 ft. of oxygen per 24 hours the consumption of coke in the furnace is about 12—15 cwt., and for plant of that or smaller size the pump-power required is about 1 I.H.P. per 1,000 ft. of oxygen produced per day, the ratio decreasing for larger plants. The cost of oxygen making must always vary according to circumstances, but will be very small. Allowing for interest on plant, depreciation, &c., the cost into the holder will range from about 7s. per 1,000 cubic ft. in works specially erected for oxygen production, to 2s. per 1,000 in large works where fuel is low, and power and labour will entail little extra expense. This cost brings oxygen within the range of practical application for industrial purposes. Already numerous applications are in practice or prospect, and manufacturers are now turning their attention to the subject, so that doubtless many more will very soon be found.

## APPENDIX.

### ENGLISH PATENTS TAKEN OUT FOR THE OBTAINING OF OXYGEN.

Year.	No. of Patent.	Patentees.	Short Title of Patent.	Method Proposed for Obtaining Oxygen.
1849	12,536	S. White.....	Improvements in the manufacture of gases, and in the applications thereof, &c.	Heating potassium or sodium nitrates in detached or moveable crucibles within retorts.
1851	13,755	C. Watt.....	Improvements in the decomposition of saline and other substances, and in separating their component parts or some of them from each other, &c.	The electrolysis of saline nitrates and sulphates.
1852	390	J. Swindells and W. Nicholson.	Improvements in obtaining oxygen gas and applying it in the manufacture of various acids and chlorine, &c.	Heating barium oxide, alone or mixed with infusible oxides, in a current of air at a dull red heat, and decomposing the peroxide formed by a current of steam at a higher temperature.
1853	998	G. K. Geyelin .....	Improvements in the manufacture of white oxide of zinc.	Heating barium peroxide or manganese peroxide.
1859	1045	Mdme. Lefebvre (Agent, W. E. Newton.)	Improvements in the manufacture of nitric acid and its application for the production of artificial nitrous or nitric salts.	"Decomposition of atmospheric air by means of dynamic and static electricity."

ENGLISH PATENTS TAKEN OUT FOR THE OBTAINING OF OXYGEN—*continued*.

Year.	No. of Patent.	Patentees.	Short Title of Patent.	Method Proposed for Obtaining Oxygen.
1860	1380	G. Bower.....	Improvements in apparatus for manufacturing, controlling and regulating the flow of gas.	Heating manganese peroxide in special retorts.
1860	1563	C. Binks (provisional only).	Improvements in manufacturing oxygen gas.	The decomposition of steam by chlorine at high temperatures, and absorption of the hydrochloric acid formed.
1860	2132	H. J. Standley (provisional only).	Improvements in the production of gas for illuminating and other purposes, &c.	From nitrates and other compounds of sodium, potassium, &c.
1860	2711	J. Webster (provisional only).	Improvements in obtaining gas (mainly oxygen) for improving artificial light, and for other purposes, &c.	By heating potassium nitrate or other nitrate to a bright red heat in iron retorts.
1860	3179	C. Binks.....	Improvements in manufacturing certain gases applicable in generating heat and light and in bleaching.	Similar to 1563/60.
1861	932	J. D. Malcolm (provisional only).	Improvements in the manufacture of nitric acid and caustic soda which are also applicable to the obtaining of other chemical products.	Heating sodium nitrate with sand, &c.
1861	983	J. Webster (provisional only).	Improvements in manufacturing oxygen gas, and obtaining certain other products.	Heating alkaline nitrates with iron oxide.
1861	2607	J. Webster .....	Improvements in manufacturing oxygen gas, and obtaining certain other products.	Similar to 983/61.
1862	1496	C. Binks .....	Improved methods of obtaining oxygen and chlorine gases.	Similar to 1563 60.
1862	2265	J. Dickson (provisional only).	Improvements in the manufacture of chlorine for commercial purposes.	Electrolysis of saline solutions.
1862	2556	L. Mond (provisional only).	An improved method of obtaining hyponitric acid and nitric acid from nitrate of soda.	Heating sodium nitrate with infusible and indifferent oxides, &c.
1862	3085	C. Binks (provisional only).	Improved methods of obtaining oxygen and chlorine gas.	Similar to 1563 60.
1865	5	J. F. Parker and J. Tanner.	Improvements in the manufacture of oxygen gas, &c.	Heating a mixture of sodium nitrate and quicklime.
1865	1833	C. Tellier (provisional only). (Agent, H. A. Dufrené.)	An improved process for obtaining oxygen ...	Passing a mixture of chlorine and steam through a red-hot tube.
1865	2634	J. T. A. Mallet.....	A new or improved process for the manufacture of oxygen.	The alternate oxidation of cuprous chloride by means of air or air and steam to cupric oxychloride, and decomposition by heat of the oxychloride, with consequent evolution of oxygen.
1866	85	C. R. Marchal and C. M. Tessié du Motay. (Agent, R. A. Brooman.)	An improved method of producing oxygen ...	Alternate formation of manganates, chromates, &c. by heating mixtures of alkalis and oxides of manganese, chromium, &c. in a current of air, and decomposition of these manganates, &c. by a current of steam.
1866	3171	J. T. A. Mallet.....	A process and apparatus for producing jointly or separately oxygen and chlorine from the same chemical substance and in the same apparatus.	Mainly special plant for 2934 65, and adding an inert material to the active agents to prevent fusion, &c.
1867	568	H. A. Achereau. (Agent, W. Clark.)	Producing and applying oxygen .....	Decomposition of sulphuric acid by heat.
1867	1040	C. M. Tessié du Motay, and C. R. Marchal, (Agent, C. S. Brooman.)	Producing oxygen .....	Acting on sulphuric acid with oxides of iron, zinc, aluminium, or with silicate of aluminium.
1868	2352	J. Lewis (provisional only).	Combustion of fuel .....	Passing current of steam over any heated oxygenous compound.
1869	291	W. Weldon (provisional only).	Manufacture of chlorine and oxygen .....	Heating or treating with sulphuric acid Weldon mud.
1869	2137	J. T. A. Mallet .....	Charging atmospheric air with oxygen.....	Utilising greater solvent power of water for oxygen than for nitrogen. Air is subjected repeatedly to action of water under pressure.
1870	498	P. Broechi (Agent, J. H. Johnson) (provisional only).	Obtaining oxygen gas .....	Similar to 2137 69 but also using alcohol, glycerin, &c. in place of water.
1870	1300	E. T. Kirkpatrick.....	Producing oxygen gas .....	Adding a small quantity of a cobalt or nickel oxide or salt to a hot solution of bleaching powder.
1872	3261	J. A. Wanklyn.....	Production of oxygen gas .....	Heating a mixture of cupric oxide and barium oxide alternately in current of air and of steam.

ENGLISH PATENTS TAKEN OUT FOR THE OBTAINING OF OXYGEN—*continued.*

Year.	No. of Patent.	Patentees.	Short Title of Patent.	Method Proposed for Obtaining Oxygen.
1873	435	C. W. Harrison (provisional only).	Treating air for manufacture of gas.....	Treating air with water, or other liquid capable of dissolving more oxygen than nitrogen, under pressure.
1873	528	C. W. Harrison .....	Obtaining oxygen gas .....	Passing air into magnetised iron vessels either containing water or not. The nitrogen passes on the oxygen is retained.
1876	5907	C. Hornbostel. (Agent, W. Morgan-Brown.)	Preparing and applying oxygenated air for combustion.	Enriching air by pumping it through a paste of manganese peroxide and strong sulphuric acid, when oxygen is liberated which combines with the air.
1879	3302	P. Aube .....	Manufacture of lighting and heating gas .....	Absorption of air by water under pressure; or from manganates, &c.
1878	5123	G. E. Dering.....	Improved means of obtaining light, heat, and motive power.	Electrolysis of water.
1879	4127	A. Brin, A. Valière, and L. Q. Brin.	Producing of light and heat .....	Passing sulphuric acid into strongly heated retorts containing siliceous matter.
1879	4650	J. Rochford (provisional only).	Generation and application of oxygen and hydrogen gases for combustion in boiler furnaces.	Steam through heated earthenware or terracotta pipes, when oxygen alone is evolved (!)
1880	1416	L. Q. Brin and A. Brin. (Agent, P. Jensen.)	Production of oxygen and nitrogen gas.....	By alternately heating barium oxide in a current of air to form barium peroxide, and heating the peroxide alone, whereby oxygen is evolved and barium oxide reformed; increase and reduction of pressure being used as accessories.
1881	781	E. B. Reynolds (Agent, H. J. L. Haddan.)	Extracting oxygen from the atmosphere .....	Passing air through fibrous material soaked in naphtha, petroleum, or other fit product and broken charcoal, whereby the nitrogen is absorbed (!).
1881	1897	W. A. Barlow.....	Obtaining, &c. gas for lighting and other purposes.	Electrolysis of water. <i>Part</i> of the oxygen and hydrogen so produced is used to drive the dynamo. (Perpetual motion !)
1881	2080	N. A. Hélonis (Agent, A. M. Clark.)	Apparatus for making oxygen and hydrogen .	Heating a mixture of silica and plaster of Paris, when oxygen is evolved. Or dialysis of air through india-rubber septa. Or repeated absorption of air under pressure by water, alcohol, glycerin, &c.
1882	2875	R. J. Gülicher.....	Gas batteries and apparatus for producing hydrogen and oxygen by electricity.	Electrolysis of water.
1884	8285	A. Stamm (Agent, G. F. Redfern.)	An improved apparatus for preparing oxygenated air.	Elaborate apparatus for obtaining, by Mallet's principle of absorption of air by water, glycerin, &c., a constant stream of oxygen.
1884	15,026	A. and L. Q. Brin (provisional only).	Separating and obtaining oxygen and nitrogen from atmospheric air.	—*—
1885	157	A. and L. Q. Brin .....	Improvements in the separation and obtaining of oxygen and nitrogen from atmospheric air.	Improvements connected with 1416/81.
1887	7268	Hommel (provisional only).	Obtaining oxygen .....	—*—
1887	16,987	E. B. Ellice-Clark and L. Chapman.	Improvements in apparatus for the production of oxygen and nitrogen gases from atmospheric air.	Apparatus connected with carrying out patents 1416/81 and 157/85.
1888	1844	E. B. Ellice-Clark and L. Chapman.	Improvements in and apparatus for or connected with the production of oxygen and nitrogen gases from atmospheric air.	Apparatus and improvements connected with patents 1416/81 and 157/85.
1888	3880	E. B. Ellice-Clark and L. Chapman.	Improvements in apparatus for the extraction of oxygen and nitrogen from atmospheric air.	Improvements on and apparatus for patents 1416/81 and 157/85.
1888	3967	A. Brin .....	An apparatus for the extraction of oxygen and nitrogen from atmospheric air.	Improved apparatus for patents 1416/81 and 157/85.
1888	15,935	D. Latchinoff .....	Improvements in the production of oxygen and hydrogen in large quantities.	Electrolysis of water.
1889	11,899	C. Huelser (Agent, G. Kassner.)	A process for preparing plumbates of calcium, barium, and strontium, and for employing the same for oxidising purposes and for producing oxygen and other bodies.	Preparation of plumbates by heating mixtures of lead oxides and alkalis in a current of air, and subsequent decomposition of the plumbates by steam, or use in various ways as oxidising agents.
1889	13,712	R. D. Bowman (provisional).	Improved process for the production of oxygen gas.	Not yet completed.

\* Since 1884 the contents of a provisional specification are not made public unless the patent is completed.

## DISCUSSION.

The CHAIRMAN said that possibly from a purely scientific point of view the process of manufacturing oxygen which Dr. Thorne had just described was not more interesting than the most ancient of all such methods, which in one respect—the alternate reversal of operations—it somewhat resembled. He referred to the method for producing oxygen by the alternate oxidation and deoxidation of quicksilver by the slightly increased or diminished heat. But those who had ever tried the latter would appreciate the vast difference, from an economic point of view, between a process giving oxygen at a cost which he would not venture to calculate, and one yielding it at such a price as made it of economic value. The whole secret of the application of scientific truth to practical life turned on the question of *2 s. d.* It was not sufficient to have a perfect scientific or engineering process. What was essential was that the process should be workable at such a price as would give a profit on its practical application. Here was a process which was not alone in this respect—many other valuable and most important processes being in the same position—namely, that it was for a long time unworkable owing to difficulties of application rather than difficulties of theory. Those of the members who were acquainted with the earlier days of the process for the manufacture of oxygen from baryta would well remember the utter disappointment which followed the first trials; and it was additionally interesting therefore to find that the study of a series of minute details had led to results which were so highly satisfactory and encouraging.

Dr. SQUIRE said that he remembered seeing at Pantin, near Paris, many years ago, the process for producing oxygen patented by Du Mothay. Very sanguine views were expressed about it at the time; but, as Dr. Thorne had said, it did not succeed, because the whole thing sintered together into a mass in the retorts, so that there was practically no surface left for the absorption of oxygen. What, he fancied, had to a certain extent interfered with the prosecution of experiments in the manufacture of oxygen was the fact that in the present condition of metallurgy there scarcely seemed to be much use for it. The use of regenerative furnaces overcame any difficulty that there might be in the heating up of the nitrogen; so that when the air, which passed over the regenerators before it entered the furnace, came into the heart of the combustion chamber, the nitrogen in it could do no practical harm. A temperature could be got equal to the temperature of the dissociation of the compounds formed by combustion, and capable of melting down the furnaces. He did not know what more they would wish than that. The application of oxygen in large industrial operations seemed to be very limited. There were certain small metallurgical operations, however, such as the fusion of platinum, and there was the possibility of employing oxygen in the coal gas industry mixed with the gas, or for the regeneration of the oxide, though for that purpose air would practically do as well. Then there was the lime-light on the stages of theatres and for use in magic lanterns. Beyond these he did not see what was to be done with the oxygen when it was obtained. Much skill had been bestowed upon the apparatus, and it was very ingenious. Perhaps some of the automatic action might have been achieved in a simpler way; but apart from that it seemed to be well constructed. He repeated, however, that there appeared to him to be very little future for the oxygen manufacture.

Mr. B. E. R. NEWLANDS entirely agreed with Dr. Squire. He had seen the Brin process several times, and each time had heard of some new application of oxygen looming in the future. Some years ago it was to be a grand thing for purifying sugar. He sent a sample of syrup to be treated, and it certainly came back with a much better colour; but the density of the solution was so much reduced that he had no difficulty in ascertaining the reason for the colour improvement. He was told that the effect of the oxygen was to polarise the liquid in some way; that the ash in the sugar consisted of animalenka, and that on passing a current of ozonised oxygen through it, they marched out like a regiment of soldiers. He was

glad to see that the company now had the assistance of a gentleman of whom they all had a high opinion. That would put the affair on a scientific basis, which was greatly needed. On his last visit to the works he was told by a gentleman that the cost of producing the oxygen was 9s. per 1,000 ft.; another put the cost at 5s., and later on in the day the inventor of the process informed him that it could be made for 10d. Now, the cost of production was put at about 2s. 6d. per 1,000 ft. But of what use was oxygen at 2s. 6d. when it could be got in the air for nothing, and when one had merely to compress the air to get the same effect practically in most cases as with pure oxygen? He agreed with Dr. Squire that the only real use for oxygen at present was in the revivification of oxide of iron in gas making; but that operation could be done almost as advantageously by air. He felt grateful to Dr. Thorne for the interesting account he had given, but did not think the subject was of much practical importance at present. Perhaps, however, Dr. Thorne's work would be the means of opening up new uses for oxygen in the future.

Dr. THORNE in reply said that he had carefully refrained from going into the question of the applications of oxygen, because such an addition to his paper would have carried him too far. He had already, in a former paper, referred to several applications which he believed would come to be of great practical importance; and he hoped before long to be able to bring before the Society further information relating to the use of oxygen in other ways. It was easy to say that one did not see the need for oxygen, and that air would do as well. The English being a very conservative nation, it was not unusual to say of a new thing that it could not possibly succeed. Probably most of the members had heard of the famous lecture given by Dr. Lardner, wherein he proved to absolute conclusion that it was impossible to cross the Atlantic in a steamer, because, amongst other things, no vessel could carry sufficient coal for the voyage. At the conclusion of the learned doctor's argument, somebody at the top of the room was heard to laugh, whereat the doctor turned indignantly upon him to ask the cause of his mirth, and received the reply: "I beg your pardon, but I could not help it, for I have just landed from the first steamer that *has* crossed." When the question of oxygen production was introduced a few years ago in connexion with the Brin process, the company was met with similar arguments. It was said that it was impossible that baryta should retain its power of absorbing the oxygen; and learned members of the Society he was addressing, and of the Royal Society, were heard to state openly that they did not believe in the possibility, and that the oxygen produced by day must have been produced by fresh baryta brought in every night. The company had, however, been going on for three or four years, and last year they had sold by retail about a million cubic feet of oxygen. It was hardly necessary to say that they could not have gone on selling at that rate if they had had to import baryta into the works every night and put it into the retorts. They had in hand many chemical reactions, and were noticing the effect of mass in determining them. That, he thought, was one of the points too much overlooked when it was said that it was impossible to have large applications for oxygen. He would mention, though he had not intended to do so, that he had been making experiments on the thickening and oxidation of oils, and had succeeded in getting results with oxygen which had not been obtainable with air. The large amount of nitrogen which had to be pumped through the oil if it was to be oxidised by atmospheric oxygen interfered greatly with the result of the reaction. He fully believed that the oxidation of oils would in time become a very important outlet for oxygen. There were many other applications which he might speak of, but which he would prefer to leave over till a later date, when the experiments relating to them should have been more fully worked out than at present. He might mention, however, that over and above seven furnaces now erected and more or less closely connected with the company, they were putting up furnaces at two chemical works (one in Germany, the other in England) for use in applications worked out quite independently.

From Germany they were getting a great many inquiries with regard to the use of oxygen for the manufacture of sulphuric anhydride on the large scale, which he was convinced would be an important application of oxygen; and there were a number of others in view, and doubtless many more which had not yet occurred to them, but which might become large outlets for oxygen now that it was to be got at a price which would enable manufacturers to use it on a large scale. At the cost he had mentioned—varying from 3*l.* to 10*l.* per ton—oxygen would replace a great many other reagents that were being used at present. There was one application to which he had referred before, but which he would mention again in reply to the suggestion that air was as good as oxygen in almost all chemical reactions. He referred to the use of oxygen for bleaching paper pulp. He had proved, conclusively he thought, and on the large scale, that when oxygen was used in conjunction with bleaching-powder, a very large proportion of the latter might be replaced by a small portion of the former. In working on a ton of grass used for papermaking, which would require about 2 cwt. of bleaching-powder in the ordinary way, by using about 200 ft. of oxygen the bleaching-powder required would be reduced to about 1½ cwt. He had made careful experiments on the effect of using air in conjunction with bleaching-powder, and had found that, working in the laboratory on a scale of a quarter cwt. of material, air did not at all diminish the quantity of bleaching-powder required, whereas the use of oxygen in similar experiments reduced it in many cases by nearly 50 per cent. The difference was mainly due to the fact that, at the moment of reaction, chlorine and nascent oxygen were liberated, and the large amount of nitrogen passing through carried away some of this chlorine and oxygen before it had time to act. That showed that in many cases where one did not expect oxygen to be valuable it was so, for, he confessed, that when he entered on the bleaching experiments he did not expect a very beneficial effect. In conclusion, he could only say that, personally, judging from experience, he felt decidedly sanguine that there were large—very large—applications for oxygen, and that in the course of a few years it would be found to be playing a very important part in chemical manufactures.

## ESTIMATION OF ALUMINA BY TITRATION.

BY C. F. CROSS AND E. J. BEVAN.

In the current issue of the Bull. Soc. Ind. de Rouen (17, 402) there is an account of a paper by ourselves on "The Economy of Pure Caustic Soda" which appeared in this Journal (30 April, 1889). In the course of the investigations forming the subject of that communication, we obtained results showing that alumina in solution as alkaline aluminate could be estimated by titration in presence of methyl orange, the colour change taking place in the case of sulphuric acid at a point corresponding with the formation of a sulphate,  $2\text{Al}_2\text{O}_3 \cdot 5\text{SO}_3$ . It is noted in the transcript of our paper that in this observation we have been anticipated by M. J. Baeyer in a paper published in the *Moniteur Scientifique* (1886, 1133), and reappearing in a paper by M. E. Blondel in the Bull. Rouen 1887, 205. We take this earliest opportunity of acknowledging the priority of Baeyer's researches, the publication of which we had altogether overlooked.

Mr. P. BRAHAM exhibited an improved pinch-cock for regulating the flow of liquids, as from burettes, or gases in elastic tubes.

One form of the pinch-cock consists of two plates of suitable material—metal, vulcanite, xylonite, &c.—having

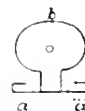
V-shaped slots (Fig. 1) and small clips *a*, turned over so as to retain the reverse slotted plate in position (Figs. 1 and 2, *a, a*). The plates have thumb-plates at right-angles thereto, for convenience in compression (Fig. 2, *b*). The pinch-cock, ready for use, is shown in Fig. 3. In action the slots

Fig. 1.



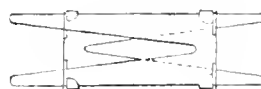
Plan of one Slide.

Fig. 2.



View of end showing Thumb Plates.

Fig. 3.



Plan of both Slides in position.

gradually overlap, and as their apices approximate, compression of the flexible tube takes place. The advantages appear to be the ease with which the pinch-cock can be removed without disturbance, and the manipulation with one hand while the other hand is left free to agitate or stir flasks or beakers, and for other necessary manipulation.

Another form of pinch-cock, in which the constricted-slot principle is effected by the compression of thumb-

Fig. A.

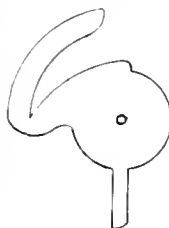


Fig. B.

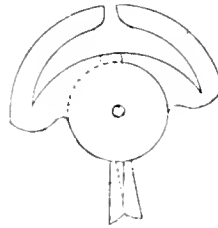
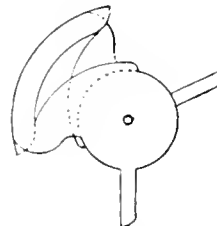


Fig. C.



Pinch-cock showing Curved Slots.

pieces, diminishing the size of the slot by rotary motions of the plates from a common centre. (See Figs. A, B, C).

The principle is adapted for pinch-cocks on a large and even technical scale.

MESSRS. CROSS AND BEVAN exhibited a set of electrical apparatus made by Mr. Hume, of Edinburgh, useful for demonstrations. The interest of the exhibit was its unusual cheapness.

Mr. THOS. CHRISTY exhibited a Bunsen burner with a device attached, in the form of a bow of platinum wire over the orifice of the burner, which prevented "backwardation" of the flame.



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SESSION 1889-90.

Notices of Papers and Communications for the Meetings to be sent to the Local Secretary.

Meeting held on Wednesday, 5th March, 1890.

DR. F. HURTER IN THE CHAIR.

## SULPHATE OF LIME IN CEMENT AND A DIRECT PROCESS OF CALCINATION.

BY J. S. RIGBY, F.C.S.

THE recovery of sulphur from vat waste by the Chance process having now assumed the form of a settled industry, manufacturers of that product will naturally look forward with interest to any advantageous arrangement for the

disposal of the residual impure carbonate of lime, which contains amongst its impurities a small percentage both of free and combined sulphur, which many people have thought would prove an insuperable obstacle in the production of a good reliable Portland cement.

As it is now over a year and a half since I made several tons of cement from the Chance waste, and as samples, such as briquettes and sample pats, placed on the walls of buildings fully exposed to the weather, have continued to improve in quality, it may be fairly said to have stood the test of time. The briquettes showed an increase on the tensile strain of 220 lb. on the 1 in. sectional area, the average breaking strain of the fresh cement on the usual seven days' test was 430 lb., whilst two briquettes 18 months afterwards tested as follows:—

No. 1.....	710 lb. on the square inch.
No. 2.....	590 lb. on the square inch.

giving an average of 650 lb. on the 1 in. sectional area, or an increase in tensile strength of 220 lb. as mentioned.

The bulk of the cement manufactured from the waste was quite equal in quality to the samples.

In bringing under your notice the manufacture of cement from Chance waste, it is for the purpose of showing that calcic sulphate existing in cement in small quantities does not to any appreciable extent interfere with the test of time. It will be noticed that the tensile strain exerted in the fresh cement was rather low; this I attribute to the rather excessive amount of the sulphur salt, owing to my not having at the time a good form of apparatus for purifying the waste (in quantity). The cement produced therefrom contained 2·85 per cent. of calcic sulphate, or 1·05 per cent. more than the average of three samples of London cement, which gave 1·80 per cent.

I find that the setting properties of cement can be restrained to almost an unlimited extent by the addition of plaster of Paris. Consequently the existence of calcic sulphate as an impurity in cement interferes with its testing properties very materially.

The following tests are made from cements produced from four distinct kinds of waste lime material, all of which contain calcic sulphate (naturally) in various proportions. For comparison I also include the test of a sample of London cement.

	London.	Caustic Lime Mud.	Chance Mud.	Solvay Mud.	Mud from Sugar Works.
CaSO <sub>4</sub> .....	1·80	2·20	2·85	1·60	3·60
	Lb.	Lb.	Lb.	Lb.	Lb.
Tensile strain on the square inch in seven days	480	468	440	467	390

It will be seen that the breaking strain is the lowest in those cements containing the largest amount of the calcic sulphate, but the breaking strain does not appear to decrease in proportion to the amount of the sulphur salt contained in the cement.

Although a cement containing the impurity mentioned to a limited extent may not at the end of seven days (the usual time in vogue for testing the cement) indicate a high tensile strain, it eventually increases in strength so as to compare favourably with cement of the best quality.

In making some experiments with cement by adding "plaster of Paris" by mixing the two materials in different proportions, I found that if I exceeded 4½ per cent. of the latter the briquettes subsequently made from the mixture either broke up after being placed in water for a time, or, if they retained their shape, they were very much cracked and gave a very poor test. This tends to prove that the crystallising properties of the plaster of Paris causes it to expand and so destroys the cohesive properties of the cement.

As regards the setting properties of cement, ordinary Portland cement should set in about two or three hours, so as to support the weight of a Viéts needle, but to a London cement which had been added plaster of Paris in the following proportions, 1½, 2, 2½, 3, 4½ per cent., and made

into briquettes, the setting properties of the same were restrained from 4½ hours to three days, the slight addition of 1½ per cent. of the plaster of Paris being indicated. The briquette containing the largest amount of the impurity gave a low tensile strain, and when broken I found the interior of the briquette quite soft; it had also cracked very considerably, and the surface of the briquette came off in scales.

I think the experiment goes very far to prove that although the existence of sulphate of lime in small quantities in cement is not hurtful, yet the injurious influence that is exerted by it when it exists in quantities exceeding 3 per cent. ought to render the manufacturer very cautious in purifying his raw material, when considering his production may prove a permanent source of danger to the stability of any work that may be subsequently executed. An irregular mixture of the raw materials would produce a similar effect to that produced by the calcic sulphate, especially where a large excess of the calcic carbonate has been added. The degradation or disintegration is not exhibited, however, in so marked a degree. I would also mention, as regards the presence of free alkalis in cement in small quantities, so far as I have been able to prove, I have not found them to have any injurious effect either in the tests of freshly made cement or in subsequent tests made nearly three years

Typical Merino Scales



Typical Merino Scales



Cells from  
Cortical Substance.



VARIOUS  
WOOL FIBRES.

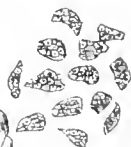
Cross Section of  
Merino Wool (Bohm)



Cross Section  
of Ilar (Bohm.)



Medullar cells



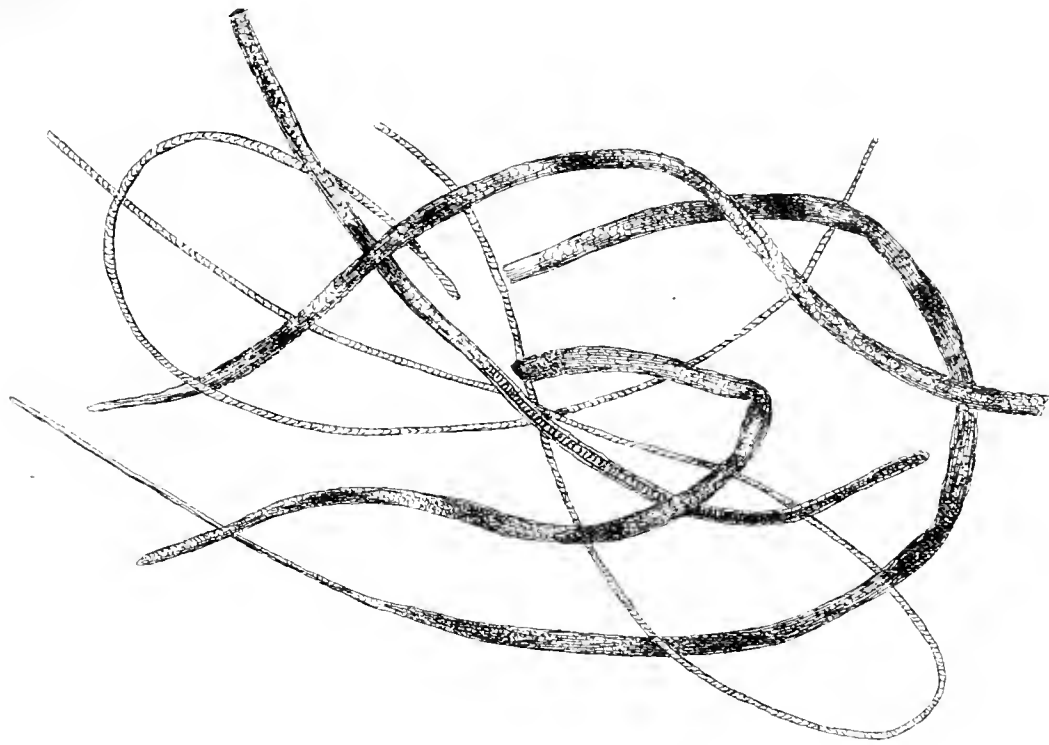
Cross Section of Lincoln Wool  
with medulla (Bohm)



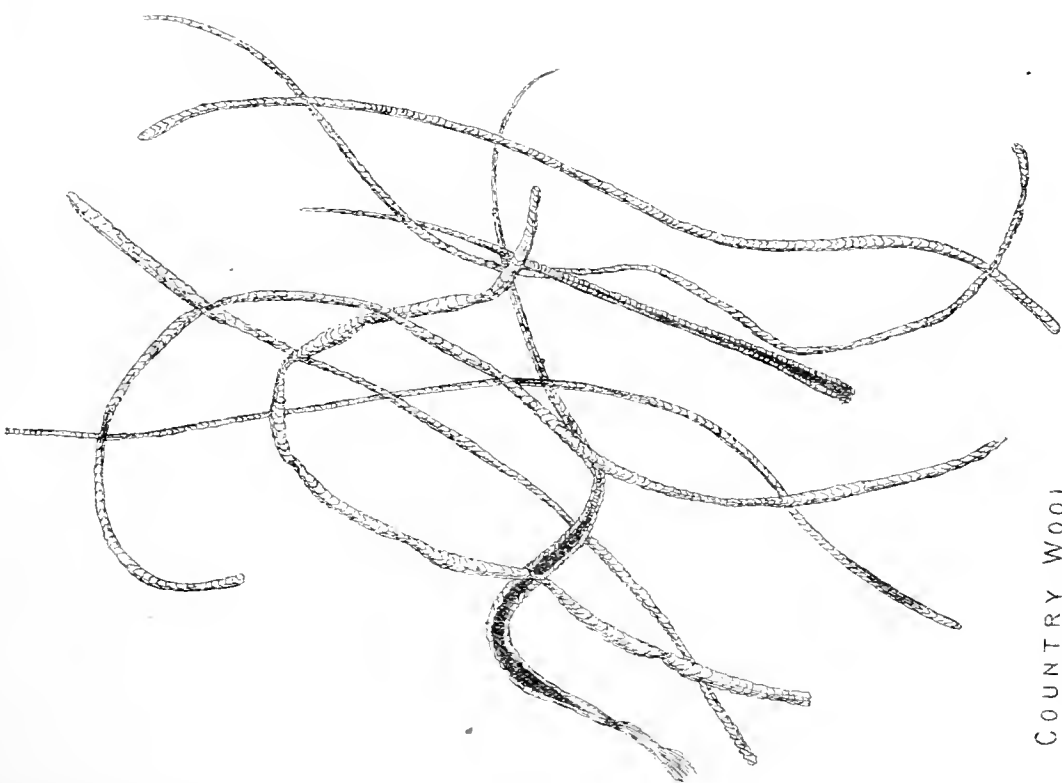
Epidermal Scales





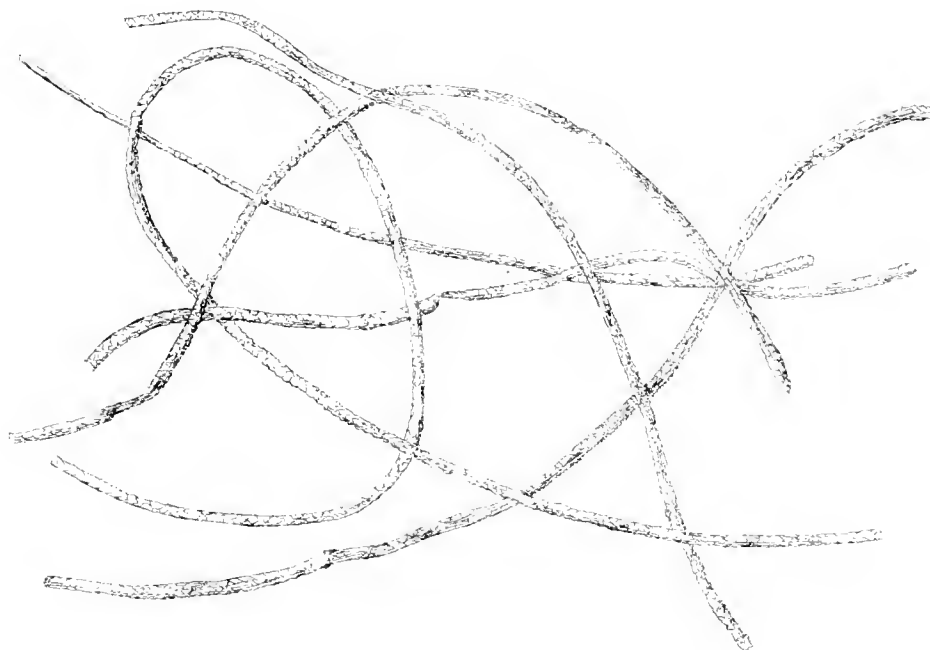


COARSE WOOL



COUNTRY WOOL  
(Land Wolle)

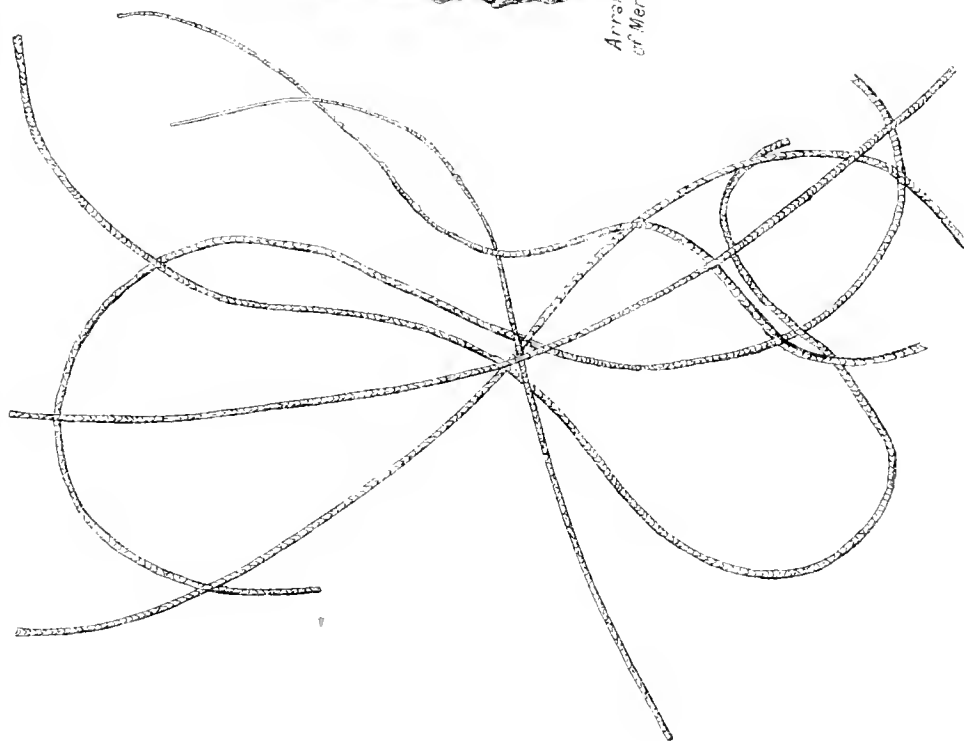




CROSS-BRED WOOL



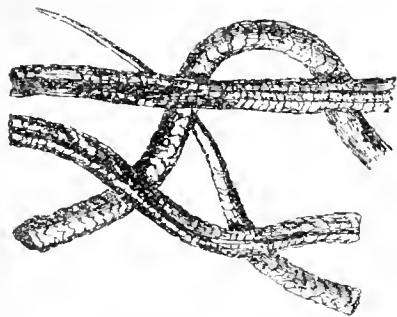
Arrangement  
of Merino Scales



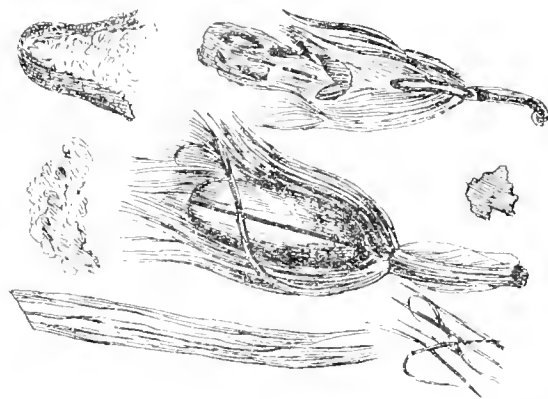
MERINO WOOL







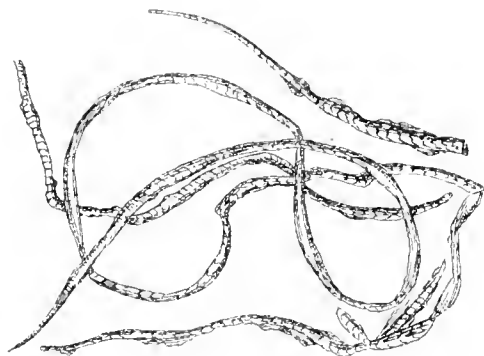
ALPACA WOOL.



Burrs & Seeds from Port Philip Wool



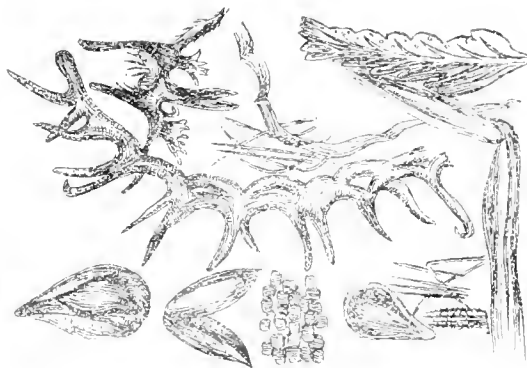
LEICESTER WOOL.



GREASY PORT PHILIP WOOL.

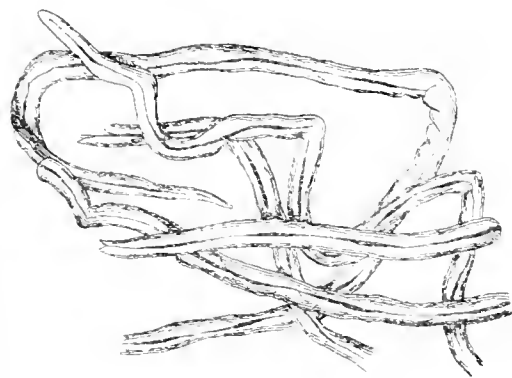


MERINO WOOL.

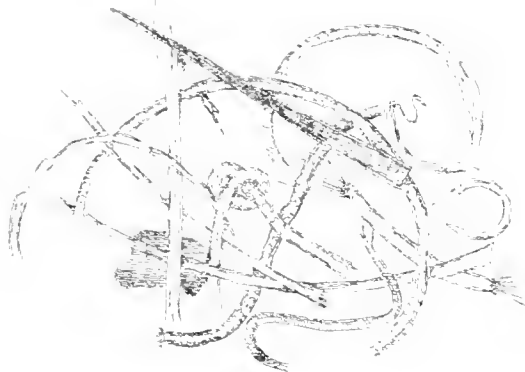


Burrs & Seeds from Port Philip Wool

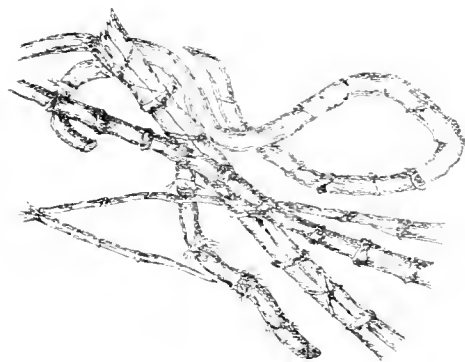




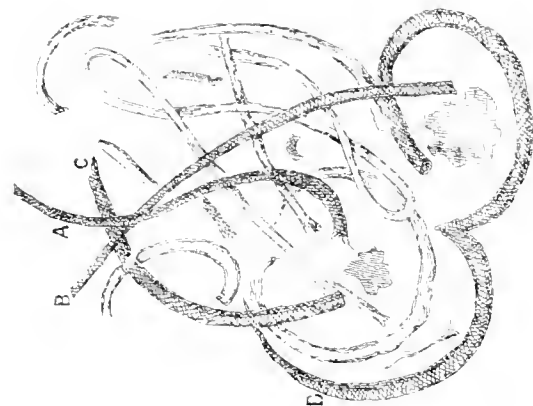
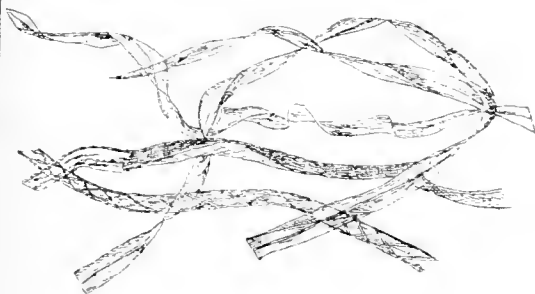
CHINA GR.  
(Ends of Fibres.)



FLAX.



COTTON.



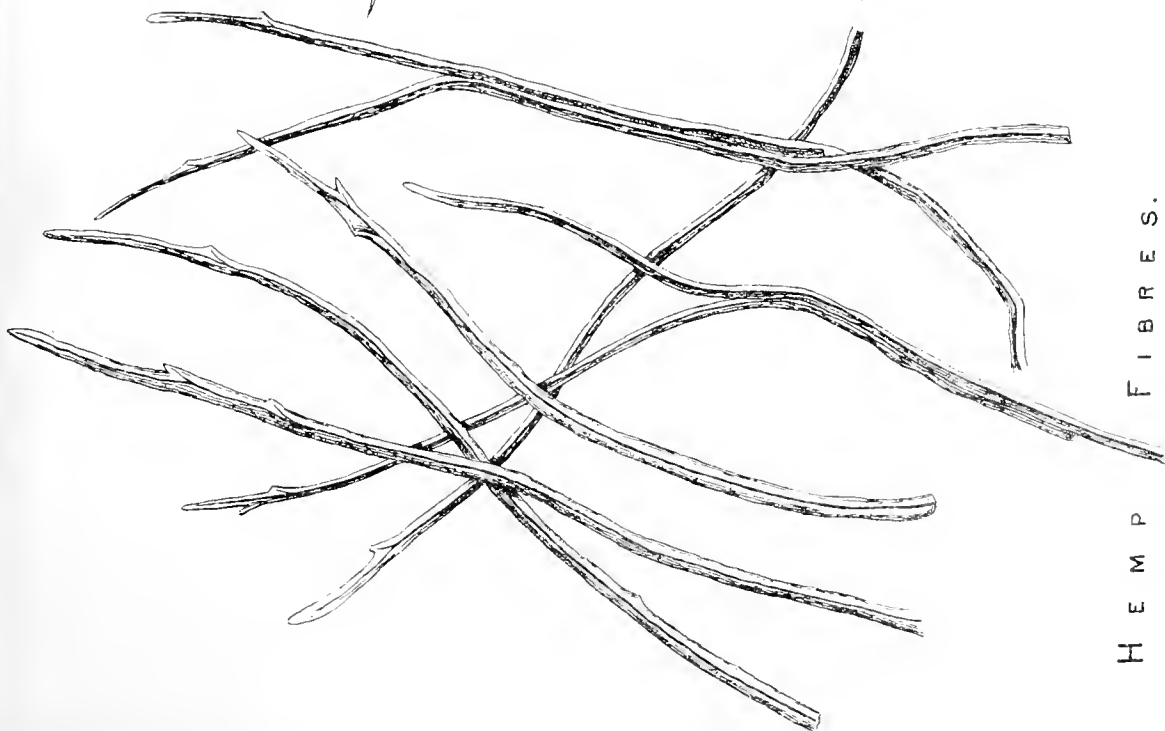
SHODDY A D U L T E R A T I O N S.

The fibres A B C D are dyed various dark shades.





COTTON.



HEMP FIBRES.





SILK FROM COCOONS  
*(The double fibres are separated)*

SILK





afterwards. Burnell, in his work on limes, mentions "that General Treussart's experiments upon artificial hydraulic limes would lead to the belief that their quality would be much improved if they were prepared with water in which soda or potash had been dissolved. The subsequent researches of M. Kuhlmann appear to confirm General Treussart's views upon this subject. Even so far back as 1841 M. Kuhlmann recorded the result of some experiments which induced him to propound the opinion that in the cement stones the alkalis served as carriers in the combination between the silica and the lime."

Sulphides are found in all cements to a limited extent. Where it occurs in quantities exceeding 0.5 per cent. of calcium sulphide it may be generally put down to the draught of the kiln being choked, and is due to the reduction of the sulphates. The clinker produced where the draught of the kiln has been obstructed will be found to be of a blue colour, quite different to the bronze appearance that good clinker presents. This colour in the first-mentioned clinker (which contains sulphides also) is due to the presence of a ferrosilicate of iron which gradually oxidises to the detriment of the cement. The ferrosilicate of iron, being a more stable compound, remains unchanged. During the manufacture of cement from substances containing the calcic sulphate in large quantities it will be found that the silicate of alumina (of the clay) will have decomposed the calcic sulphate to a great extent, forming the double silicate of alumina and calcium (which is cement); and even in the presence to the extent of the calcic sulphate mentioned, very little sulphide will be found in the cement if plenty of air has been admitted during the calcining operation. The cracking or disintegration of cements cannot always be put down to its containing sulphur salts, but in many instances it is due to careless working. A perfect mixing of the raw materials is indispensable; the ultimate result almost entirely depends upon this operation. The difference there is in nearly all the various cements made may be attributed to a very great extent to the laxity of the operation named. Of course the complete calcination or perfect decomposition of the mixed chalk and clay must also be taken into consideration.

During the present method of continuous mixing (carried out at all those works where cement is manufactured from chalk and clay), such a regular and consistent quality of cement cannot be expected, for in most instances the mixing is made entirely by guess-work. A certain number of barrows of chalk and mud are emptied into the mixer, and replaces a corresponding bulk of mixed material that overflows on the addition of a fresh quantity to the mixer. In some instances this overflow is tested from time to time by means of a calcimeter, which estimates the carbonic acid, the amount of chalk in the mixture being calculated therefrom. Errors are apt to be made by this process of testing by overheating the slurry in drying it preparatory to testing. Some of the  $\text{CO}_2$  would be expelled. The state of the atmosphere has also to be taken into account.

In no instance is it possible for a man, however experienced he may be, to exactly gauge the mixture of raw materials without testing, when we consider that the moisture in the mud varies in every lot used. Some of it may be taken direct from the boat or bed of clay, or it may have been deposited on the ground for some time, when either wet or dry weather would make a considerable alteration in the amount of moisture it would contain. The same condition would also apply to the chalk, but in a less marked degree.

In the case of cement made from comparatively dry materials, and where the mixing or blending is effected by a dry process, a more uniform control can be exercised, and consequently, as in cement made from the Lias formation, the quality is more regular, and can only be accounted for by the manner in which a perfect mixture of the materials is produced. By an intermittent system of mixing chalk and mud or other materials containing lime, and by a regular system of weighing and testing, a state of perfection in the resultant product could be obtained.

Manufacturers of chemicals know the importance of having the materials they are working upon in proper proportions in order to obtain as perfect a decomposition as possible. The same thing applies to cement, but in a lesser

degree. The difference in the test of the latter (that is, the tensile strain), although appreciable, would not be considered so long as the strain came up to the standard test, which is 1,000 lb. on  $2\frac{1}{2}$  in. sectional area in seven days. Of course, it is quite clear that if, by careful working, the cement could have been made to produce a higher strain by 25 per cent., there would be a decided advantage in it.

In the first instance, for the benefit of those persons who may not be acquainted with the manufacture of cement, I will give the outlines of the process as now generally employed. Cement can be made from any substances containing lime or carbonate of lime in such quantities that will produce after calcination a material having in its composition about 60 per cent. of calcium oxide. At the same time it is essential that the lime-bearing substance should be as free as possible from soluble salts or calcic sulphate. Chalk or limestone is the material that is now chiefly used, but any other form of lime, such as shells or corals, will answer equally as well, the essential point being to have the material naturally in as finely a divided condition as possible, as in the case of chalk, otherwise the grinding of limestone, or the other forms of carbonate of lime mentioned, becomes a rather costly operation. The next process is to incorporate this ground or finely divided lime with a requisite quantity of silicate of alumina either in the form of clay or shale; this is arrived at by the wet process by mixing it in a wash mill, which is a circular trough about 15 ft. diameter, in which a series of harrows are caused to revolve by suitable machinery. The mixture is made of a consistency to admit of its being pumped on to the drying beds, or elevated and allowed to flow on the beds by gravitation. After the mixed product has been dried either by the waste heat from the kilns or, in the case of separate drying beds the fire is caused to pass under iron plates, the dried material is conveyed to the kilns to be calcined. This operation is performed by placing alternate layers of coke and the dried mixture of carbonates of lime and clay in the kilns until they are fully charged, taking care in the first instance to have a sufficient quantity of wood and coke laid on the bars of the kiln to start the fire after the eyes or charging places of the kiln are built up. The charge is then allowed to burn itself out, which it generally does in about three or four days. The resulting product is a hard vitreous clinker, which is then ground by means of suitable grinding machinery and produces the well-known Portland cement. The grinding of this clinker is one of the greatest items of expense in cement-making, the wear and tear on the machinery being very great.

You will now be able to compare the working of the present process of cement-making with the one I shall now describe.

In the first place, in the mixing of the lime or chalk with the clay, it is requisite that as small a quantity of water be used as possible compatible with a perfect mixture of the material. After ascertaining that the proper proportions of raw materials have been used, the slurry is caused to pass through a cylindrical mixer having a series of revolving arms placed therein, or a conveyor would answer the same purpose. During its passage through this mixer, slack, small coke, or breeze is added, and the mixed compound is deposited at the exit end of conveyor or mixer (about the consistency of mortar) on to a band or conveyor that removes it to a place alongside the kilns (the latter being generally built in a line). A guide is placed over the band opposite each kiln to discharge the material as it is required. In commencing the calcining operation a quantity of coke is placed on the bars of the kiln; fire is applied in the usual way, and as soon as there is sufficient heat the mixture above mentioned is placed round it, and gradually covers the coke fire. This charging is continued at irregular intervals, care being taken to place the charge at the point where the flames are breaking through the mass and where it appears the most active. Should the draught be impeded, an iron rod, having a sharp point, is passed through the crust of the burning mass, in order to allow the heat from below to pass through it; this process is continued until the kiln is fully charged. These kilns have been worked continuously, but it has been found better to allow them to burn out in the ordinary way. By so doing, a further

quantity of dried slurry may be decomposed by the heat evolved from the decomposing mixture already in the kiln.

Upon drawing the kiln the clinker will be found of very uniform quality if the operations of charging have been carried out properly. The source of heat having been embodied in the slurry ensures a perfect calcination and obviates the tendency to slag exhibited in most of the usual form of cement clinker. Generally the outside of large pieces present this feature, and at the same time on breaking them the interior of some will be found to be only partly calcined.

The clinker is also in such a friable condition, owing to the product of combustion being compelled to pass through it, that the grinding of the material is reduced to a minimum. At the works I saw in operation, ordinary perforated clay pans were being used with comparatively light edge runners. Millstones are not used in the operation at all, yet the finished cement is in as fine a condition as that produced by any other process of grinding, and is quite as fine as is required. It will be found that the process just mentioned reduces the initial outlay in the erection of works very materially; it does away with the construction of costly kilns and drying beds, and also effects a saving in steam power, as the heavy forms of machinery now in use are not required to grind the clinker mentioned.

A further saving is effected by the use of slack instead of coke (only a very small quantity being used in the calcination of cement by this process). It will admit of works being erected that are far from this source of supply of that article. The kilns used for this process are of the simplest construction; any of the old-fashioned dome kilns now in use will answer the purpose, but as a rule they are rather small, and consequently the operation of calcining is rendered slow.

The finished cement produced by the above process is in every way equal to that made on any other plan.

#### DISCUSSION.

The CHAIRMAN (Dr. HURTER), in inviting discussion, said that he understood that the great difference between the process described and the old one was that in this the fuel was mixed with the slurry and the mixture charged as in blast furnaces, the difference in the two products being that the one was much easier to grind than the other. That would doubtless be of interest to cement manufacturers and to alkali manufacturers who hoped to become cement manufacturers, therefore some questions would doubtless arise.

Mr. E. CAREY said that Mr. Rigby had given them results of experiments with his cement, and that the tensile strength was something like 700 lb. per square inch as compared with 400 or 500 lb. per square inch of the ordinary cement. He would ask Mr. Rigby if he could give them any instances of his cement having been tried under water, and how it appeared at the end of two or three years, trial. The experiments mentioned, he understood, took place in the open air. He (Mr. Carey) had been informed by a civil engineer that some Portland cements had the unfortunate habit of becoming what he called "rotten," undergoing some chemical change which had destroyed their tenacity and rendered them in course of time valueless, the reason being, so he understood from the engineer, that the cement contained an extremely small percentage of sulphide. If the lime from the Chance process contained any sulphide, he would ask Mr. Rigby if he anticipated any difficulty from that cause. Mr. Rigby had also stated that it was generally understood that the presence of a small quantity of alkali was necessary in order to make a good and durable cement. Could Mr. Rigby inform them in what form these alkalis exist in the cement? As far as he could gather from the paper, his method appeared to promise a considerable improvement on the old one, and he hoped it would prove to be so.

Mr. W. P. THOMPSON said that some years ago he had heard of certain so-called "selenitic cements," in which there was a large amount of sulphate of lime. Did Mr. Rigby know anything about that kind of cement, and whether they failed in practice after extended use? Also whether a

small amount of cement mixed with a large amount of plaster of Paris would crack up or do any harm, or whether it would not actually improve the plaster of Paris? He would also like to know whether the works referred to by Mr. Rigby were abroad or in England?

Mr. RIGBY: In England.

Mr. J. W. MACDONALD said he would be inclined to question the engineer's opinion, quoted by Mr. Carey, that cement with even a small quantity of sulphide of calcium was of very little value, seeing that all cements (even the best London cement), contained more or less sulphides, produced by the reduction in the kilns of a portion of the sulphates contained in the clay and chalk. If sulphides were really harmful, he would very much like to know what amount it was unwise to exceed. Mr. Rigby did not estimate the sulphides; possibly they might also have something to do with the weakness of some of the cements he had examined. This new way of burning cement was said to enable them to use slack instead of the more expensive fuel, coke. It seemed that if sulphates and sulphides were so harmful, it was very dangerous to introduce slack, which usually contained much sulphur and its compounds. Possibly it might be right where the ingredients were pure, but such ingredients as Chance's lime mud already contained a great deal of sulphur, so that the mixing of slack with the "slurry" would produce a greater quantity of sulphur compounds in the cement. He thought that where the ingredients were pure, impure coal could be used, but if they had bad stuff to start with they should use the purest coke possible.

Mr. JAMES FORT asked whether a trial had been made for the making of cement from the spent lime of gas-works, if so, what results were obtained? The quality of cement produced by Mr. Rigby appeared rather poor, and not adapted for general use; but as it was from a waste product which had been brought to some utility, a considerable amount of business might be done by employing it in the manufacture of concrete paving. Concrete "flags" were used in several large towns, particularly in Warrington, where the slag from the destructors was ground up, mixed with common cement and formed into flags, &c. The Corporation thus got rid of the refuse from the ash pits and dust heaps.

Dr. HURTER asked whether  $4\frac{1}{2}$  per cent. of sulphate of lime mixed with the finished cement would not be much more harmful than an equal amount of sulphate of lime contained in the raw materials and intimately mixed therewith. Also whether the waste limes were always sufficiently free from magnesia, since that substance had been found to interfere with the resistance of the cement to the action of sea-water. Also whether lime mud from Chance's process must of necessity undergo a process of purification, and if the profits on the manufacture of cement were sufficient to cover the costs of such purification?

Mr. RIGBY, in replying, said, in regard to trying the cement under water, the briquettes when made for testing were always placed in water for six days. The cement had been used by contractors and corporations for gas-holder reservoirs, quay walls, and for all purposes where cement was required. As regards the presence of alkali in ordinary London cement, he thought it existed as aluminate or silicate of soda. Selenitic cement could scarcely be called a cement. It was a plaster, and was made by mixing borax or alum with plaster of Paris in certain proportions, with subsequent heating and grinding. The sulphate of lime sufficiently explained the disintegration of cement; he did not think it was sulphite in London cement. In London cement (the average of two samples) he found 0.20 per cent. of sulphide, in cement made by him 0.35 per cent., and in another sample of his 0.19 per cent., so that so small a quantity of sulphide could not possibly affect the cement. When the impure muds were properly treated the sulphates and sulphides could be eliminated. Sulphur in the sulphide of iron was carried away in the calcination as sulphurous acid.

In using Chance mud which had been made for some time the sulphide of iron was gradually oxidised into

sulphate, which in its turn was decomposed, forming sulphate of lime. Chance's waste should always be treated freshly to extract the sulphur. He did not think the use of slack instead of coke would much affect it or put any more sulphate or sulphite in except a very inferior fuel were used.

In the ordinary way of drying the slurry they took the hot gases over long beds, and more sulphurous acid was evolved from burning coke by carrying the gases that way than by putting the cement under the burning mass. On the other hand, he pointed out that cinders could be used, if washed, instead of slack. Breeze could also be used if thought necessary.

It would not be a difficult matter to purify lime from gas-works by carbonating it in the usual way. By passing carbonic acid through the gas lime the sulphide would be eliminated as sulphuretted hydrogen. The carbonate of lime could then be washed and treated in the usual way and would make good cement. He did not think it had been purified.

With regard to the action of sea-water on the cement, he thought he had mentioned that it had stood the trial, but it had not been left in for a long period. Cement had been sold to steam-tug owners who were perfectly satisfied with it.

As regards magnesia in cement, he had read that the action of sea-water was detrimental, but in New York cement which contained 10 per cent. of magnesia was made, but he believed it contained more. How far sea-water would affect it he could not say, but he believed it would have some effect.

If Chance mud were treated direct, the extra cost would not much affect the cement profits. But it would not stand much handling. It was a simple matter to purify Chance's mud. Two samples out of six he had made direct without purification at all turned out satisfactory.

He could scarcely say whether if a small amount of cement were mixed with plaster or scintic cement it would do good or harm. If they used a large quantity of sulphate of lime it would cover it, and it would be exactly as if it were not there. If they used equal quantities it would fall to pieces in water.

Science and Art Department might exercise over those local authorities who adopted the Act. Under these circumstances the Committee thought it advisable to invite Mr. Mather to be present at this meeting, so that he might be able to fully explain the application of the Act, as probably no one could speak with greater authority on this subject. Unfortunately Mr. Mather had been prevented from attending, but he had sent the following letter, which very clearly and conclusively set forth his views on the Act. Mr. Wm. Mather, M.P., wrote:—

"The apprehension of the Science and Art Department becoming master of the situation by the 'Technical Instruction Act' is obviously entertained by all the opponents to the proposal for levying the rate. This apprehension is absolutely groundless.

"The object of the Act is to enable local authorities to establish or aid schools of science or art, with special branches of instruction adapted to the requirements of their respective districts; and the local control of such schools is as safely guarded under the Act against interference in management on the part of the South Kensington Department as municipal control is guarded against interference of the Local Government Board.

"The 'science and art' classes hitherto established have derived their income almost entirely from the grants of South Kensington and school fees. The grants are bestowed for results of certain examinations, the character of which I have never approved. The system of examination has been accommodated to *class* instruction because the Department has mainly encouraged the establishing of classes all over the country, rather than the promotion of practical *schools* of science and art, in fewer centres, fully equipped with apparatus and appliances for experimental and laboratory work. But where public subscriptions, or individual generosity, have enabled the managers of such classes to add the premises and appliances necessary for sound and thorough training—as, for instance, in the case of the Manchester Mechanics' Institute, formerly consisting of science classes, now a technical school—the Department has never exercised the slightest control though continuing its grants for certain results.

"In like manner the science school of my firm has always received an annual grant from the Department for certain results, but the teaching is adapted beyond this to the requirements of our apprentices, at our own cost, and managed in the best way we can conceive of without reference to the Department.

"Again, the Birmingham School of Art receives a large grant from the Department, but it is a municipal school maintained by the Corporation, and is controlled entirely by the council on a system they deem best. These illustrations show that the obtaining of a grant does not, even at the present time, restrict the operations of a school to the subjects or methods contained in the directory of the Science and Art Department. An efficient and thoroughly-equipped school embraces, of necessity, all the Department requires, to qualify it for the Parliamentary grant, without adapting its teaching to any method recommended by the Department.

"If there are no other means at the back of a school but the Department grant and low fees, of course the teaching will be feeble and unsound and the effect thereof transient. This, in my opinion, is the condition of most of the science and art classes throughout the country. But the responsibility for this state of things no longer rests with the Department. The Technical Instruction Act, though incomplete in its range, at least enables every community throughout the country to supply efficient science and art schools, or to aid such as exist, for its industrial population.

"The unstable support given to the few efficient schools we now possess, dependent on subscriptions or individual generosity, ought to be immediately supplemented by steady and substantial aid from the local authority. The Parliamentary grant will be bestowed as heretofore, though the Act provides (clause 3), and very properly, that the Science and Art Department may alter its conditions. The sooner a change is made the better, for the grants are now given for results of a most unsatisfactory kind. Any change must necessarily be in the direction of encouraging more

## Manchester Section.

Chairman: Ivan Levinstein.

Vice-Chairman: Edw. Schunck.

Committee:

J. Angell.

G. H. Bailey.

R. F. Carpenter.

G. E. Davis.

H. Grimshaw.

Harold B. Dixon.

J. Grossmann.

P. Hart.

A. Liebmann.

Sir H. E. Roscoe, M.P.

C. Truby.

D. Watson.

Hon. Local Secretary:

J. Carter Bell, Bankfield, The Cliff, Higher Broughton Manchester.

SESSION 1890.

Notices of Papers and Communications for the Meetings to be sent to the Local Secretary.

Meeting held at Victoria Hotel, Manchester, on  
Tuesday, 4th March 1890.

MR. IVAN LEVINSTEIN IN THE CHAIR.

## DISCUSSION ON THE TECHNICAL INSTRUCTION ACT, 1889.

THE CHAIRMAN said that at the last meeting a discussion took place on the Technical Instruction Act, and that some objections were raised by members to the control which the

sound and practical instruction, such as the rate-aided schools can well afford to supply.

"Now, let us look at the Act with a view of finding some support for the assertion that the Science and Art Department will be 'master of the situation' in the future.

"The sub-section (*d.*) of clause 1 provides for aid to be given to schools for the time being in receipt of grants from the Science and Art Department. This is a guarantee to the local authority that the claims of such schools have at least some title to be considered. But the amount of aid depends entirely on the local authority. It may decline to entertain the application on various grounds. The 'requirements of the district' are to be kept in view; the quality of instruction, as compared with other such schools, must be regarded, and questions of management adjusted satisfactorily to the local authority.

"A further sub-section (*f.*) of clause 1 provides that differences of opinion may be referred to the Department of Science and Art, should they arise between the managers of any school and the local authority. The determining of the question is limited to giving an opinion which the Department has no power whatever to enforce. The function of the Department will be purely consultative on questions of this kind, and probably seldom used.

"In these two instances where the Science and Art Department is mentioned in the Act there is no ground whatever for apprehension that local control will be interfered with.

"In clause 3 the Department is again named, but I have already dealt with this instance. It does not affect local control in the least degree.

"In clause 8 we have a broad definition of technical instruction and manual training. The clause is drawn to protect a local authority against being surcharged by the auditor for having bestowed aid out of the rate on a school, in which, according to his view, subjects were being taught not included in the terms of the Act. To prevent all possibility of dispute, or any perplexity to the local authority or its legal adviser, a complete list of the subjects of instruction which a local authority may desire to aid—beyond those mentioned in the directory of the Science and Art Department—must be sent to the Department. The subjects named in the directory are *ipso facto* included in the terms of the Act, and the auditor cannot question the right to aid them. It is obvious that this obligation on the part of the local authority is expressly imposed for its own protection and assistance. The only possible control of the Department will be limited to a rejection of any subject in the list sent up which did not come within the scope of the Act. The power to give an authoritative interpretation of the scope of the Act must of course rest somewhere. A court of law is the only alternative, unless a department of the State has this power.

"Now, it would be altogether misleading and unreasonable to assert that a department of the State, in performing this function, necessarily becomes 'master of the situation.' It will simply interpret what may be done under the Technical Instruction Act, as the Local Government Board interprets what a local authority may do under the Municipal Acts. Were some authority not created by the Act which could define, for instance, what character of workshop practice and the use of tools was included in the term 'technical instruction,' it might be that an auditor would hold that a certain trade was being taught in some rate-aided school. It is most important that the trades unions throughout the country shall have ample security that the Act will not be used in any way other than for strictly educational purposes. This security is fully given by the power vested in the Department of rejecting any subject in the list submitted by a local authority which is outside the scope of the Act. In the exercise of this function the Department has no power whatever to dictate what shall be taught under the Act or to inspect the methods of teaching. All or any subjects covered by the Act may be aided by a local authority according to the requirements of the industries of the locality without let or hindrance. The Department can only assist local interests when consulted, and cannot thwart them at any time under any pretext whatever. The initial inconvenience

or delay in applying to the Department to lay on the table of the House a list of subjects which a local authority intends and determines to encourage the teaching of out of the rate, is but a slight matter, and once done the Department may never be appealed to again.

"Suffice it to say in conclusion—and I do so positively without fear of authoritative contradiction—that from every point of view the most complete control will ever rest with the local authorities and local managers over all schools for technical instruction aided by the local rate under the Technical Instruction Act. All beginnings are more or less difficult. This Act will encounter some difficulties no doubt, but I am sure they will not be serious if not created by unreasonable opponents. In the course of even a short time, amendments may be suggested from experience which Parliament will readily sanction. Meanwhile we ought to use 'whatever of good the present brings,' and so make the best and surest preparations for better things to come."

The CHAIRMAN went on to say that the Act appeared to him to be the practical outcome of the desire of those who were in favour of more extended and efficient technical instruction. Schools in which technical subjects and scientific principles pertaining to industries were taught, never could be self-supporting for the simple reason that the fees would have to be fixed at such a high rate as to make them prohibitory to the poorer students, and thus defeat the primary object, viz., the extension of the teaching of technical subjects to the masses. The primary object of the Act was to provide a fixed monetary contribution out of the rates for the purpose of technical instruction. Whatever its minor faults might be, members could hardly object to the principle of the Act, and no doubt, as necessity arose, any shortcomings which might come to light would be amended.

Mr. CARPENTER said they were all aware that very recently a strong body of memorialists, representing all the most important educational institutions in the city, had waited upon the town council of Manchester, praying that it would adopt the provisions of the Technical Instruction Act, 1889, and he hoped that his remarks would not be out of order with the resolution to be submitted to that meeting requesting that a memorial should also be presented from the Manchester Section of Chemical Industry to the same effect. To Mr. Mather they were indebted not only for his share in the Bill as it stood, but at various times for his earnest advocacy of its adoption, and for his constant endeavours to remove the misconceptions that still existed in the minds of many as to the real powers conferred by the Act. The history of some of these misconceptions in Manchester it might be interesting and instructive to trace. To begin with, when the Bill was before Parliament in August last year, the deputy town clerk called the attention of the Parliamentary Committee of the Corporation to an amendment proposed by Mr. Mather, making it compulsory for local authorities to contribute out of the rates, with the result that the Corporation petitioned against the proposed amendment. Mr. Mather, however, withdrew his amendment from the paper before committal of the Bill, and the whole action of the Corporation Parliamentary Committee therefore fell through. This might cause people to think that because the Corporation had petitioned against the Bill there must be some objection to it, whereas the only objection was simply as to whether they "*may*" or "*must*" contribute out of the rates, the Bill as drawn by the Government having been always an "enabling" Bill. Early in September the National Education Association held a meeting, which was addressed by the Hon. Lyulph Stanley, who unfortunately made a very severe attack upon the Bill, stating, *inter alia*, that it assisted denominationalism, and doing all in his power to raise the denominational bogey. Mr. Mather was not able at the time to reply, but on September 13th he did reply, and being at some distance from home he was not aware of the action which the Corporation had taken in the matter. He (Mr. Carpenter), on September 12th, explained the real state of the case as above detailed. On November 27th a conference was held in Manchester at which, in addition to the explanatory address of Mr. Mather, Sir Henry Roscoe read an

address showing how far the Act had been put in operation, and General Donnelly was present from the Science and Art Department, South Kensington, but he certainly was not too communicative in his replies to the questions addressed to him, and so not only failed to remove previously existing misconceptions, but unfortunately was the innocent cause of establishing fresh ones in the minds of some of those who were present. The next feature that presented itself was the memorial of the various bodies who hoped to obtain grants under the Act, led by Mr. Oliver Heywood, on behalf of the Manchester Technical School; and looking over the Manchester papers he was surprised to see a letter written by a Bradford gentleman, in which town the Bradford Technical School had been applying for a grant, in which the writer seemed to be labouring under the same misapprehension as to the powers of control exercised by the local authorities under the Act, as he stated that the Bradford Corporation had allowed the memorial to lie on the council table with contempt, and he hoped the Manchester Council would assume the same attitude as had been shown to the Bradford memorialists. He (Mr. Carpenter) hoped this spirit did not prevail in Manchester. Another feature which he desired to bring before them was the difficulty which evidently existed with regard to school boards. He knew that an opinion prevailed among some of the members of the Manchester School Board that because the Act did not give them everything—that therefore it was badly drawn, and should not have been passed. He thought that those school boards which had not made use of the powers which they already possessed should not cry out. Take, as an instance of what can be done in technical instruction, the action of the Birmingham School Board in establishing, in conjunction with the Science and Art Department, South Kensington, their advanced classes. The strong point about the Birmingham system was that the pupils in these technical classes, who had already gone through the higher standards, were obliged to enter for not less than two years, and optionally for three, allowing ample time for sound and methodical instruction, and he thought that was the basis on which all technical instruction should proceed.

Dr. BAILEY said, as he had introduced this question at the last meeting, perhaps it was due to those present that he should say something about their position in the matter. A sub-committee was appointed last month to draw up a memorial for presentation to the City Council urging them to support the Bill, and it had been his intention to have brought this before the last meeting of the Section, but being informed on good authority that the Science and Art Department would dictate subjects, appoint examiners, and practically control the working of the Act, he declined to have anything further to do with the memorial. However, Mr. Mather now told them in his letter that the apprehension of the Department becoming masters of the situation by the Act was absolutely groundless, as the Department would simply act as a board of reference. Under these circumstances it seemed to him to be very desirable that they should memorialise the City Council in the strongest manner possible, urging them to adopt the Bill. They had in Manchester a number of schools engaged in technical instruction, and this was also encouraged, to some extent, by the City Guilds, but in neither case was adequate support given to such teaching. They, as a Society engaged in the practical application of science, recognised that the subjects taught by the Science and Art Department did not, in so far as referred to the elementary stage (and a very large majority never got past this), come up to such a standard as would enable a student even to follow up those studies which would be useful to him in his special industry, and it was in this respect they felt that the Department, whose system was such as to encourage the most superficial knowledge in a multitude of subjects rather than sound knowledge in two or three, was not the proper body to carry out the provisions of the Bill. Whatever advantages or disadvantages they might suffer under with regard to Germany or other countries, there could be no doubt about the fact that such classes as were held in those countries were attended by those actually engaged in industrial pursuits, whereas, in this country a very large percentage of those

who attended science classes were school boys and persons who were unconnected with the industries. Yet there must be, in Manchester, 50,000 people engaged in industries who would derive benefit from a knowledge of those scientific principles and by a further knowledge of the subjects connected with their particular industries. But from inquiries he had made, the number who were receiving scientific instruction did not exceed 5 per cent. of those so engaged. Now, these were facts which ought to appeal to everyone, and especially to the City Council, and, unless such a matter was dealt with by those immediately interested, it is not likely to be dealt with by any central organisation, and for these reasons they were fully convinced that the City Council had a great work before them, if they could efficiently carry it out. What was wanted was a bond of union between the civic authorities and manufacturers in order to make technical education in this district a practical success, and for this reason he hoped the resolution he had to propose would be well supported.

Mr. GRIMSHAW said that as there were a number of gentlemen present who were not members of the Society, but who were directly interested in the teaching of technical subjects, he would offer a few remarks as to the position which the Society held in the matter. He believed he was right in stating that the point which they had wished Mr. Mather to enlighten them upon was:—Whether, under the Technical Instruction Act of 1889, education could be given in a way not prescribed by the syllabus of the Science and Art Department and in subjects not contained in that syllabus? If such was not the case, then he believed that the Manchester Section of the Society would not concern itself in any way with the Act, or waste any time in considering its provisions. The Section only professed to speak for the chemical industries, as they did not know the exact form of technical instruction required by the cotton, iron, coal, and engineering trades, or others, except at the points in which those trades touched the manufacturing of chemicals. With regard to technical instruction having for its object the elevation and improvement of chemical industries, they had an opinion in which he believed they were inclined to be rather bigoted. At a meeting of this Section in February 1889, before the passing of the Act, a pretty general opinion was expressed that what they wanted to see erected was a system or a set of institutions by means of which they could have professors of industrial chemistry, and in addition highly-educated masters and owners of chemical works, and a superior class of technically-educated managers and foremen. With regard to the workmen engaged in chemical industries, they considered that a good elementary education, with due attention to scientific subjects as part of the curriculum, would render them fit to do their best as workers in chemical yards when they had had a few years under the properly equipped foremen, managers and masters, whom they wished to create. Not that the chemical technologist was non-existent at the time, but where was the professor of technical chemistry? Mr. Mather would see that if this position was correct, the chief concern of this Section was not with elementary education and what could be taught under the South Kensington syllabus, but “Whether it was impossible for new authorities to use new powers to commence a system of scientific technical education outside and above the Science and Art Department curriculum.” Now, when this Act had got into operation, he hoped there would be more inspection and less examination. They also wanted higher elementary and good secondary schools, and technical schools, and the production of better teachers. He believed the City of London Guilds, more particularly the Finsbury School and Central Institute, did more fully approach the realisation of a proper scheme of education than the South Kensington Department. Instruction under the Act should be more practical and less professional; for true technical instruction resided somewhere between the secondary schools—of which this country was almost devoid—and the colleges. What was wanted was something in place of the mechanics’ institute, where sound technical instruction at a small rate of cost could be obtained. To-day they had the merest shadow of such places, and they felt curious to know whether the penny provided under the Act would be the shadow of the

money which would be provided by a far-seeing Government to accomplish such a nationally-important object. He knew the difficulties which Mr. Mather and Sir Henry Roscoe had had to deal with in the House of Commons. What was really wanted was home rule in technical education, so that each locality could deal with its own industries, and he was very glad that Mr. Mather had given them some hope of this being accomplished under the Act.

Mr. J. H. REYNOLDS, Secretary of the Manchester Technical School, said he was himself engaged in administering technical education in the city of Manchester, and no one knew better than he did how deficient were the means at the command of the technical school to fully accomplish the object its council had in view in establishing an institution where a thorough system of technical instruction could be carried out. The *1d.* in the *£* allowed by the Act would produce about 10,500*l.* a year, but the memorialists who recently approached the Corporation thought that a  $\frac{1}{2}$ *d.* rate would be sufficient to meet present demands. He fully concurred in the Chairman's remarks that no thorough system of technical instruction could be made self-supporting, as the expense of workshops and appliances would be too costly. He agreed with Dr. Bailey, that only about 2,500 students in the city and district took up the study of technical subjects with a view of improving their knowledge with regard to their respective trades, and that the bulk of the students attended the science and art classes simply for the purpose of extending their general knowledge. Only those practically engaged in the various industries would be able to say in what way and to what extent technical instruction benefited them; and for this reason he thought no such system could be usefully and wisely administered by the school board, which was elected for a very different purpose. He was of opinion, therefore, that the City Council, in association with a committee of commercial and manufacturing experts, could deal with the matter more efficiently. If the Act was to be of any value it must be administered by those who fully understood local requirements; but this could not be effected if the matter was left in the hands of a central authority in London, which could not be in touch with those mostly interested in carrying out the provisions of the Act. There was, however, no real ground for apprehension that the Science and Art Department would unduly interfere in the administration nor with the subjects dealt with under the Act.

Mr. J. S. THORNTON said there was a widespread feeling that under the Act the influence of the Science and Art Department would be confined to schools in art centres, and if that were so he agreed with Dr. Bailey and Mr. Grimshaw that that would be contrary to the meaning of the Act. Those gentlemen were very much convinced of the wisdom of the Science and Art Department, and their proposal seemed to be that a scheme should be adopted which would make the Act more widespread in its influence and increase its power. The Science and Art Department went in for many things, but not much of any, and in his opinion it was an Augean stable which required cleaning out. The question was:—How far would the adoption of the Act, that is to say, the extension of the science and art examinations, assist technical education in the direction they so much desired? The report of the Commissioners on Technical Education stated that in our staple industries we were still ahead of other countries, and that in not one of our special lines did we need much improvement; but the question was:—Would the Science and Art Department produce a supply of such chemists as our manufacturers of beetroot sugar required, and such as Germany possessed? The present system of examinations encouraged teachers to make money out of them, and this debased both teacher and pupil.

Mr. Wm. THOMSON wished to know whether there was any stipulation as to how the money raised by levying the rate was to be spent? He contended that it was quite impossible to teach people trades by any system of technical education. If they wished to learn a trade they must go into the workshops. Technical education ought to aim at teaching the fundamental principles involved in

all trades. He thought the Science and Art Department would be the proper authority to control these matters, as they would have more experience than would be likely to be found in any local body of men.

Dr. BAILEY then moved, and Mr. GRIMSHAW seconded, the following resolution, which was put to the meeting and carried unanimously:—

"That this meeting of the Manchester Section of the Society of Chemical Industry, having considered Mr. Mather's statement, expresses its hearty approval of the Technical Instruction Act, 1889, and desires the committee of the Section forthwith to prepare and arrange for the presentation of a memorial to the mayor and town council of Manchester, praying them to carry out the provisions of the Act."

## Newcastle Section.

Chairman: T. W. Stuart.

Vice-Chairman: P. P. Bedson.

Committee:

A. Allhusen.  
G. T. France.  
G. Gatheral.  
John Glover.  
T. W. Lovibond.  
John Pattinson.

H. R. Procter.  
B. S. Procter.  
W. W. Procter.  
W. L. Rennoldson.  
C. H. Ridsdale.  
J. E. Stead.

Hon. Local Secretary and Treasurer:  
Dr. J. T. Dunn, The School, Gateshead.

Notices of Papers and Communications for the Meetings to be sent to the Local Secretary.

Meeting held in the College of Science, Thursday,  
March 6th, 1890.

DR. BEDSON IN THE CHAIR.

### ON GANTLER'S METHOD OF ESTIMATING TANNIN.

BY HENRY R. PROCTER, F.C.S.

It must be admitted that a rapid and exact method of estimating tannin in dilute solutions is still a desideratum, and it was therefore with much interest and some hopefulness that I began experiments with the method recently published by F. Gantler (*Zeits. f. angew. Chem.* 1889, 20; *Jour. Soc. Dyers and Colourists*, V. 176), in which the determination is made with permanganate in a boiling solution. He employs a solution of permanganate of about 4 grms. per litre, acidifies 10 cc. of the tannin infusion with 10 cc. of dilute sulphuric acid (strength not stated), and adds the permanganate in small quantities to the nearly boiling solution, pausing between each addition till decolouration takes place. When in the end a precipitate of manganese hydrate forms which no longer disappears even on vigorous boiling, about 1 cc. of permanganate is added in excess, and the strong boiling is repeated several times, which causes the disappearance of the red colour, and the separation of a dense brown precipitate. This is then removed by the addition of a known quantity of oxalic acid, and the titration completed in the usual way without boiling, and the permanganate equivalent to the oxalic acid is deducted.



Gantler shows by a series of estimations that the result is not materially affected by the concentration of the permanganate employed or the strength or quantity of the tannin, and finds as the average result of his experiments that 1 gm. of pure tannin consumes 3.988 grms. of permanganate, while he states that the amount reduced by other tannins is not very different, but that on this point he is experimenting further.

My experiments have so far been confined to solutions of pure tannin and infusions of oak bark, and in the main bear out these results; but I find, as indeed was rather to be anticipated, that the amount of permanganate consumed is materially influenced by the excess of permanganate added, and the amount of boiling to which it is submitted. In this way I obtained with an infusion of oak bark results varying from 17.6 cc. to 19.8 cc. of permanganate, and it is probable that still higher figures would have been given by employing still greater excess, or repeating the boiling and addition of permanganate. Gantler assumes that he obtains a complete oxidation, but calculating from received formulae, 1 molecule of tannin,  $C_{12}H_{10}O_6$ , would require 24 atoms of O for complete oxidation to carbonic acid and water, and this is equivalent to 4.7 grms. of permanganate for 1 gm. of tannin. No possible theoretical changes in the formula would materially affect this result, which is in agreement with the observed percentages of ultimate analysis. It is therefore clear that towards the end of the reaction some product is formed which is very difficult of further oxidation, and it also seems probable that in presence of manganese sulphate, dilute solutions of permanganate on long-continued boiling are gradually decomposed with loss of oxygen. At any rate, if Gantler's method be applied to the oxidation of oxalic acid, results much in excess of the truth are obtained.

It is probable that by adherence to a rigid scheme of operation, concordant results might be obtained, but such results could have at best but a comparative value, and no greater claims to accuracy than those of the Löwenthal method, which, when a standard solution of pure tannin is treated in exactly the same way as the infusion under examination, gives fairly satisfactory and reliable results.

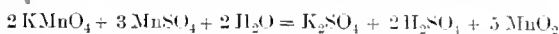
I have had no opportunity of consulting Gantler's paper in the original, but if the translation in the Journal of the Society of Dyers be a correct representation of his views, it is quite clear that he has not fully grasped the principle of the Löwenthal method as improved by von Schroeder. He speaks of part only of the tannin being oxidised in this method, but as I have explained in a previous paper read before this Society (this Journal, 1886, 79—82), the whole of the tannin present is oxidised, though not to its ultimate limit of carbonic acid and water, but to some intermediate products which have not been determined; and the office of the indigo is not merely that of an indicator, but of a regulator of the degree to which this oxidation is carried. I need not repeat details which have been fully explained before, but the gist of von Schroeder's improvement is that the solution under examination is directly compared with one of pure tannin titrated under exactly the same conditions, and therefore oxidised in the same way, and he has specially insisted on the point that such comparisons as Gantler makes of the absolute quantity of permanganate consumed are of no scientific or practical value. Much may be said against the special so-called "one cubic centimetre" method which von Schroeder adopted as his working process, but nothing against the sound system of direct comparison which he advocated. Gantler is also quite wrong in his assumption that the Löwenthal method, properly executed, does not give results very approximately agreeing with the gravimetric one, and seems quite to have overlooked the fact, well-known to all chemists who have examined the subject, that in dealing with tannins other than gallotannic acid it is necessary to employ reduction-factors dependent on the nature of the tannin in question, that of oak bark consuming approximately only two-thirds as much permanganate as gallotannic acid.

Gantler also states that by his method the amount of matter not tannin estimated is so small that for technical purposes it may generally be neglected. This is decidedly incorrect, as in four samples of oak bark which I examined by this method it averaged over 25 per cent. of the total,

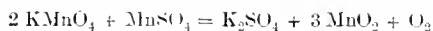
and it is to be borne in mind that while Löwenthal's method is practically unaffected by any gelatinous matter which may be dissolved from the hide powder used in removing tannin in order to estimate by difference, the same is by no means the case with Gantler's, and gelatine precipitation, which as a technical method with Löwenthal is rapid and satisfactory, is with Gantler not to be thought of. While I do not deny the possibility that a useful method may ultimately be based on Gantler's idea, I think I have said enough to prove that in its present condition it cannot supersede Löwenthal's.

#### DISCUSSION.

Dr. DUNN said that there was no doubt as to the evolution of free oxygen when permanganate was boiled with solutions of manganese salts in presence of acid, even with dilute solutions. The result varied with the proportions of the two. So long as the proportion of permanganate was less than that corresponding to the equation—



no free oxygen was given off, and the whole of the available oxygen of the permanganate was present in the precipitate of manganese dioxide; but as the proportion of permanganate increased, a gradually increasing amount of oxygen was given off, till the state of things represented by—



was reached, after which any excess of permanganate appeared to be unaffected.

Mr. PROCTER thought that perhaps the acid became sufficiently concentrated, during the boiling in Gantler's method, to act on the manganese dioxide formed, because in practice the amount of permanganate added was never so great as that in the first equation given by Dr. Dunn.

Dr. BEDSON inquired whether the actual products of the oxidation of tannin by permanganate had been investigated, as the amount of oxygen used, according to Mr. Procter, was always less than that needed for the complete conversion of the tannin into water and carbon dioxide. He suggested as a probability the formation of one of the hydroxybenzoic acids, themselves compounds of considerable stability.

Mr. PROCTER then drew attention to a curious case of corrosion of a glass bottle, which had been used to contain potassium carbonate solution, and in which this solution had stood without being used for some years. The whole upper part of the bottle, from the level of the surface of the liquid upwards, was found to be roughened and corroded, while the portion in contact with the solution was not so.

Dr. BEDSON then gave a demonstration of a gas analysis apparatus. The apparatus was practically that of Dittmar, with a modification in the mode of transferring the gas from the laboratory tube to the absorbing pipettes and back again. This consisted of an ordinary Hempel's pipette, provided with a three-way tap between the reagent bulb and the capillary tube. The capillary tube, the reagent bulb, and a small moveable mercury reservoir can thus be made to communicate with one another two at a time, so that the capillary tube can be filled with mercury, and the transfer of gas made in both directions without allowing any of the reagent to pass the tap and get into the capillary tube.

## A BRIEF HISTORY OF THE CHEMICAL MANURE INDUSTRY, WITH SPECIAL REFERENCE TO THE NORTH OF ENGLAND.

BY JOHN MORRISON, CHEMICAL ENGINEER, NEWCASTLE-ON-TYNE.

**General Features.**—Liebig, in his "Agricultural Chemistry," published in 1840, states that "perfect agriculture is the true foundation of all trade and industry," and "of the riches of States." In it he further prophesies that "a time will come when plants growing upon a field will be supplied with their appropriate manures prepared in chemical manufactories—when a plant will receive only such substances as actually serve it for food, just as at present a few grains of quinine are given to a patient afflicted with fever, instead of the ounce of wood which he was formerly compelled to swallow in addition." Liebig likewise in the same work makes the important declaration that "the most practical method of effecting the division of bones (*for manurial purposes*) is to pour over them, while in a state of fine powder, half their weight of dilute sulphuric acid, sprinkling the acid mixture before the plough."

**Origin.**—To the latter statement may be credited the origin of the artificial manure industry, though it should not be overlooked that so early as 1817 Sir James Murray had experimented with, and had proven the manurial value of soluble phosphates.

In the year 1838, when the British Association first met in Newcastle-on-Tyne, Messrs. Lawes and Gilbert began their agricultural researches. And in the same year Peruvian guano was first imported into England as a manurial curiosity, being sold at 20*l.* per ton.

Guano was at first, however, regarded by English agriculturists with considerable distrust, so that its use did not rapidly extend until in 1841 it was specially brought before the notice of the Royal Agricultural Society. After this introduction its sale rapidly increased, so that in the year 1863, when the British Association for the second time met on Tyneside, its annual British consumption had reached about 100,000 tons, its price having descended also to about 12*l.* per ton. Mr. Lawes, at Deptford, and Mr. Procter, at Bristol, began manufacturing chemical manures about the year 1843, the former having discovered in dissolved mineral phosphates an excellent phosphatic substitute for Liebig's dissolved bones. And as this was only about a year before Dr. Richardson commenced the business on the Tyne, the North of England was pretty well to the front in the race.

**Prejudices.**—The trade in those early days, however, was looked upon with considerable suspicion. Agriculture had been carried on for centuries without any adventitious aids, and what it had never hitherto seemed to want it was thought it could never hereafter possibly miss. The digestive organs of his own sheep and cattle formed the farmer's universal mixing machine, and the excreta of his live stock, with occasional doses of marl, lime, and seaweed when obtainable, comprised his sole manurial repertory. And being very conservative in his disposition, and very much wrapped up in his ancestral methods, he hated innovations with a perfect hatred, regarding in the light of a personal injury the advocacy of revolutionary agencies of which his respected forefathers had never so much as dreamed.

In 1863 the total British make of dissolved manures had reached about 200,000 tons, of which Mr. Lawes himself produced about a tenth. Indeed, so important had the English manure trade then become, that Liebig trembled, and solemnly declared that England was "robbing all other countries of their fertility;" that "in her eagerness for bones, she had turned up the battle fields of Leipzig, Waterloo, and the Crimea;" that "she annually removed from the shores of other countries to her own the manurial equivalent of three millions and a half of men;" that "like a vampire she sneaks the heart blood from nations without a thought of justice to them;" and that "it was impossible that such iniquitous interference with the Divine order of the world should escape its rightful punishment."

Liebig was not evidently a believer in international commerce.

**Mineral Phosphates.**—The introduction of prepared bone materials formed the dawn of the chemical manure trade, as the introduction of mineral phosphates formed its sunrise. Without the latter, chemical manuring must have been crippled almost in its infancy. Had not, indeed, dissolved bones quickly competed with the earlier guanos, the price of guano must have tremendously increased. And had not mineral phosphates risen to the relief of bones, the price also of bones must have rapidly advanced. When all were in use together, it became purely a question of the survival of the fittest. And now over 40 years since the contest for supremacy began, almost the youngest combatants seem the victors in the fight.

**Worthless Compounds.**—The chemistry of the early manure trade was not of a high order. Lots of people with a greater excess of assurance than of honesty ransacked almost the heavens, the earth, and the sea to discover refuse which, cheaply wrought into friable condition, they might ostentatiously palm off upon the British farmer as genuine manure. Not a few merchants seemed verily to believe that guano itself was specially created but as a flavouring to their sand, clay, or pulverised bricks. And the farmer was unfortunately then too ready to give an absurd price for scandalous commodities if only impregnated with a good solid stink. One manure, largely sold about the year 1856 at 12*l.* per ton, was for the most part apparently a mixture of burnt ore and nitre cake, and even years later than that, mixtures were widely disseminated which, though pleasantly remunerative to the seller, could but be of infinitesimal benefit to the consumer.

**Dissolved Bone Compounds.**—But the yearly increasing demand for chemical manures, and the corresponding increase in the value of bones, tended perhaps to one regrettable result. This was the introduction of materials other than simple bones and acid into manures retaining the original name of *Dissolved Bones*. The augmenting scarcity of bones of course sternly demanded some cheaper substitute. Consequently a class of manures rapidly came into use possessing certain well-defined mechanical characteristics, and a certain well-recognised chemical analysis. That the successful production of such manures has immensely benefited the farmer, there can be no question. And that consumers have been thoroughly satisfied as to the results from their use is proven by their widespread popularity, and the enormous annual demand for them. But that for five and twenty years they have been associated with a name which does not accurately describe them is certainly a pity.

Bones have, ever since the manure trade began, been in some sense regarded as the standard of phosphatic purity. And yet when calcined and sold as bone ash their purity is substantially inferior to that of various mineral phosphates now in extensive use. Phosphatically, indeed, it is difficult to see the difference between either bones, bone ash, or spent bone charcoal, and good mineral phosphates of similar analysis. And this especially when, as in most cases, the mineral is really a fossilized bone phosphate, and when, as is now usual, it is before treatment very finely ground—much more finely indeed than bone materials. Neither is it easy to perceive the difference—manurially considered—between two fertilisers of similar analysis, but solely produced in one case from bones and acid, and in the other compounded of bones, fish, or other guanos, animal matters, mineral phosphates, and acid. So far as the soluble is concerned there *can* be no difference, while as to the insoluble it is for the most part a question of mechanical division.

**Pure versus Impure Dissolved Bones.**—In the Merchandise Marks Act, 1887, chemical manures were not apparently contemplated, and how this Act will eventually deal with the above case of dissolved bones is by no means certain. Something, however, should speedily be done. At present it is utterly beyond the power of any commercial analyst to categorically certify the purity of many samples of professedly pure dissolved bones. A chemist may be able to

express an opinion as to the presence or absence of mineral phosphate in the insoluble portion, or generally as to the composition of the manure being solely *animal*. But to positively declare that any given sample is composed solely of bones and acid is beyond his powers. And his position is materially aggravated by the many varieties of local and foreign bones and meals now in daily use. This is very burdensome to the conscientious manufacturer (and surely he is in the majority), for the manures he designates and supplies as pure have to compete against others much more cheaply produced, but which are of similar appearance and analysis, and equally satisfactory as to purity to the certifying chemist, and are really fully as good, if not better, for the farmer. For lots of so-called pure dissolved bones are of an ammoniacal analysis so high that they could not possibly be produced from the best bones and acid alone. If this, then, be the case, why bother so much about purity? And why seek a guarantee which is of no consequence, and which does not admit of ready proof? Why not let the manure maker produce his soluble phosphate in the cheapest way he can? For does it not seem quite as unreasonable to demand that in any manure the soluble shall be wholly from pure bones, as it would be to require that sulphate ammonia should at least not be made from coals?

**Reverted Phosphates.**—But, in addition to this perplexity, there is the vexed question of reverted phosphates. Usually it is admitted that soluble phosphate is, on application to the soil, first precipitated, and then gradually redissolved by a rainfall slightly acidulated with carbonic anhydride. And if this be really the case, why should not reverted phosphate be every whit as good as the soluble, instead of ranking only with the ordinary insoluble portions of a manure? That the chemical change has taken place in one case before sale, and has in the other been deferred until after use, should not surely as at present involve any *very* substantial difference in the commercial value of the manure. There seems indeed but little doubt that the ordinary existing treatment of phosphates with acid is eventually but a round-about method of reproducing the original tricalcic or bone phosphate in a chemically fine state of division. And under such circumstances reverted phosphates are worthy of more considerate treatment than in this country they have hitherto received.

**Anomalies.**—It is certain, therefore, that much not only of the nomenclature, but of the usages of the chemical manure business, demand substantial revision. The manure trader has too long been dealt with in a rather unfair way, having been regarded not only as a sort of scientific expert to his customers, and as responsible for the analyses and condition of his products, but as liable to be called into account for the materials employed in his handicraft, as well as for the effects of his manufactures upon the crop. Surely this ought not so to be, for while it is highly desirable that, to the best of his ability, the manure maker should aid the farmer, there seems no equitable necessity that he should be his nurse. Wurtz, in his *History of Chemistry*, published about the time of the 1863 British Association meeting in Newcastle, declared chemistry to be a French science. But, had his thoughts momentarily rested on manurial chemistry in its commercial aspects, he would surely have made a special exception in its favour, for of all branches of science it seems the most painfully inexact. In the matter of analytical methods this is notorious. For even in so simple a question as manurial moisture, no existing method of analytical statement expresses the actual fact; while in the examination of phosphates and ammonia there still remains—after two decades of grumbling—the most trying discrepancies in different operators' results. What excuse also can decently be made for continuing to express and to value soluble phosphate not as soluble or monocalcic phosphate pure and simple, but by the quantity of tricalcic or bone phosphate consumed in its production? The thing seems absurd on the very face of it.

**Superphosphate Reaction.**—Then, again, from Ruffles' researches it seems as if the superphosphate reaction had been much misunderstood. That, in fact, the change taking place does not simply involve the conversion of two molecules

of lime into calcic sulphate, and of the remaining molecule into monocalcic phosphate, but that when in ordinary superphosphates the maximum sulphuric acid has been used to secure the desired chemical and mechanical result, there must of necessity be present a considerable amount of free phosphoric acid from the partial conversion into calcic sulphate of the third molecule of lime; and that there cannot be present any free sulphuric acid. In the case, however, of superphosphates prepared with sulphate ammonia, Ruffles also shows that the great bulk of the phosphoric acid present exists in the free state; that part of the sulphate ammonia is converted by this free acid into ammonium phosphate; and that of the rest, with the gypsum, a double salt of ammonia and lime is probably produced, which explains the hardening peculiarity of such superphosphates.

**Tyne Industry.**—About four years after the publication of Liebig's work previously referred to, or about the year 1844, Dr. Thos. Richardson began the manufacture of artificial manures at Blaydon. In connection with this venture he seems to have attempted the utilisation of the phosphates contained in the Lemington iron slag deposits, but his experiments in this direction were not apparently very popular with the farmers of that early period.

About the year 1848 Mr. Ramsay commenced manure making at Dunston; and in the year 1849 Mr. Sampson Langdale began the business at the Mushroom. By 1863 the number of manure works on the Tyne had increased to seven, making a total annual output of some 15,000 tons. But there were not until long afterwards any factories on the Wear or the Tees; while on our own river only two firms, viz., the Blaydon Manure and Alkali Company and the Monkton Manure Company were at that time makers of their own vitriol. The other firms were supplied with sulphuric acid by Mr. Christopher Myers, of Dunston, Messrs. Imcary and Co., Felling, the Jarrow Chemical Company, and the Walker Alkali Company.

The raw phosphatic materials employed were chiefly confined to bones, bone ash, spent charcoal, Cambridge, Bedford, and Suffolk coprolites, and the poorer guanos. These were allied with nitre cake, various waste nitrogenous materials (such as blood), and sulphate ammonia, the whole, excepting the guano and a portion of the bones and ash, being British products.

**Machinery.**—The bones were reduced about this time (1863) by means of serrated rolls, and the mineral phosphates by means of heavy edge runners. The materials were for the most part hand-treated in open tanks or troughs, being stirred with wood or iron rakes, but mechanical mixers were in process of adoption. Apparently, however, the knowledge brought to bear upon the dissolving operation was extremely crude, for the product was as a rule very pasty. One lot of Tyne manure seems to have been delivered at Dunbar in a state specially characteristic of the Scottish national dish—porridge—and to have required damming up to prevent its outflow from the store. Hence stove-drying had frequently to be practised (though it certainly could not have been good for the soluble), and absorption of excessive moisture by gypsum or tank waste was an every-day mode of treatment. Probably acid of too high strength was first employed under the erroneous impression that weaker acid would be less effective. And the reaction was rendered less complete by the imperfect reduction of the phosphatic materials, as a result of the employment of unsuitable grinding machinery, and of the absence of sifting appliances. The importance, too, of the presence of calcic carbonate was not then fully recognised—this being shown by one patent whose main object was its expulsion, by calcination of the raw phosphates before use. It is also remarkable, by way of contrast with the present exigencies of the trade that, owing to the high cost of sulphuric acid (about 5*l.* 10*s.* to 6*l.* per ton of "O.Y."), the great aim of the early traders was to spare it as far as they possibly could. Hence the standard of soluble in superphosphates was at first very low, being only some 16 per cent., rising to 20 per cent. somewhere about the year 1860. This is in marked contrast to the present ordinary standard of 29 to 31 per cent., and to the existing production of strengths, rising up to over 40 per cent.

*Statistics.*—In 1863 there was, as regards the Tyne, neither any import trade in raw phosphates or export trade in chemical manures, the entire business of that date being local and coastwise. The sole exception to this was bones, of which in that year 507 tons were imported from the Continent, and 811 tons from South America, making a total of 1,318 tons. But this figure, of course, by no means represented the entire consumption, which included a considerable proportion of British collected bones.

The whole of the Peruvian guano then used and sold in the North of England reached the Tyne coastwise, in transhipped cargoes, the first direct imports arriving in 1866. But as unfortunately neither phosphates nor guanos arriving coastwise were notified in the Tyne import lists before the Tyne Improvement Act of 1877, there are no obtainable figures to show the consumption of these in 1863. The total production of manure that year, however, appears to have been about 15,000 tons.

Nitrate soda was then little, if at all, used in the North of England as a direct manure, or as a source of nitrogen in phosphate manures. Its employment indeed for the latter purpose has never been very extensive, as, unlike sulphate ammonia, it cannot be passed through the mixer. The direct sale to farmers, however, of nitrate soda now forms a branch of the manure trade of growing importance, and probably not less than 2,000 tons are now annually disposed of in the North of England in this way.

The price of nitre has been extremely fluctuating. In 1863 its average would be about 15*l.* per ton, rising during the Chili-Peruvian war of 1880 to something like 20*l.*, and declining during the past 12 months to an average of under 9*l.*

*Sulphate Ammonia* making forms an important branch of the chemical manure industry, and one which in recent years has undergone considerable development. The total North of England make for 1863 is extremely difficult to estimate, but it was probably under 1,200 tons, of which some 370 tons were made by the Newcastle and Sunderland Gas Companies alone. In 1887 the two latter companies together produced nearly 1,600 tons, and the total district make in that year had nearly reached 3,000 tons, of which 300 tons were produced at Crook in the manufacture of coke. The rest was entirely obtained from gas liquors, and there is no local plant in present operation for ammoniacal recovery from other sources. Probably about one-half the entire district sulphate ammonia make goes to the Continent for use in beetroot culture. As with nitrate soda, the price of this commodity is an extremely fluctuating one. In 1863 the average would be about 15*l.* per ton (at which time the sulphate frequently contained injurious cyanides), but in 1889 it had fallen to some 11*l.* 15*s.* In the interval, however, it had temporarily risen to considerably over 20*l.*

*Peruvian Guano* has in latter years formed a declining trade, which has been partly due to unreasonably high prices, and increasing inferiority of quality. The total United Kingdom annual consumption will not at present probably exceed 30,000 to 35,000 tons, of which a very small proportion is sold in this locality.

In 1863 the chief sources of *manurial potash* were the German muriates, or the sulphates chiefly manufactured from them, for the British kelp trade had then become almost extinct. But more recently the Stassfurt kainit has chiefly been relied on, and of this a considerable quantity is supplied to farmers direct, as it has since its introduction been gradually assuming the position in agriculture formerly occupied by common salt. For manures, however, of high potash analysis, the German muriates are still extensively employed.

Since 1863 the North of England manure trade has made rapid forward strides. The number of works on or near the Tyne has now increased to eight, added to which there are also seven factories in the districts of the Wear and Tees. Six of these works possess vitriol plant. This list, however, does not include numerous bone grinding, offal, blood, fish manure, and sulphate ammonia works scattered over the Tyne, Wear, and Tees, *where phosphates are not treated with acid*. Neither does it include the half dozen works with a gross annual output of some 16,000 tons at Spittal (near

Berwick) and North Sunderland; nor the ten works in the Carlisle and Silloth districts, in which probably about 8,000 tons are annually made.

The total 1889 season's output from the above 15 factories on the Tyne, Wear, and Tees, may be approximately estimated at 40,000 tons, of which fully one-half is shipped to the continents of Europe and America.

And by way of contrast with 1863, practically the whole of this large output is manufactured from foreign materials, the 1887 phosphatic imports into the Tyne, for example, having been as follows:—

	Bones.	Mineral Phosphates.
	Tons.	Tons.
From Continent of Europe.....	492	6,803
From Continent of America, including West Indies.....	279	10,782
Coastwise (partly transhipped) ..	107	1,035
Total.....	878	18,680

But, as in 1863, an important portion of the total consumption of bones consists of those locally collected.

*Basic Slag.*—Within the past two or three years a new local industry has sprung up—in the utilisation of the basic slag from the works of the North-Eastern Steel Company (Limited) at Middlesbrough. The total annual output is about 40,000 tons, which is all contracted for by a German firm of chemical manure makers. The great bulk of it is used in Germany in the cultivation of sugar-beet, for which it appears to be a success. A small quantity, however, has been used in this country and in this district, but as the use has hardly yet passed beyond the experimental stage, little may at present be said as to results. Its phosphates are claimed to be more readily assimilable than the ordinary tricalcic phosphates ground to a like degree of fineness, and it is sold under the name of Thomas phosphate powder in a very fine state of mechanical division, 85 per cent. being guaranteed to pass through a sieve of 10,000 meshes per square inch. Phosphate equal to 17 per cent. phosphoric acid is also guaranteed.

It is boldly asserted to be even superior to ordinary superphosphates of like phosphoric acid test, but this seems on the face of it extremely problematical. For assuming the soluble phosphate in a superphosphate to be precipitated by contact with the soil, it can scarcely be supposed that a mineral phosphate in however intimate a state of mechanical division can favourably compare with as it were a phosphatic emulsion in a division chemically perfect, and consequently of maximum susceptibility to atmospheric influences.

*Present Mechanical Resources.*—In mechanical accessories the manure trade of the Tyne has undergone great recent development, appliances of the most advanced description being now in use. The old crushing rolls and edge runners which formerly sufficed for the comparatively rough treatment of the gravelly coprolites of early days, have now been abandoned for machinery able to cope successfully with the most refractory foreign phosphates, and to reduce them sufficiently for the necessary final treatment between heavy French burr millstones and sifting through revolving timpses of 2,000 to 3,000 meshes per square inch. The powerful Sturtevant mill of the Langdale Company, for instance, is equal to the reduction of over 300 tons phosphate rock per 24 hours. And generally a considerable amount of care and attention is now locally devoted to the comminution of phosphates as a means of attaining the highest dissolving efficiency. For as sulphuric acid is just about one-third its price in 1863, the great aim is to employ it as freely as is compatible with a good mechanical result.

There have also been substantial improvements in the mechanical treatment of bones, and the introduction of the high-speeded disintegrating class of machinery for the production of fine bone meals has tended to a large development of the bone meal trade.

The Noxious Vapours Acts of 1863 and 1874 did not concern the manure maker. But he was carefully provided for in the Act of 1881, and the North of England manufacturers generally were not slow to conform to the requirements of this. Condensing arrangements, therefore, of considerable efficiency are now almost universal. Their character is somewhat diverse, but the apparatus recently described by Mr. Macadam in a paper read before our Glasgow Section, and by Dr. A. B. Grifflits in his treatise on manures, is perhaps the most common and satisfactory to our Local Government inspectors. There are a number of sets (designed and erected by the writer) now at work in the North of England, and it is interesting to notice that of the seven chemical manufacturing districts throughout Great Britain and Ireland under Government inspection, the average escape of acid vapours from manure works in the North of England for the year 1888 was extremely satisfactory, and for the year previously was 50 per cent. less than in any of the other districts.

The manure trade is, in spite of general commercial activity, still struggling through a severe crisis in its history. Prices remain very discouraging, in spite of increased cost of raw material and increasing difficulties and troubles in the labour market; and yet until there be some substantial amelioration of the existing conditions of British farming, it seems almost hopeless to forecast any important or permanent improvement in a trade which is wholly dependent for its existence upon the successful pursuit of agriculture.

## Glasgow and Scottish Section.

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*Vice-Chairman:* Wm. Dittmar.

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Wm. Boyd.	G. McRoberts.
A. Cruin Brown.	E. J. Mills.
J. Christie.	J. M. Milne.
W. S. Curphey.	T. L. Patterson.
D. B. Dott.	E. Smith.
Wm. Foulis.	E. C. C. Stanford.
J. Gibson.	A. Whitelaw.

*Hon. Treasurer:* W. J. Chrystal.

*Hon. Local Secretary:*

G. G. Henderson, Chemical Laboratory, University of Glasgow.

### SESSION 1890.

April 1st (Glasgow):—

Annual General Meeting of Section.

Mr. Jas. Hope. "The Estimation of Nickel and Cobalt."

Mr. R. R. Tatlock. "Note on the Fatty Acids of Olive and other Oils."

May 6th (Edinburgh):—

Dr. J. B. Readman. "The Manufacture of Phosphorus, Part II."

Notices of Papers and Communications for the Meetings to be sent to the Local Secretary.

*Meeting held in the Societies' Rooms, 207, Bath Street, Glasgow, on Tuesday, 4th March 1890.*

MR. R. R. TATLOCK IN THE CHAIR.

## THE MANUFACTURE OF BLASTING-GELATINE.

BY GEORGE MACROBERTS, F.R.S.E., F.C.S., F.I.C.

BLASTING-GELATINE was patented by Mr. Nobel, the inventor of dynamite, in 1875. It is a compound of nitro-glycerin and nitro-cotton. When properly made it has a specific gravity of 1.5 to 1.55, and is a clear semi-transparent substance, slightly elastic, somewhat resembling

india-rubber in that respect. It usually consists of 92 to 93 per cent. of nitro-glycerin, and 7 to 8 per cent. of nitro-cotton. Blasting-gelatine is thus described by its inventor:—

"The object of this invention is to convert nitro-glycerin and analogous explosive liquids from their liquid state to a solid or semi-solid consistency. The liquid state is found to be dangerous in regard to storage, transport, and, in some cases, use; whereas the solid state is found to be highly conducive to safety and offers great facility for use, storage, and transport. In carrying out the invention, these liquid explosive substances are incorporated with another substance which is capable of gelatinising or thickening them, and for this purpose a substance is chosen which will detract little or nothing from their explosive power. Nitro-glycerin, which is the most powerful of liquid explosives, may be gelatinised by dissolving in it nitrated cellulose, such as the nitrated cotton which is known as collodion gun-cotton. The explosive compound becomes more and more gelatinous in proportion to the quantity of nitrated cellulose which is dissolved therein, and it forms, when 7 to 8 per cent. of the nitrated cellulose have been incorporated, a solid jelly, which is very safe and highly suitable for every purpose to which very powerful explosives can be applied. I do not, however, confine myself to that exact proportion, since it must be suited to the degree of hardness or consistency required for each special object in view."

The inventor then goes on to describe various mixtures with gelatinised nitro-glycerin which may be made to suit special purposes such, for example, as blasting, where a somewhat slow explosive is required, or for torpedo purposes, &c.

In the patent there is no mention made of any method of preparing the nitro-cotton required, nor of any special apparatus for mixing or incorporating the ingredients of the explosive; nor is there any mention made of apparatus for converting the finished explosive into cartridges. I soon found that special apparatus of some kind would have to be devised for incorporating the nitro-glycerin and the nitro-cotton, as these substances, when thrown together, gelatinise very slowly and very imperfectly. After numerous trials, I devised the apparatus shown in the drawing (Fig. 1), which is a rough sketch, full size, of a gelatine-making machine, such as I have used at Ardeer. The machine consists of a stout framework of wood, preferably teak, to avoid warping, with a copper or brass pan for holding the explosive ingredients, and of shafting. The horizontal shaft drives, by means of bevelled wheels, two vertical shafts to the bottom of which are fixed stirrers, which, when the machine is in motion, pass the ingredients in the pan from side to side and through each other, and in that way effect a thorough mixture in a very short time. The pan is double, the space between the outer and inner shells being filled with hot water during an operation. The outlet and inlet for the water are shown on the drawing. They may be arranged on opposite sides of the pan or on the same side, to suit the arrangements of the building in which the operation is carried on. During an operation, hot water flows continuously through the pan, otherwise a constant temperature, which is necessary, could not be kept up. The whole of the metal work of the machine is, as far as possible, of brass or gun-metal, and the greatest care is requisite in fitting up the machine, to guard against any contact between metal and metal where the explosive is. If the stirrers were to strike against each other, or against the pan, there would be danger of explosion. It will be noticed in the plan of the pan (Fig. 2) that there are four small wheels or rollers. The object of these is to facilitate the running in and out of the pan when a charge is put in or taken out. There is a kind of cradle or carriage, in which the pan is jammed during an operation. This cradle is capable of being elevated and lowered by means of screws. The screws are actuated by the lower horizontal shaft. When the pan is filled with an explosive mixture it is run in under the stirrers and, by means of the side screws, elevated until the stirrers are fully immersed in the mixture of nitro-glycerin and nitro-cotton. The pan is firmly held in that position during the stirring, and when the operation has proceeded until the explosive

mass is thoroughly gelatinised, the pan is again lowered by means of the screws, run out, and the contents removed. The capacity of the pan is such that about 2 cwt. of explosive can be treated at one operation. Each operation usually takes about an hour. The temperature of the water circulating round the pan is kept at 60° C. during the whole time. At that temperature the nitro-glycerin and nitro-cotton act readily on each other, and are resolved into a jelly. Without the hot water the gelatinisation would take place much more slowly and much more imperfectly. The temperature of the explosive in the pan is never allowed to get above 40° to 45° C. The temperature of the mass is taken from time to time, and should it show any tendency to go beyond the normal figure of 45° C. or thereabouts, means can be taken at once to cool down by cold water. The hot water is supplied from a barrel or tank standing near the gelatine-making machines. The water in the tank is heated by steam, and as the water is drawn away for the supply of the machines, it is renewed automatically, and no difficulty is found in regulating the temperature of the water in the tank to the constant figure of 60° C. At Ardeer I had eight machines in one house, ranged along the sides—four on each side. They are driven by belting from a shaft along the centre of the house, the shaft being actuated by a steam engine outside the building. As may be expected in machines intended to be used with a dangerous explosive, the motion is very slow, the stirrers revolving at from 20 to 30 revolutions per minute.

All factories for explosives in this country are regulated by orders from the Home Office Explosives Department, and only limited quantities are allowed in each building at one time. The buildings themselves are separated from each other by considerable distances and by the intervention of embankments, so that, in case of explosion in any building, the destruction may be confined to as small an area as possible. For that reason it is necessary to have the buildings in which the nitro-glycerin and nitro-cotton are made at considerable distances from those in which the gelatine is manufactured. The nitro-glycerin and the nitro-cotton and other ingredients that may be used in making the gelatine have, therefore, to be brought from considerable distances to the houses in which the gelatine is made.

In carrying out the making of blasting gelatine, therefore, the following routine is observed:—The nitro-cotton required is first brought from the nitro-cotton drying stove, weighed off in the quantity required for each charge, and put into gutta-percha or brass-lined wooden boxes capable of holding enough of nitro-glycerin and gun-cotton together for the charge of a gelatine-making machine. A number of these boxes, containing a quantity not exceeding the total allowed by the regulations, is then taken to the nitro-glycerin washing-house, and the proportion of nitro-glycerin required is weighed off and added to each box. The boxes are then conveyed to the gelatine-making house, and the contents transferred to the pans of the respective gelatine machines. As each pan is charged it is put under the stirrers, screwed up, and the machine started, and in the course of about an hour the gelatinisation is complete, and the pan ready to be cleaned out for another charge.

At first great difficulty was experienced in meeting the Government requirements for gelatine. Before a new explosive can be manufactured in this country, the permission of the Home Office has to be obtained, and in order to obtain that permission, samples of the explosive have to be submitted for trial, and from the behaviour of these samples when under chemical and physical treatment the Government regulations for the manufacture and testing of the explosive are deduced. The Home Office had prescribed that in the case of blasting-gelatine offered for sale the explosive was to be free from every species of exudation, and that a sample taken from a box of the explosive as offered for sale should, when exposed to freezing and thawing three times successively, show no signs of exudation; and that a 50 grms. sample exposed in a test tube for 10 minutes to a temperature of 160° F. or 71° C. should show only very faint action on iodised and starched test paper. The conditions required by these tests were found, at first, to be difficult to fulfil, and fault was found by the Government inspectors with certain samples which failed to comply with the prescribed tests.

After careful investigation I found that the nitro-cotton was almost entirely to blame for the failure to pass the tests, and I then set about to discover a method of making nitro-cotton which would enable the finished product to fully comply with the Government requirements. The kind of nitro-cotton for making blasting-gelatine is a mixture of bi-nitro- and mono-nitro-cellulose—not the ordinary tri-nitro-cellulose or real gun-cotton. By careful attention to the proportions and mixing of the acids for the nitration of the cotton, and to the temperature at which the nitration was effected, and by thorough washing and boiling of the nitro-cotton, I succeeded in producing an article of uniform quality which made an invariably good gelatine. The nitro-cotton requires to be finely divided and well dried. The dividing I effected by means of a paper pulping machine, and the final washing by boiling and steaming the nitro-cotton, sometimes for days. I effected the drying by means of hot air. The nitro-cotton is placed in a lightly constructed stove in which there are frames with trays on which the nitro-cotton is placed, and a current of hot air is driven through the stove, the temperature being kept as nearly as possible between 100° and 110° F. It is not allowed to exceed the latter limit. In the earlier made gelatine it was thought unnecessary to go beyond centrifugal drying for the nitro-cotton. By means of a centrifugal machine the nitro-cotton can be dried down to 25 per cent. of water, and that proportion of water, in a substance which formed only 8 per cent. of the whole mixture, appeared to be a trifle, but experience soon showed that the presence of water tended to make the nitro-glycerin exude from the finished gelatine. The gelatine now made at Ardeer with thoroughly dried nitro-cotton, prepared as described above, complies fully with all the Government tests, and is found to keep for any length of time without showing signs of exudation. Her Majesty's Government bought, about three or four years ago, 30 tons of blasting-gelatine for the War Department. It is kept in magazines under water, and up till now it has shown no signs of change. When 3 or 4 per cent. only of nitro-cotton is incorporated with nitro-glycerin, the result is a thin liquid jelly which is of itself unsuitable for making into cartridges, but such a jelly is well suited for mixing with charcoal and nitrate of potash, or with wood meal, nitrate of ammonia and charcoal, so as to form other varieties of blasting-gelatine. Blasting-gelatine made with those ingredients is sold under various names, such as gelatine-dynamite, gelignite, &c. By using a small percentage of the thin gelatine and a larger percentage of the nitrate of potash and charcoal, or wood meal mixture, a cheap and useful variety of gelatine is formed, suitable for many kinds of blasting. Pure blasting-gelatine, consisting of 7 per cent. nitro-cotton and 93 per cent. nitro-glycerin, is the strongest. The varieties formed with other ingredients have for their object the cheapening of the explosive as well as its adaptation to particular kinds of blasting.

Of all the nitro-glycerin explosives, blasting-gelatine is the strongest. If the energy of dynamite be represented by 100, that of blasting-gelatine will be 150. Nitro-glycerin itself shows less energy than blasting-gelatine.

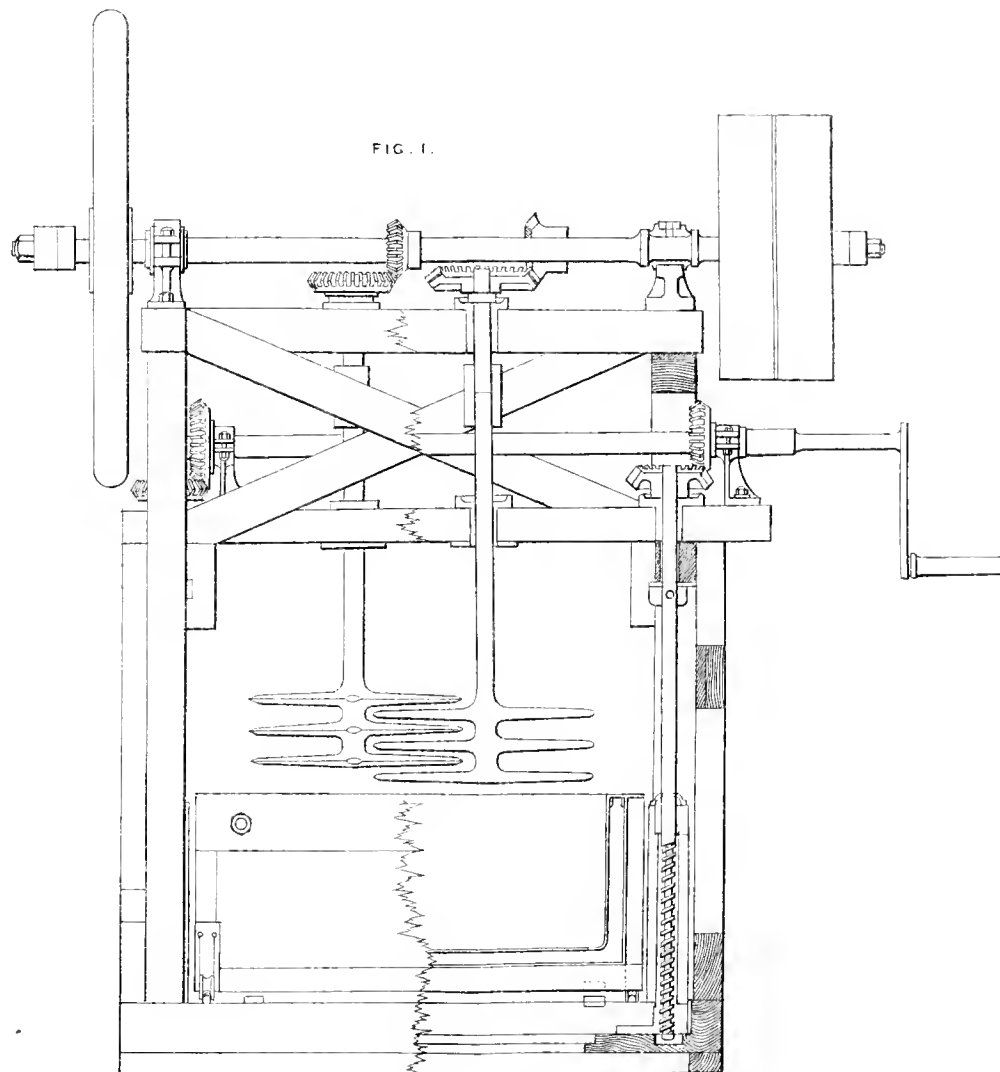
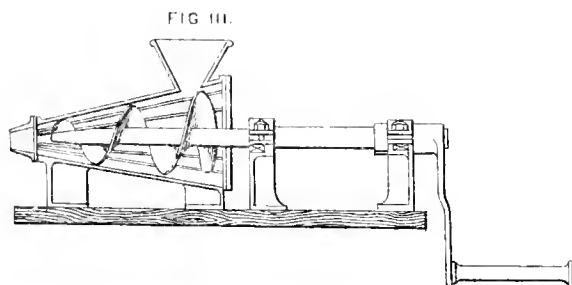
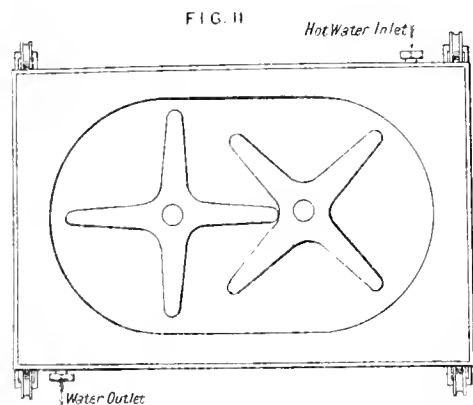
It is specially suited for blasting in water, as its explosive properties are in no way affected by water, and it may remain any length of time under water without change. On the other hand, dynamite under such conditions loses its nitro-glycerin, and becomes in fact resolved into its ingredients; so that to blast with it effectively under water, the cartridges require to be protected by some waterproof material.

Blasting-gelatine in all its varieties is exploded by means of a detonator, as is the case with dynamite, but detonators for exploding gelatine require to be much stronger than those for dynamite.

Blasting-gelatine made as described above has to be converted into cartridges to suit the various mining operations. The machine I devised for that purpose is a kind of sausage-making machine, of which a rough sketch is here shown (Fig. 3). The whole machine is made of brass or gun-metal. It consists of a case with a shaft and screw. The revolution of the shaft causes the thread of the screw to push forward the gelatine introduced by the square opening at the top, to the nozzle, whence the gelatine issues in a



# PLANT FOR THE MANUFACTURE OF BLASTING GELATINE.



Scale of One Inch to the Foot.







continuous rope or cord. The nozzles are of different sizes to suit the various sizes of cartridges required. It is an essential principle of these machines that no metallic contact shall be allowed inside. When the machine is in operation the screw revolves quite clear of the case, and the thrust of the machine is taken up by the collar shown against the second plunger-block. If by any chance the screw and shaft should be thrust back, there is a leather washer against which it would rub, so that there would be no danger. The way in which the machine operates is as follows:—

The girl who works the machine lifts a lump of gelatine and pushes it into the opening with her left hand, at the same time turning the handle with her right hand. The threads of the screw push the gelatine rapidly forward to the nozzle, and in a few revolutions the whole of the gelatine is converted into a long rope. When the girl has expelled a yard of two of this rope from the machine, she seizes it with her left hand, and with a wooden knife in her right quickly cuts it into cartridge lengths, which are taken up by other girls and rolled in cartridge paper. Each cartridge but contains four girls, and one machine worked by one girl turns out sufficient cartridges to keep the whole four busily employed. Each cartridge-making machine can form into cartridges from 5 to 10 cwt. a day, depending on the diameter of the cartridges.

It is wonderful to observe the great dexterity the cartridge girls attain in working these small machines. The cartridges, although cut into lengths by the eye and folded without any measure as to length or diameter, are very nearly uniform in size. You will notice in the section of the cartridge-making machine case that there are lines running from the back converging towards the nozzle. These lines represent grooves in the casing like the rifling of a gun. Without these grooves the efficiency of the machine would be very much reduced or altogether destroyed. The grooves prevent the gelatine from revolving with the screw and hold it in position, while the screw pushes it forward. Each machine has an assortment of nozzles which can be taken off and put on in a few seconds, so that it is adapted for cartridges of any diameter.

Regarding the safety of the operations described in the foregoing, it is satisfactory to state that from the commencement of the manufacture until now there has been no accident in the blasting-gelatine department at Ardeer; so that it may be fairly inferred that the principle on which the machines are constructed and worked is a thoroughly safe one.

As the patent for blasting-gelatine was taken out in 1875, and as its duration was only for 14 years, it expired last year. The manufacture of blasting-gelatine in this country is confined to one firm, that of Nobel's Explosives Company, and is likely to continue in their hands alone. Although anyone is now at liberty to commence the manufacture of nitro-glycerin preparations, so far as patents are concerned, yet the manufacture is surrounded with so many difficulties, the difficulty of obtaining a suitable site, difficulties with the local authorities, difficulties with the Home Office, and last though not least the difficulty of obtaining suitable technical management, that it is not likely Nobel's Company will find their monopoly disturbed for a long time to come.

I have thus briefly described the manufacture of blasting-gelatine as carried on by me at Ardeer, and I have avoided touching on many interesting points connected with the manufacture, because of the time that would be taken up by such references. There are, connected with the manufacture of blasting-gelatine and nitro-cotton, many interesting points which would require for their discussion much more time than is allotted for a single paper.

#### DISCUSSION.

Mr. STANFORD said he was sure they would all regret very much that Mr. MacRoberts was unable to be present to read the paper himself. Regarding the monopoly in the manufacture possessed by Nobel's Explosives Company, he quite agreed with what was said in the latter part of the paper, that it was not likely to be disturbed for some time. The

solution of the collodion cotton by the nitro-glycerin was a great discovery because it enabled them to employ the nitro-glycerin in a partially solid form. He used to think the manufacture of this blasting-gelatine a terrible thing for any chemist to undertake until he had the pleasure, with some others, of going over the works at Ardeer, when he was struck with the ingenious manner in which the various processes were carried out, which showed that where there was great care and thorough knowledge there was no danger.

Mr. KATER said that the reason why collodion cotton and not gun-cotton was used in the manufacture of blasting-gelatine was that gun-cotton is insoluble in nitro-glycerin.

The CHAIRMAN said that he thought that the invention and the devising of the apparatus for producing this most perfect of all explosives—blasting-gelatine—was one of the most striking examples they could find of what often happens with regard to inventions, viz.:—that they lay dormant for a long period without any possibility of applying them, until some one appeared who had the skill or the genius to devise and work out methods and machinery for their application as practicable and profitable commercial processes. Previous to 1867 nitro-glycerin had been employed in America, but its use became a dead letter in this country until Mr. Nobel patented methods for absorbing the liquid nitro-glycerin into porous substances, which resulted in the preparation of dynamite, containing 75 per cent. of nitro-glycerin and 25 per cent. of kieselguhr. But even this extraordinary product had its disadvantages, for when brought into contact with water the nitro-glycerin exuded. This was obviated, however, by the discovery of blasting-gelatine, a more perfect explosive than nitro-glycerin itself.

#### THE MACARTHUR-FORREST PROCESS OF GOLD EXTRACTION.

By J. S. MACARTHUR.

In order to understand the present systems of gold extraction to properly appreciate the work already done, and to gauge the difficulties to be surmounted, it is necessary for us to look back and trace the connexion between the known forms of gold as found in nature and the methods used to separate it from its base environments.

In the earliest ages gold would naturally only be found in nuggets and grains (our word carat is derived from a Persian word meaning a grain) which required no means of separation beyond picking up—happy age when gold had only to be picked up!

From grains the ancients would come down to gold dust, and this was no doubt separated from the sand or earthy matter with which it was associated, by the skilful use of air currents which were caused to blow away the sand, leaving the precious metallic dust. This method is still practised in Arabia and the East. Wherever water was plentiful it was found that it did the required separation better and more economically than air, a method of separation which has been and still is in use everywhere all over the world. The prospector, digger, explorer, and even the tourist nowadays provides himself a "pan" in which to wash a sample of "dirt" at the nearest stream. From the pan comes the "cradle," "long tom," and the innumerable mechanical arrangements for the separation of gold from earthy matters. Up to this point we have only mechanical means of separation which depend on the high specific gravity of gold compared with sand, clay, &c., but at this stage in the evolution of gold extraction we find the first move made towards a chemical method. When there was a lot of rich dirt in his pan the digger found it very difficult to wash away the last of the sand without losing a large proportion of the finely divided gold, so he hit on the expedient of pouring in a little mercury, which formed a heavy, massive, though fluid alloy with the gold, making the separation of the last portions of the light

granular sand a very easy matter. This plan is still in world-wide use alike by the solitary digger who lives in a lonely cañon and by the well-organised company that "hydraulics" 1,000 tons of "pay dirt" per day.

Let us now look at the present state of the gold industry. Let us imagine a digger who has exhausted all the gravel and pay-dirt in the cañon or gully; he travels up the gully looking to right hand and to left for traces of the precious metal; occasionally he finds a piece of gold-bearing rock and is led on and on till he finds the source of these auriferous stones to be a reef. Now begins gold-mining proper. A shaft is sunk on the reef or a tunnel driven into it and great masses of rock are brought to the surface, and naturally the miner is led to imitate nature by crushing this rock to a fine sand and then he modifies the treatment formerly given to alluvial deposits to adapt it to the new circumstances. The ore, immediately on being crushed to powder (which is generally done by huge gravitation stamps), is carried over an amalgamated copper plate by a stream of water. In passing over the amalgamated plate the gold is caught by the mercury, while the sand, now called tailings, is washed off. The gold is recovered by scraping the amalgam off the copper plate at stated intervals, generally once a month, when by distillation the mercury is recovered as well as the gold separated. If all the gold the ore contained existed in the heavy metallic form the recovery by this method would be complete and the loss nil; but the tailings are frequently found to contain a little gold, and on close examination particles of pyrites and sulphides of the various base metals are found diffused through the mass. When these metalliferous particles are separated from the mass of tailings, it is found that they principally contain the gold, so that it is now usual in practice to pass the tailings through some form of concentrating machinery, of which the well-known Frue-vanner is a good example, whereby the pyrites and other sulphides are retained in virtue of their higher specific gravity and the sand washed away. The rich proportion now called "concentrates" may contain up to 20 oz. of gold per ton, though four or five ounces is much nearer the average figure.

If the concentrates are examined closely, even microscopically, no free gold can be distinguished, and if they be treated with mercury they will yield to it little or none of their gold contents. Sometimes a considerable portion of gold may be got from them by a long continued grinding in cast-iron pans in presence of mercury; this grinding seems to force the sulphur and base metals as well as the gold into combination with the mercury, so that the bullion got from the amalgam often contains over 95 per cent. of copper, lead, and other base metals, while there is a corresponding loss of mercury which is carried away partly as sulphide and partly in a "floured state"—the flouring is caused by the small globules of mercury being coated with a film of sulphide of mercury which prevents the particles coalescing. Thus there is a double loss: chemical, by the formation of sulphide of mercury, and mechanical, by flouring. This loss is so well known that concentrates are only treated by this form of amalgamation in localities where nature forbids any more elaborate process. Sometimes the amount of loss may be lessened by roasting the concentrates before amalgamation, but this is by no means a perfect remedy, as the roasting removes only volatile constituents of the concentrates, principally sulphur, while the base metals, lead, zinc, &c., are left in the form of oxide to oxidise and waste their equivalent of mercury. Attempts have also been made to decrease the loss of mercury and increase the yield of gold by adding to the contents of the pan all sorts of chemicals to "doctor" the "sickened"—that is, floured mercury. These chemicals are generally mixtures of sulphate of iron, sulphate of copper, salt, lime, soda, &c. This kind of "doctoring" may be effective in some cases, but it is seldom practised with intelligence. Where the percentage of concentrates is limited, and where there are a number of mines, it is common for some enterprising man to set up a smelting or chlorination work which is made large enough to deal with the concentrates of the district. I will not take time to describe the various processes of

smelting, nor could it be done at any evening sederunt, but we may bear in mind that all smelting processes end in alloying the gold with lead, and cupelling in the usual way with which we are all familiar. Because of the obvious impracticability of smelting, requiring well-built brick furnaces, coal, fluxes, &c., at the typical gold mine high up on the mountain range, possibly near or beyond the timber line, chlorination is much more commonly practised and consequently merits a full description. In giving this description, I will confine myself chiefly to the old standard Plattner process as I have seen it practised in California, where timber is abundant and cheap.

The first and essential operation prior to chlorination proper is roasting. It is obvious that a mixture of sulphides and arsenides of iron, copper, lead, zinc, and metals generally will absorb an almost unlimited amount of chlorine, so that the small proportion of gold present would fare badly in a general scramble of the molecules for chlorine. That the metals may have the least opportunity to combine with chlorine, the sulphur is expelled, and their affinities satisfied as far as possible with oxygen by roasting. The roasting is generally done in a large reverberatory furnace, that has no very noteworthy feature in its construction. The concentrates are charged into the furnace at the cold end, heated very gradually, continually stirred and slowly worked forward into the hottest part, care being taken to admit air freely during the whole operation. Where chlorination is practised on the most extensive scale this roasting generally takes about 24 hours, and consumes from a minimum of half a cord to a cord of wood per ton roasted. (A cord of wood gives about as much heat as a ton of coal.) It is a common saying that the success of chlorination depends more on the furnaceman than on the chlorinator, and on looking into hard chemical facts we find that this is really the case, for if only one-half per cent. of iron were left unoxidised it would absorb nearly 1 per cent. of chlorine, equal to about 3 per cent. of bleaching-powder, and all this before the gold gets a single molecule of chlorine. Towards the end of the roasting, and about 15 or 30 minutes before the ore is discharged from the furnace, it is usual to stir in a small proportion of common salt. The object of this is to satisfy with chlorine as far as possible copper, zinc, and other metals whose oxides have a tendency to form chlorides when they get chlorine presented to them in the free state. This chloridising device is, however, only partially effective, as the chlorides formed are apt to be immediately decomposed under the influence of hot air, and in the case of lead the oxide or sulphate will not combine with chlorine in the furnace, but combines with it readily when offered moist and at the ordinary atmosphere temperature. The roasted and oxidised ore is now sprinkled with water to make it slightly moist, and is then charged into a wooden vat having a perforated false bottom. The chlorine, generated at an outside source, is led in between the true and false bottom, and gradually permeates upwards through the mass of the porous ore. The small amount of water held by the ore then becomes a saturated solution of chlorine, which gradually acts on the gold, so that in the course of one or two days it may be washed out as the soluble chloride by a further amount of water. This weak solution of chloride of gold is run into a tank, a solution of ferrous sulphate added, the precipitate of gold allowed to settle for 48 hours if possible, and the supernatant solution allowed to flow off. When sufficient gold precipitate has accumulated, it is collected and run into bars. Besides the Plattner form of chlorination which I have described, there are others which vary from Plattner and among themselves in the manner of application of the chlorine—most of them using revolving barrels instead of open vats for the chlorination proper.

The best known of these are:—

The Mear's process, where chlorine is used under its own gaseous pressure.

The Newbery-Vautin, where air pressure is used.

The Pollok, recently described before this Section of the Society, where hydraulic pressure is used, and

The Thies, where the chlorination is done in a barrel without any pressure, as described in this Journal, 1889, 895.

Let us now return to the mine. The deeper we sink down into the reef we find that while the richness of the ore may remain constant, the proportion of free gold, that is amalgamable gold, becomes smaller and smaller, while the proportion of gold-bearing sulphides becomes greater and greater till we come to the water level, where it often happens that the free gold leaves entirely and becomes wholly refractory, that is, non-amalgamable. The water level is generally regarded as the point where atmospheric and weather influences cease, and that down to that point the rain water has penetrated and gradually washed away the oxidisable base metals, leaving the inert gold in the crevices of the equally inert quartz, whereas the ore found below the water level, not having been subjected to oxidising influences, retains all its metals untouched and unchanged.

An interesting problem now presents itself:—An ore consisting of a complex mixture of silica and the various compounds of iron, copper, lead, zinc, antimony, arsenic, and sulphur (for convenience I will include all these compounds under the general term sulphides) and gold in the proportion of 10,000 parts of silica and sulphides to one part of gold, is very much richer than the average auriferous ore, and the question is how can we best separate the one from the ten thousand. Under favourable circumstances the gold and the useful metals may be recovered by smelting, but these favourable circumstances, which are proximity of the gold mine to coal, clay, limestone, and other fluxes, are quite exceptional, as auriferous reefs are generally found in primary formations. As before implied, chlorination is frequently inapplicable—no attempt is ever made to chlorinate gold ores containing an appreciable quantity of lead—and where applicable is always troublesome and never cheap. When this question presented itself to the Drs. Forrest and myself, we tried to find some solvent which, unlike chlorine and mercury, would have a stronger affinity for gold than for sulphides. Acting on this principle we drew out a list of all probable or possible solvents fulfilling this condition. This list included cyanides, and we found that these salts solved the problem.

Our experiments were conducted first on a small scale, and on ores of all kinds and from all mines in all parts of the world. The result of these small trials was so satisfactory that we gradually worked from less to more, and in no long time larger quantities were worked, and now the process is in, or on the eve of being put in operation in all quarters of the globe. I have now much pleasure in describing the method of working most generally applicable. The ore is ground to about the fineness of sea sand. If instead of ore we are working tailings from the amalgamation process, these are generally not re-ground, but treated as delivered. The finely divided material is mixed with a solution of a cyanide, say cyanide of potassium, containing on an average 0.4 per cent. of cyanogen as the cyanide of potassium or other alkali or alkaline earth. The ore and solution are stirred together for about six hours, more or less, this being the average time required to dissolve the gold: in practice the time required is determined by direct experiment. When the gold is known to be dissolved, the pulp is discharged into an ordinary filtering tank, where the filtration may, if necessary, be assisted by suction, and where the ore is washed by water or by the waste cyanide solution from a previous operation. The ore, after treatment with cyanide solution, is unchanged to the eye, as almost nothing but the imperceptible proportion of gold present has been removed. The gold now being in solution, the next object is to get it precipitated, and here we encounter a serious difficulty. Gold and cyanogen have such a strong mutual affinity, that it is difficult to get any substance that will separate them. The gold cannot be precipitated by any ordinary method, such as the use of ferrous sulphate or oxalic acid; even sulphuretted hydrogen and sulphide of sodium will not precipitate gold from its cyanide solution, though they precipitate silver. On referring to books on electro-gilding, we got no assistance, as the invariable method given for the recovery of gold from cyanide solutions was, evaporate to dryness and fuse the residue. We had noticed, however, by experiment, that zinc precipitated gold very feebly, and tried this in the same way that copper is precipitated from its ordinary solutions by scrap iron, but

scrap zinc had no effect; then granulated zinc was tried, with a most imperfect and disappointing result; then heating in presence of scrap and granulated zinc, but this had only the effect of forming urea, and assisting the precipitation very little indeed. Further, we tried zinc dust, but still there was no success; finally we prepared some zinc in a form like sawdust, porous and with a large surface of bright metal. On allowing the cyanide of gold solution to trickle through a mass of the zinc, we found that it trickled out gold-free, and, better still, we found that the action became more vigorous and pronounced after a portion of the gold had been precipitated on it, doubtless as gold and zinc formed together a more powerful electro-chemical precipitant than zinc by itself. An arrangement of a porous mass of zinc like a sponge formed a chemical filter, which at once precipitated and collected the precious metal; indeed, so like an ordinary water purifying device was this zinc filter, that many non-technical visitors formed and held tenaciously to the idea that the gold was in suspension in the cyanide solution, and the zinc was used merely because of its durability. Improvements in detail were made in the direction of increasing the surface and decreasing the weight of the zinc; till now we have it in threads, 1 lb. of which occupies about two gallons measure. The zinc in this form is possessed of enormous chemical activity, of which the strongest and most direct evidence is the fact that it burns in the air like thin shavings of wood. When the gold has been deposited, it is necessary to separate it from the excess of zinc present. The filiform structure of the zinc, and the exceedingly fine powder, as which the gold is deposited, render this an easy matter. The filiform mass of zinc with gold powder adhering is vigorously shaken in water, when the gold falls off, and the fibrous particles of the zinc may be collected in a sieve. The gold settles easily, is collected, and fused directly into bullion.

Having now described the chief points in the process from the technical standpoint, let us look at the purely chemical aspect it presents. Reference has already been made to the fact that a cyanide solution acts on the gold in ores in preference to the sulphides of base metals with which it may be combined or associated; this selective action is the keystone of the whole process. It is known that metallic gold is dissolved by a cyanide; it is also known that sulphides of copper, zinc, and iron artificially prepared are readily acted on by a cyanide solution; but we found a very different state of affairs in treating ores where these or similar compounds exist prepared by the hand of nature herself. Nature seems to render the base metallic compounds insoluble, while the gold combined with or contained in them is more easily acted on than the metal itself. This at once raises the question—how does gold exist in base refractory ores, is it combined or free? This question I admit I cannot answer satisfactorily to myself. Judging from theoretical considerations only, I think gold should exist almost invariably in the free state, for if we assume that sulphides, pyrites, &c., were formed by deposition from aqueous solution, the gold would be deposited in the metallic state. This may be shown by dropping some powdered pyrites into a solution of chloride of gold, when gold will be precipitated as metal; on the other hand let us assume that the pyritous formations carrying gold were formed by igneous agency in this case the gold ought to be metallic too, because any sulphide of gold already existing would be decomposed by the high temperature, and of course sulphide of gold cannot be formed at a high temperature. It is true that sulphide of gold may combine with an alkaline sulphide and resist decomposition by heat, but such a combination as a matter of fact does not occur in nature. Telluride of gold, and I think antimonide of gold are not decomposed by heat, but they occur in small quantities only and do not affect the general argument. In spite of these theoretical considerations, however, we have strong evidence to show that gold exists in several different states of combination or of molecular structure. Let me give one case in illustration. A sample of tailings from the ordinary process of stamping and amalgamation was received, and I think it may safely be assumed that the mercury had extracted some gold from the ore which produced these tailings. We treated the finely ground tailings by alkaline solution of bromine which

extracted a considerable portion of gold, and the bromine treatment was repeated time after time till it ceased to extract gold; then it was treated with a hot solution of ferric bromide which yielded a further quantity of gold. This treatment was repeated time after time till it ceased to extract gold, and finally the residue of tailings was smelted and a still further quantity of gold produced. As this ore yielded its gold in stages to the four different processes of amalgamation, treatment with bromine, treatment with ferric bromide and smelting, we infer that the gold existed in four different states, mechanical or chemical. Notwithstanding the complex nature of these tailings which consisted of all sorts of sulphides with the four-fold gold, the cyanide acted on it, almost perfectly extracting 93 per cent. of the precious metal, whilst the highest extraction by any of the bromination methods was about 40 to 50 per cent. The cyanogen seemed to have an affinity for gold and a power of penetration so much stronger than mercury or bromine, that it broke through barriers impregnable to them and captured the gold.

Elsner has stated that metallic gold dissolves in cyanide of potassium only in presence of oxygen. Not having seen the original account of Elsner's researches, I am not in a position to criticise his experiments, but I never could find that the presence of oxygen was necessary either to dissolve gold by itself or from ores by cyanide. If a piece of gold be immersed in cyanide solution so that air to act on it would have to penetrate 2 in. or 3 in. of the solution, the gold will dissolve in its usual slow and steady fashion. The equation shows that either oxygen must be absorbed or hydrogen evolved. I have seen no evidence of the former, and can adduce no proof of the latter; but I think the latter the more probable, because I cannot conceive oxygen penetrating even a film of cyanide solution without at once oxidising the cyanide to cyanate, whereas in the other case, as suggested to me by my friend Mr. Ellis, the nascent hydrogen may be at once seized by the excess of cyanide present and ammoniacal compounds formed. However, we do not concern ourselves much with the reactions of pure gold, but as a matter of fact we cannot find that oxygen plays any part in the cyanide extraction of gold from ores. We have treated an ore with cyanide with free access of air, and then a parallel experiment was done with boiled water, the bottle filled to the stopper with solution and ore, and the stopper sealed. The extraction was the same in both cases.

We have so far only considered ores which are refractory from chemical as well as mechanical causes, but ores are frequently found in which the gold is refractory from a purely mechanical cause, which is the extremely fine division of the gold. The cyanide process is quite as applicable to this class of ore as to any other. These ores are generally spongy and absorbent, and this we find enables us to dispense with stirring the ore and cyanide solution together, the same end being accomplished by allowing the cyanide solution to percolate slowly through the mass—a clear saving in power.

The advantages claimed for the cyanide process over smelting and chlorination are—as compared with smelting, it requires no furnaces and no coal, and no fluxes, and thus may be used successfully in remote situations where smelting is utterly impossible. As compared with chlorination, the cyanide process involves no roasting, therefore no furnaces and no fuel. Moreover, by the cyanide process, ores containing lead, zinc, or earthy carbonates which cannot be worked to profit by chlorination, may be as easily and profitably treated as any other. For chlorination, about 7 per cent. of the weight of the ore to be treated often has to be carried to the ore in the form of bleaching-powder, say  $1\frac{1}{2}$  per cent., bisulphate of soda 2 per cent., and sulphate of iron,  $2\frac{1}{2}$  per cent., with packing, say, of 1 per cent., whereas about 1 per cent. of the ore to be treated will cover the weight of the necessary cyanide, zinc, and packing. Thus, by chlorination, one ton of chemicals will treat only about 14 tons, whereas by the cyanide method one ton will treat 100 tons.

Moreover, chlorination does not extract any silver, but by the cyanide method the most of the silver—invariably

associated with gold in ores—is extracted along with the latter metal at the same operation. For the sake of simplicity I have not named silver in the body of this paper, but the remarks made in reference to gold generally apply to the silver associated with it.

From what I have seen of gold mines and gold mining in the Sierra Nevada, in the Rocky Mountains, in the Southern States of America, in Nova Scotia, from the many kinds of ore I have seen from all parts of the world, and from what I know of the selective and energetic action of cyanides, I confidently predict that cyanide of potassium, hitherto used only to polish amalgamated plates, will take a front rank as chief agent in gold extraction.

#### DISCUSSION.

Mr. BUCHANAN said that Mr. MacArthur in his able paper had pointed out at some length the great contrast between the weight of chemicals used in the chlorination and cyanide processes. He admitted that the former required a greater weight of reagents than the latter, but the author should also state the relative prices of these chemicals, the cost of the chemicals for chlorination being, he believed, but a fraction of the cost of cyanide. What was the cost of cyanide and other reagents used per ton? He would also be obliged if Mr. MacArthur would give some information as to the mode of, and time taken for filtration.

Mr. POLLOK remarked that he had experienced no difficulty in chlorinating ores containing lead and earthy carbonates up to 5 per cent.; larger quantities he had not met with. Such ores had also been successfully chlorinated by other processes in the United States.

Mr. RODGER asked how Mr. MacArthur proposed to produce the cyanide in sufficient quantities to cyanise the ores, and also how he produced the zinc in the gossamer-like form, such as the sample he had shown.

Mr. FAWSITT remarked that it seemed to him the price of cyanides would rise very much if this process came into operation. He would like to ask if Mr. MacArthur recovered his cyanide, and if so, how, or if it was lost. It was a most important point if it could be recovered.

The CHAIRMAN asked if, in treating the tailings from an amalgamation process, would any mercury left behind affect the cyanide? How did the process behave with such ores as contained a large proportion of silver in the form of sulphide? The amount of gold extracted was only one point, and must be taken in conjunction with other circumstances. To say that a certain percentage of gold was extracted from an ore did not convey an accurate idea of the value of the process, as its success, or otherwise, depended on a number of conditions. There could be no doubt as to the extreme simplicity, efficiency, and cheapness of cyanides for dissolving gold and silver, but he supposed this process, like most others, would not be perfect in every little particular, nor could it, any more than any other, give precisely the same uniformly successful results on every class of ore.

Mr. MACARTHUR, in reply to Mr. Buchanan, said that in his paper he did his best to be accurate in comparing costs, but the actual costs of chemicals could not properly be compared in Glasgow where there were no gold mines; the proper estimate and comparison must be made on the spot where the chemicals were to be used. The best answer to the question of costs was to give the latest return from Australia, which showed that 100 tons had been worked by the MacArthur-Forrest process at a total cost of 25s. per ton. Of this total amount 17s. 6d. was the price of the cyanide used, including all charges of transit and a high duty. The average time for a filtration washing was about two hours.

In regard to what Mr. Pollok said, all he could say in reply was that he did his best to be accurate in giving the weights of chemicals necessary for chlorination, and had got the figures for bleaching-powder and bisulphate of soda from a pamphlet issued by the Pollok Company; but as the amount of ferrous sulphate was not given it was calculated

as the exact equivalent of the bleaching-powder. About ores containing lime he must congratulate Mr. Pollok on his successful modification in roasting by which this difficulty, which Mr. Pollok too had recognised, had been overcome. Regarding lead, he had found that sulphate of lead absorbed free chlorine readily, and that oxide of lead actually precipitated gold from its solution as chloride.

In reply to Mr. Rodger, he said that the cyanide used in this process was prepared in the usual manner from ferro-cyanide of potassium. The zinc shown was prepared by mounting discs of sheet zinc in a turning lathe and taking off shavings by a turning tool.

In reply to Mr. Fawsitt, he thought that a larger demand was more likely to lower than to raise the price of cyanide, and though they had recovery processes for the cyanide they had not been put in practice, and in estimating costs nothing had been allowed for the recovery of cyanide.

Replying to the Chairman's questions he said that the presence of mercury in tailings presented no obstacle whatever to the action of the cyanide, and though silver as sulphide was not very easily dissolved in some cases, usually it could be extracted. A 20-ton lot might be quoted, where the silver (as sulphide) and the gold were equally well extracted, though there was an enormous excess of silver over gold. In fact the cyanide had an almost human intelligence in its preference for gold.

#### DISCUSSION ON MR. FAWSITT'S PAPER "ON THE FIXATION OF NITROGEN."

(This Journal, 1890, 30.)

MR. HENDERSON said that, contrary to a statement in Mr. Fawsitt's valuable paper, it was usually stated in textbooks that metallic derivatives of acetylene,  $C_2HK$  and  $C_2K_2$ , could be obtained by passing acetylene over melted potassium, and he thought that a similar calcium derivative,  $C_2Ca$ , had also been prepared. He was doubtful also of the probability of the formation of  $K_2C_2O_2$ , seeing that potassium was said to decompose acetylene at high temperatures, with separation of carbon, while  $K_2C_2O_2$  was said to be obtained by the action of potassium on acetylene at about  $80^\circ C$ . He should be glad of further information on these points.

MR. STANFORD said he thought that the paper might be looked upon as a concise résumé of what was known upon the subject of the formation of cyanide on a large scale. Regarding the process mentioned by Mr. Fawsitt of the passing of ammonia over charcoal highly heated, he would like to ask if the decomposition was at all complete, and also at what temperature the reaction took place.

The CHAIRMAN said that, considering the great importance of this subject, it seemed strange that so little work of a practical character had been done with the object of fixing nitrogen in some useful form. This might be partly accounted for, however, by the failure of the extensive trials made by Bramwell and Hughes some 40 years ago. At first these experiments promised very well, both on the small and larger scale, but when the process was tried in manufacturing it was found that the loss was so great and the expenses so heavy that it had to be abandoned. There had been, he thought, a good deal of misapprehension with regard to the reactions which were said to have taken place in the experiments conducted by Bunsen and Playfair with the object of determining the presence of cyanogen in a particular zone of the blast furnace. He had not understood that cyanogen had ever been produced by the action of nitrogen upon carbon. It was all very well to withdraw gases from a furnace in operation and test these for cyanogen, which might have required a base for its fixation. Undoubtedly it would be found, if the gases were taken off

at the proper point, but it was necessary to insure not only the absence of bases, but also that the cyanogen even then found in the free state had not been fixed by the metal of an alkali or of an alkaline earth. Another question was whether cyanide of ammonium had ever been produced by passing ammonia over charcoal at any temperature whatever. He did not believe that had ever been done, although it was stated by Kuhlmann, about 50 years ago, to have taken place. If ammonia could be converted into cyanide of ammonia so easily, then the problem, it seemed to him, was already solved; but if so, it was inexplicable that so simple and so very desirable a reaction had never found application in practice. He was not stating his own opinion so much as that of others who had paid much attention to the subject practically, when he said he thought that was the crucial point in the question.

MR. PLAYFAIR said that in the production of cyanides the difficulty was to get an apparatus suited to stand the work, and he wished to ask if such an apparatus could not be found in that employed for the making of water-gas.

MR. FAWSITT, in reply, said that in regard to Mr. Henderson's remarks, he was obliged to him for drawing his attention to the existence of calcium acetylene, of which fact he was ignorant. He was quite aware of the existence of potassium acetylene, as prepared by passing acetylene over melted potassium, and when he spoke in his paper of it not having been produced, he really meant under the conditions occurring in the preparation of cyanides previously described. If it were produced under these conditions, it must be produced in the manufacture of potassium, a fact which had not been established. Again, supposing it were produced, it did not follow that it united with nitrogen to form KCN, at least he had seen no proof of it.

As regarded the non-existence of  $K_2C_2O_2$  at high temperatures, he always understood that it was produced at the highest temperatures in the manufacture of potassium, and the method adopted to prevent its formation was to cool the vapour quickly.

As regards Mr. Stanford's question about the production of ammonium cyanide by passing ammonia gas over heated carbon, he could not speak from experience, but judging from the statements made by several able chemists, he should say that there was strong evidence of its production, but whether with facility or not he could not say. In reference to Mr. Tatlock's question as to whether or not cyanogen could be produced by passing nitrogen over heated carbon, the only affirmative statement he had seen was by Delbriich, and he was inclined to believe it; but after testing it as described in the paper, should say, that under the conditions existing in processes described under classes 1 and 2, it was not so produced.

Lastly, the question asked by Mr. Playfair he could not answer from experience. The regenerative principle was patented and applied to an apparatus made by Mr. Mond, but with what success he did not know. As the temperature favourable to the production of cyanide was very high, he did not think that an apparatus worked on this principle could be kept sufficiently hot for the successful working of the process.

## Journal and Patent\* Literature.

### I.—GENERAL PLANT, APPARATUS, AND MACHINERY.

*The Exhibition of Preventive Appliances in Berlin in its Relation to Chemical Industry.* T. Oppler. Chem. Ind. 12, 501—508, 528—532, and 549—556.

THE exhibition was promoted for the display of methods and appliances having for their object the minimising of risk to life or limb where the circumstances include the element of danger. The author has limited his article to a notice of those means which can be employed for the protection of workers in the chemical industries, but the number of these is too large to admit of more than a general reference here. The notices are arranged under several heads.

(a.) *Signal Thermometers.*—A very delicate means of signalling abnormal changes of temperature has been found in the use of a spiral formed of ribbons of steel and brass soldered together, one end being fixed and the other free to close an electric circuit. The difficulty of soldering these two metals tightly enough to withstand prolonged contraction and expansion, however, prevented any great extension of the use of the apparatus, until a method of coating the steel ribbon with nickel has made it possible to make a permanent joint with solder, and of this improved form of thermometer several examples were exhibited, with the addition also of a method of registering the actual changes of temperature by a diagram.

(b.) *Eye Glasses, Respirators, Clothing.*—Specially mentioned is a so-called normal eye-glass, set in a mask of galvanised iron wire, easily adaptable to irregular features, allowing of free circulation of air and of comfort to the wearer by the use of leather padding. Free circulation of air being indispensable to keep down excessive perspiration under the mask, this is provided for in an invention of Director Stroof by a slit between the glasses and the mask. Respirators to be worn in an atmosphere containing noxious gases consist of wet or dry filters of suitable material, or are fitted to a wash-bottle appliance for cleansing the air before breathing. In some cases a breathing apparatus connected by tubing with the outside air, similar to that used by divers, is the only possible. In a report presented by a German Industrial Union, emphasis is laid on the advisability of wearing close-fitting garments by workers attending on machinery. Complete outfits in leather and asbestos were exhibited for use in gunpowder factories.

(c.) *Lighting and Safety Lamps.*—Petroleum lamps were shown constructed on lines similar in principle and object to the "Shaftesbury" safety-lamp, whilst the illumination of spaces containing inflammable vapours or materials is provided for by gas or electric light transmitted from the outside through glass windows. Of the various forms of safety-lamp, the basis rests essentially on the principle invented by Davy, but by enclosing the flame in a cylinder of glass, greater illumination is obtained, and by allowing for the entrance of air at the bottom of the lamp a steadier and brighter light is maintained, with the further advantage that the lamp gives timely warning of the presence of fire-damp. Various contrivances for locking the lamps and for extinguishing and relighting the flame from outside are referred to, and

some forms of safety-lamp are arranged for use with benzene. A safety-lamp for indicating approximately the proportion of explosive gas present, depends on the employment of a flame fed with absolute alcohol, which is readily susceptible to alterations in height and colour in presence of small quantities of inflammable gas.

(d.) *Transport of Corrosive Liquids.*—For drawing off the contents of carboys, a rubber cap fitting over the neck is provided with bent glass tubes and a force pump, the arrangement working like that of an ordinary wash-bottle. For preventing the spitting of liquid in emptying a carboy, a rubber nose-piece having a vent hole is fitted on the neck. Instead of the usual straw packing it is proposed to surround the carboy with infusorial earth which would absorb the corrosive liquid in case of fracture, the packing being easily regenerated after an accident by washing and drying. An automatic arrangement for controlling the admission of compressed air to a montejus is described.

(e.) *Mineral Water Manufacture.*—All the exhibits in this department involve the use of liquid carbonic acid which avoids many of the dangers incidental to the preparation of the gas itself. The safety arrangements comprise separate expansion vessels, pressure gauges, safety-valves, and automatic pressure-reducing valves. As a provision against possible fracture of the iron cylinder containing liquid carbonic acid, it is enclosed in a strong case of iron trellis work.

(f.) *Varnish and Pitch Manufacture.*—To minimise the chance of firing in case the liquid should boil over the sides of the vessel, the furnace is separated from the boiling house by fire-proof walls. Vessels for boiling lined oil are movable on wheels along a line of rails, so as to afford easy removal when necessary. Foul smelling vapours are drawn by a draught through a coke fire.

(g.) *Soap Manufacture.*—To avoid the dangers incidental to shallow boiling vessels, the rendering copper is made deep and is surmounted by a hood.

(h.) *Lamp-black Manufacture.*—An automatic apparatus for the deposition and collection of lamp-black in a closed chamber provided with windows for observing the progress of the operation. A vertical revolving shaft carries plates cooled with water, on which the lamp-black accumulates, the deposits being removed and delivered into receptacles by scrapers.

(i.) *Match Manufacture.*—Dipping machines for pointing the matches with igniting composition, and a mixing vessel for preparing the composition, are made to work automatically and in a closed chamber, by which all danger to the workmen from phosphorus fumes is avoided.

(k.) *Glue Manufacture and the Grinding of Bones and Slag.*—The bleaching of glue with sulphurous acid and the subsequent drying of it are carried out in a closed chamber through which a current of air is maintained, carrying the sulphurous acid and moisture to a chimney. This method is applied to the grinding and treatment of bones, mineral phosphates and Thomas slag, where injurious dust and acid vapours are given off. Roller mills are closed in by curtains or boxes, and the dust is conveyed by a draught of air to a chamber containing baffling arrangements to cause the deposition of the dust. The ground material is sifted as it comes from the mill, and the coarser particles are returned automatically to the mill, thus confining the grinding practically to one operation. Some of the mills consist of revolving drums of metal containing steel balls. Draughts of air are used to remove foul smells from raw bones and the vapours evolved in the treatment of phosphates in all cases where the workmen would be inconvenienced by them.

(l.) *Disposal of Dust.*—The means noticed under this head are comprehended under two general methods, that of the well-known "Cyclone" dust collector, and of chambers containing flannel or other kind of filtering cloth from which the accumulated dust is periodically removed by shaking or beating.

(m.) *Soda. Caustic Soda.*—The revolver furnace for making soda ash represents the latest successful method

\* Any of these specifications may be obtained by post, by remitting the cost price, plus postage, to Mr. H. Reader Lack, Comptroller of the Patent Office, Southampton Buildings, Chancery Lane, London, W.C. The amount of postage may be calculated as follows:—

If the price does not exceed 8d. ....	½d.
Above 8d., and not exceeding 1s. 6d. ....	1d.
" 1s. 6d.,     "     "     2s. 4d. ....	1½d.
" 2s. 4d.,     "     "     3s. 1d. ....	2d.



of reducing the inconvenience to workmen to a minimum, whilst the use of protective shields and rails of ictal are prescribed to ward off danger in attending to caustic pots. The chemical factory "Rhenania" exhibited a model, including all the protective means applicable to a factory making soda and caustic by the Leblanc process.

(n.) *Manufacture of Explosives*.—Photographs of arrangements in use in various factories were exhibited. Gases given off in the preparation of fulminate of mercury are drawn off into a chimney, whilst danger from accidental explosions is provided for by the use of wire network surrounding the apparatus. Arrangements for submerging in water certain portions of the apparatus of a gunpowder factory are set in motion by the force of an explosion when that should occur from any cause.—G. H. B.

## PATENTS.

*Improvements in Machinery for the Separation of Solid Matters from Liquids, and for Washing Out and Exhausting the Solid Matters so Separated.* W. S. Squire, London. Eng. Pat. 3065, February 21, 1889. 8d.

THE separation of the solid and liquid matters is effected by a machine, which consists of four inclined, revolving cylinders, constructed of perforated metal, wire gauze, or of cloth stretched upon suitable frames, and having troughs underneath them to receive the liquid which passes through, while the solid matter is discharged at the lower end of the cylinder into receivers, each of which is provided with an agitator to mix the solid matter with any liquid that may be added to it, and a pump to remove the mixture so made. The solid and liquid matters are made to travel from cylinder to cylinder in contrary directions; the former thus meets liquid becoming more and more dilute, whilst the latter meets matter more and more charged with soluble material, thus a very concentrated solution of the soluble matter is obtained on the one hand, while on the other the solid matter is obtained thoroughly exhausted of its soluble constituents. When the exhaustion is not completed a larger number of cylinders may be used. There are two claims and two sheets of drawings.—E. S.

*Improvements in Apparatus for Filtering Liquids, or for Preparing Extracts from Substances.* M. J. E. Vassel, Paris, France. Eng. Pat. 3736, March 2, 1889. 11d.

THE invention relates to improvements in "apparatus for rapidly filtering liquids in closed vessels, wherein the air is exhausted from the receiving chamber below the filter, and is forced into the space above the liquid to be filtered." The apparatus can also be transformed into an exhausting apparatus, with intermittent or continuous circulation, and it may be employed in preparing extracts, such as making coffee, and for other purposes. The apparatus consists of a vessel with a tubular lower end, containing the filtering medium and the liquid to be filtered, and which fits into a receiver for the filtered liquid. The inlet valve of a small air-pump, which may be a caoutchouc ball, with valves such as are used for spraying purposes, communicates by a tube with the receiver, and the outlet valve with the top of the filtering vessel. On working the ball, the air is exhausted from the receiver, and passing into the filtering vessel, produces considerable pressure, and thus forces the liquid rapidly through the filtering medium into the receiver. Various modifications of the apparatus are shown on the six sheets of drawings which accompany the specification. There are 11 claims.—E. S.

*An Improved Method or Process of and Apparatus for the Lixivation of Various Materials.* H. H. Lake, London. From "La Société Anonyme des Parfums Naturels de Cannes," Paris, France. Eng. Pat. 10,361, June 25, 1889. 8d.

See under XX., page 325.

*An Improved Method of and Apparatus for the Treatment of Smoke.* S. Elliot, Newbury. Eng. Pat. 12,460, August 7, 1889. 11d.

A stop valve or door is inserted in factory chimneys, and the smoke sucked by means of an exhaust apparatus and driven through a tank in which, by mechanical appliances, water is kept agitated, and the soluble gases and soot are retained. For brick kilns or pottery chimneys a cone or inverted cup requires to be fitted on the top of the chimney and connected with the exhaust apparatus. There are three sheets of drawings giving details.—D. A. S.

*An Improved Machine for the Purpose of Washing and Refining Fuller's Earth, China Clay, and other similar Substances.* W. A. Sheppard, Midford. Eng. Pat. 20,561, December 21, 1889. 6d.

THE machine patented consists of a tank, in which is placed the Fuller's earth or similar material to be treated, and in which it is mixed with water, a second tank provided with an agitator to complete the mixing, and a "refining machine," consisting of two stationary concentric cylinders and an inner vessel like an inverted cone, having slots in, and projecting strips on, its sides, by the rotation of which, at about 100 revolutions per minute, the coarser particles are driven against the walls of the inner cylinder and fall on to the bed-plate at its base, whence they are swept into the annular space between the two cylinders by curved radial arms revolving with the inner vessel, and can be removed from time to time. The finer particles remain in the inner vessel and flow away, suspended in the wash water, through a pipe at its base into settling tanks.—B. B.

## II.—FUEL, GAS, AND LIGHT.

### PATENTS.

*Improvements relating to Artificial Fuel, Refractory Material for Lining Retorts, and Bricks and Tiles.* R. Stone. Eng. Pat. 13,768, September 24, 1888. Amended January 8, 1890. 4d. (Compare this Journal, 1889, 872.)

THE amendment relates to the percentage composition of one of the fuels. Instead of 3 per cent. of oil and 94 per cent. of peat to 3 per cent. of sulphuric acid, 3 per cent. of the acid is mixed with 75 per cent. of oil and 22 per cent. of peat.—D. A. S.

*Improvements in Apparatus for Generating and Burning Gases from Hydrocarbon Oil and Water.* H. Darby, London. Eng. Pat. 2796, February 16, 1889. 8d.

THE improvements consist in a feed-water regulator supplying water by syphon action from a reservoir to the steam generator, and a series of superheating pipes through which the steam passes before mingling with the oil to be burned. For details the specification must be consulted.—O. H.

*Improvements in the Manufacture of Fuel.* A. Besson, Paris, France. Eng. Pat. 4288, March 12, 1889. 4d.

WOOD charcoal in dust or powder is mixed with a silicate, the mixture diluted with water, and powdered anthracite or other mineral coal added till of suitable consistency, when the mass is moulded. 5 per cent. of silicate and 10 per cent. of wood charcoal with 85 per cent. of anthracite, are said to give a good result.—D. A. S.

*Improvements in or relating to Apparatus for the Manufacture of Water-Gas.* F. H. Briggs, Torquay. Eng. Pat. 6090, April 9, 1889. 8d.

A NUMBER of retorts are arranged and worked in series in connexion with a superheater, consisting of a coil covered with a thin protective coating of chalk or fireclay and heated by the products of combustion of the fuel used in heating the retorts or chambers. Arrangements are described for throwing each retort, as the carbonaceous matter contained in it becomes exhausted, out of the series, and connecting again to the end of the series as soon as recharged and reheated.—O. H.

*Improvements in Appliances for Burning Heavy Hydrocarbon Oils, which Improvements are also Applicable to other Purposes wherein it is desirable to Maintain a Fluid at a Constant Level.* M. Blaman, London, and F. M. Sims, Manchester. Eng. Pat. 10,724, July 3, 1889. 8d.

To render it easier to burn heavy hydrocarbon oils, the patentees combine an air-tight pneumatic reservoir with an overflow chamber on a level with the burner so that the level of the oil can be automatically maintained in the burner.—D. A. S.

### III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

*Fucosol.* K. Bieler and B. Tollens. Ber. 22, 3062—3064.

THE authors claim priority over Maquenne (this Journal 1889, 974) in having established the constitution of fucosol, of Stenhouse. Fucosol obtained from marine algae is not a homogeneous compound, but consists of a mixture of methyl furfuran and furfuran identical with that contained in bran.—J. B. C.

*Fichtelite.* E. Bamberger and L. Strasser. Ber. 22, 3361—3368.

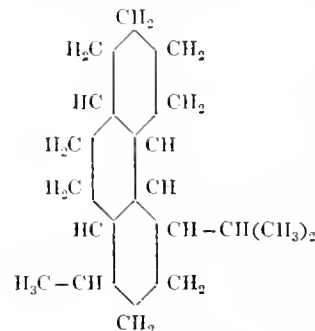
ONE of the authors (this Journal, 1889, 382 and 383) pointed out some time ago that all endeavours, his own included, to get at the constitution of fichtelite by chemical means had failed. At the end of the communication a reaction was described, which has since, in conjunction with the researches of Liebermann and Spiegel, thrown a great deal of light on the subject. In order to definitely fix the empirical formula and molecular weight of fichtelite, a number of carbon and hydrogen determinations made by different observers were collected, and the boiling point and vapour density were also carefully determined. The boiling point (corrected) of fichtelite was found to be  $355.2^{\circ}\text{C}$ . at 719 mm. pressure,  $235.6^{\circ}\text{C}$ . at 43 mm., and  $233.6^{\circ}\text{C}$ . at 42 mm. These numbers exclude the possibility of fichtelite being a di- or tetrahydrosesquiterpene, since the sesquiterpenes boil at about  $250^{\circ}\text{C}$ ., and the hydrides would boil at a still lower temperature. The vapour density, determined in an atmosphere of nitrogen, was found as the mean of two experiments to be 8.69. This vapour density, in conjunction with the elementary composition of fichtelite, points to the formula  $\text{C}_{18}\text{H}_{32}$  for that body. This formula differs from that of retene by a plus of 14 atoms of hydrogen; and as retene and fichtelite both occur in peat, and probably both are fossil products of vegetable resin, it was considered not improbable that a connexion might exist between the two.

Now Liebermann and Spiegel obtained by the exhaustive reduction of retene, not perhydroretene, but dodecahydroretene, which contains two atoms of hydrogen less than fichtelite. After vainly employing various reagents the authors succeeded in abstracting two atoms of hydrogen from fichtelite by means of iodine.

*Dehydrofichtelite* forms a clear, colourless, viscid liquid, possessing a splendid blue fluorescence. It boils at  $344^{\circ}$ — $348^{\circ}\text{C}$ . under a pressure of 714 mm. It is with difficulty soluble in cold glacial acetic acid and in alcohol, but readily soluble in ether, benzene, chloroform, and carbon disulphide. Dehydrofichtelite,  $\text{C}_{18}\text{H}_{30}$ , possesses all the properties ascribed by Liebermann and Spiegel to retene dodecahydride. The boiling point of the latter is given by Liebermann and Spiegel as  $10^{\circ}$  lower than that of dehydrofichtelite; but this seeming discrepancy disappears when it is considered that their boiling point is uncorrected. A point deserving more consideration is, that although the authors obtained from dehydrofichtelite a hydrocarbon which yielded on oxidation an orthoquinone, they were yet unable to positively identify either retene or retenequinone. Still, as Liebermann and Spiegel state that retene-dodecahydride yields on distillation with zinc-dust but a small quantity of retene, it is quite possible that owing to the small amount of substance worked with, these bodies escaped detection. On these grounds the authors are of opinion that the identification of fichtelite as perhydroretene is almost a certainty. By the further action of iodine on dehydrofichtelite, tetrahydrofichtelite is obtained, which is very similar in all respects to dehydrofichtelite. All attempts to carry the dehydrogenation still further have failed. By the treatment of dehydrofichtelite in the cold with bromine, beautiful glassy prisms were obtained, apparently containing bromine in an additive form. The authors hope to continue their investigations on this substance.—H. T. P.

*The Constitution of Fichtelite.* L. Spiegel. Ber. 22, 3369—3371.

It has been shown by various observers that fichtelite is exceedingly stable towards most reagents. The author and Liebermann had noticed this same property in the perhydrides of aromatic hydrocarbons. This fact led the author to suspect the existence of a connexion between retene and fichtelite, which both occur in peat. The perhydride of retene would be  $\text{C}_{18}\text{H}_{32}$ , which is also the formula of fichtelite. The author and Liebermann (Ber. 22, 779; this Journal, 1889, 451) were unable to obtain perhydroretene, but they succeeded in preparing retene dodecahydride,  $\text{C}_{18}\text{H}_{30}$ , which possesses all the properties ascribed by Bamberger (see preceding abstract) to dehydrofichtelite,  $\text{C}_{18}\text{H}_{30}$ , and which is prepared by the action of iodine on fichtelite. To prove the correctness of the assumption—based on the above facts—that fichtelite is perhydroretene and possesses the constitutional formula—



the author is endeavouring to prepare fichtelite from retene. Already some time ago the author obtained by freezing and pressing a large quantity of crude retene dodecahydride a small proportion of a crystalline body, melting at  $48^{\circ}\text{C}$ . The substance also seems to be formed by the treatment of retene dodecahydride with phosphorus and hydriodic acid. The author is continuing his researches on this compound and hopes to prove its identity with fichtelite.—H. T. P.

*The Sulphur Compounds in Crude Petroleum and in Petroleum Residues.* C. F. Mabery and N. W. Smith. Ber. 22, 3303—3305.

This is a research on the form in which sulphur exists in petroleum from the Ohio oil-fields.

The sulphur compounds occur chiefly in the higher boiling-point fractions 200°—300°. From this they were readily obtained by treatment with strong sulphuric acid. The diluted acid solution was neutralised with lead carbonate or calcium hydrate. The salts so formed were very unstable and easily decomposed in aqueous solution by distillation with steam, giving as distillate a clear yellow oil.

The authors worked on a measured quantity of this oil of sp. gr. 0.9245 at 16.5°, and containing 14.97 per cent. of sulphur. When distilled without steam at ordinary pressure it decomposed, giving off sulphuretted hydrogen. It was therefore distilled under a reduced pressure of 100 mm. and collected in fractions.

Fractions containing sulphur:—

	Per Cent.
80°—90°.....	None.
100°—105°.....	18.23
135°—140°.....	15.52
150°—155°.....	16.44
185°—190°.....	14.21

The absence of mercaptans and the presence of sulphides in all was found.

Bromine vapour caused a thick white cloudiness with all fractions; liquid bromine combined with explosive violence. Bromine water formed readily a heavy oil.

80°—90° fraction. Bromine compound gave by analysis  $C_{17}H_{14}Br_2$ . The hydrocarbon may thus be identical with that of Pelletier and Walther (Berzelius, Jahresb. 21, 470).

HBr formed an oily product heavier than water with fractions below 135°, the 80°—90° fraction being wholly converted. This heavy oil had the characteristic odour of substituted ethanes.

100°—150°. An alcoholic solution of mercuric chloride formed hard crystals either on standing or by crystallisation from benzene.

100°—120°. The mercuric chloride addition compound crystallised from benzene answered to the composition  $(C_{17}H_{14})_2SHgCl_2$ .

135°—140°. The addition compound showed  $(C_{17}H_{14})_2SHgCl_2$ . It decomposed on melting at 140°.

150°—160°. The addition compound, as in case of the higher boiling point fractions, was a thick viscid oil. Instead of corresponding to  $(C_{17}H_{14})_2SHgCl_2$ , it consists probably of a mixture of ethyl and propyl sulphides.

98°—101°. The addition product, as in case of lower boiling point fractions, formed hard crystals, and was probably a mixture of propyl and butyl sulphides.

The bromine compounds were not further examined on account of the difficulty of purifying them.

The authors are further investigating the nature of these compounds.—D. A. S.

*On Cumaron in Coal Tar.* G. Kraemer and A. Spilker. Ber. 23, 78—83.

CUMARONE may be obtained from the portion of the light tar oils boiling at 168°—175°, after phenols and pyridine bases have been removed by extraction with alkali and acid. One kilo. of this purified tar oil is slowly mixed with 450 grms. bromine, the temperature not being allowed to rise over 0°. The oil is first coloured intensely red, then reddish-brown, and a yellowish-white crystalline mass separates out, which is at once filtered off through glass wool. The filtrate is left for 24 hours at 5° to 10°, when a further 80 grms. of large prismatic crystals are deposited, which are purified by recrystallisation from chloroform. The formula of the substance is  $C_8H_6Br_2O$ , and it must be regarded as the dibromide of cumaron. By heating with alcoholic potash, the monobromide is formed, crystallising from dilute alcohol in prismatic crystals, melting at 39° and boiling at 219°—220°. By acting on a dilute alcoholic solution of the bromide with sodium amalgam the bromine

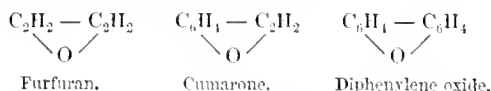
is slowly replaced by hydrogen. The oil obtained is almost pure cumaron, boiling at 170°—171°. The dichloride is readily prepared by passing chlorine into a dry ethereal solution of cumaron. It is a yellowish oil, boiling at 245°—248°. The monochloride is obtained from it by the action of alcoholic potash. It melts at 74°—75°, and boils at 215°—217°.

Cumaron is not acted on by aqueous or alcoholic or even melted potash. It is likewise indifferent to the action of hydrochloric acid and potassium cyanide, ammonia or zinc-ammonium-chloride at 300°. Oxidising agents, such as permanganate and chloride of lime, act on it vigorously.

Cumaron is so stable a body that if 100 grms. be passed through a red-hot tube 94 grms. are recovered unchanged. Sulphuric acid, however, readily acts on it with formation of a polymer, which is called paracumaron. The latter is resinous, and its alcoholic solutions are said to be of use for covering wood and metal, as the layer is perfectly unaffected by acids or alkalis.

By using an excess of sulphuric acid another polymer of cumaron is produced. It is at first soft and finally turns into a brittle infusible mass insoluble in all solvents. On heating cumaron with sulphuric acid, sulphonic acids probably result.

Cumaron stands midway between two other well-known products of dry distillation, thus—

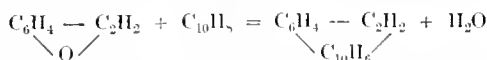


This series is typical of a large number of similarly constituted bodies found in coal tar, a systematic table of which is appended (see top of next page):—

Besides cumaron there are similarly constituted bodies in various fractions of coal-tar oils. In fact it is the presence of these substances which cause the characteristic colourations when solvent naphtha is treated with sulphuric acid. The authors have also discovered benzonitrile in the tar oils which they used for obtaining cumaron. This is no doubt the source of the benzoic acid found by K. E. Schultze in phenolic residues, for it is not probable that benzoic acid is present in the distillation products of coal tar, seeing that at the high temperature it would be sure to unite with ammonia with ultimate formation of benzonitrile.—A. R.

*Synthesis of Chrysene and Similar Hydrocarbons.* G. Kraemer and A. Spilker. Ber. 23, 84—87.

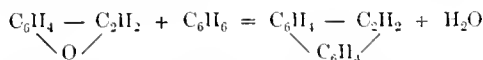
KLOTZ discovered some years ago that the best method of preparing chrysene from certain fractions of solvent naphtha was to pass the latter through heated tubes, and he was able to obtain about two per cent. of chrysene from the original oil by this process. The authors having placed beyond doubt the presence of cumaron in heavy tar oils (see previous abstract), were led to believe that that substance had a good deal to do with the production of chrysene. When passed through red-hot tubes cumaron remains unchanged. But if mixed with naphthalene considerable quantities of chrysene are formed. This was to be expected, seeing that 1 mol. of cumaron and 1 mol. of naphthalene need only lose 1 mol. of water to give 1 mol. of chrysene. The quantities of the latter which were obtained in this manner only amounted to about  $\frac{1}{3}$  per cent. of the mixture, but no doubt more will be formed when the tube through which the mixture is passed can be heated more effectually or is filled with dehydrating agents. It may be added that naphthalene vapour alone gives under the above conditions no trace of chrysene. The reaction which takes place is plainly visible from the equation—



The ready manner in which this process could be carried out led to a consideration as to whether it were not possible to explain the production of a variety of other bodies in coal tar by equally simple reactions. It was but natural to

$\begin{array}{c} \text{C}_2\text{H}_5 - \text{C}_2\text{H}_5 \\ \diagdown \quad \diagup \\ \text{S} \end{array}$	..	..	..	..	$\begin{array}{c} \text{C}_6\text{H}_5 - \text{C}_6\text{H}_5 \\ \diagdown \quad \diagup \\ \text{S} \end{array}$
Thiophen.					Diphenylene sulphide.
$\begin{array}{c} \text{C}_2\text{H}_5 - \text{C}_2\text{H}_5 \\ \diagdown \quad \diagup \\ \text{NH} \end{array}$		$\begin{array}{c} \text{C}_6\text{H}_5 - \text{C}_2\text{H}_5 \\ \diagdown \quad \diagup \\ \text{NH} \end{array}$			$\begin{array}{c} \text{C}_6\text{H}_5 - \text{C}_6\text{H}_5 \\ \diagdown \quad \diagup \\ \text{NH} \end{array}$
Pyrrol.		Indole.			Carbazol.
.. .. ..	..	..	..	..	$\begin{array}{c} \text{C}_6\text{H}_5 - \text{C}_6\text{H}_5 \\ \diagdown \quad \diagup \\ \text{C}_2\text{H}_5 \end{array}$
					Phenanthrene.
$\begin{array}{c} \text{C}_2\text{H}_5 < \begin{array}{c} \text{CH} \\   \\ \text{CH} \end{array} > \text{C}_2\text{H}_5 \end{array}$		$\begin{array}{c} \text{C}_6\text{H}_5 < \begin{array}{c} \text{CH} \\   \\ \text{CH} \end{array} > \text{C}_2\text{H}_5 \end{array}$			$\begin{array}{c} \text{C}_6\text{H}_5 < \begin{array}{c} \text{CH} \\   \\ \text{CH} \end{array} > \text{C}_6\text{H}_5 \end{array}$
Benzen.		Naphthalene.			Anthracene.
$\begin{array}{c} \text{C}_2\text{H}_5 < \begin{array}{c} \text{CH} \\   \\ \text{N} \end{array} > \text{C}_2\text{H}_5 \end{array}$		$\begin{array}{c} \text{C}_6\text{H}_5 < \begin{array}{c} \text{CH} \\   \\ \text{N} \end{array} > \text{C}_2\text{H}_5 \end{array}$			$\begin{array}{c} \text{C}_6\text{H}_5 < \begin{array}{c} \text{CH} \\   \\ \text{N} \end{array} > \text{C}_6\text{H}_5 \end{array}$
Pyridine.		Quinoline.			Acridine.
.. .. ..	..	$\begin{array}{c} \text{C}_6\text{H}_5 - \text{C}_2\text{H}_5 \\   \\ \text{C}_2\text{H}_5 \end{array}$			$\begin{array}{c} \text{C}_6\text{H}_5 - \text{C}_6\text{H}_5 \\   \\ \text{C}_2\text{H}_5 \end{array}$
		Dihydronaphthalene.			Dihydrophenanthrene.
.. .. ..	..	..	..	..	$\begin{array}{c} \text{C}_6\text{H}_5 - \text{C}_6\text{H}_5 \\ \diagdown \quad \diagup \\ \text{CH}_2 \end{array}$
					Fluorene.

try the synthesis of phenanthrene from eumarone, and here also the authors were successful. On passing the vapour of eumarone and benzene through red-hot tubes, these substances reacted on each other with formation of phenanthrene according to the equation—



Now most of the phenanthrene in coal tar cannot be derived from the eumarone. Gräbe has in fact shown that the former is always produced when the vapours of toluene, stilbene, or dibenzyl are passed through hot tubes. Of these, however, it is only the first that we can regard as a great source of phenanthrene; eumarone no doubt comes next. Whether on passing the vapour of toluene and eumarone through red-hot tubes, the latter remains unchanged and the former alone takes part in the production of phenanthrene, or whether both substances react on each other with formation of methylphenanthrene, has not yet been ascertained. The authors in fact believe it will be possible to build up a variety of hydrocarbons starting with eumarone. Thus they expect that an isomer of naphthalene, if not naphthalene itself, can be prepared from ethylene and eumarone. Moreover there is large scope for interesting work in trying to bring about condensations between eumarone and phenols or indoles (carbazol) as well as with furfuran. The authors are however unable to pursue the subject further, and invite others to devote themselves to this promising field of research.—A. R.

*Phenyl-thiophen.* A. Renard. *Compt. Rend.* **109**, 699—700.

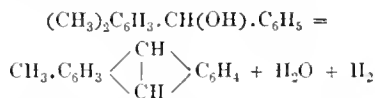
This body is prepared by passing a mixture of toluene and sulphur vapour through an iron tube heated to redness. The toluene is introduced into the tube drop by drop at the same time that small fragments of sulphur are introduced through a lateral tube, the proportions of the two substances being equal. A black product is obtained, which solidifies

on cooling, and gases consisting of sulphuretted hydrogen and carbon bisulphide are evolved. On distilling the black product some  $\text{CS}_2$  and toluene pass over; the temperature then rises and a liquid distils which solidifies in the receiver to a yellow mass. This product may be purified by recrystallisation from alcohol, in which another thiophen derivative dissolves which has not been fully investigated. Phenyl-thiophen crystallises in colourless crystals melting at  $170^\circ$ . It gives with isatin and concentrated sulphuric acid a blue colouration. The author has prepared from this compound dibromo-di- and di- and tetra-sulphonic acid derivatives.—J. B. C.

*On Homologues of Anthracene and Anthraquinone.* K. Elbs. *J. prakt. Chem.* **41**, 1—32. (See this Journal, 1886, 321; and *J. prakt. Chem.* **35**, 466.)

THE following method for the production of homologous *o*-benzoyl-benzoic acids is described. One part of aluminium chloride and two parts of carbon disulphide or petroleum ether are introduced into a large flask with a reflux condenser, a mixture of one part phthalic anhydride and the respective hydrocarbon (one mol. to one mol. phthalic anhydride) is then added. The reaction usually commences directly with a strong evolution of hydrochloric acid, if not, the flask may be warmed. The mixture is left on the water-bath till the evolution has almost ceased; 6—12 hours usually suffices. The cooled residue is agitated with a little water, some crude hydrochloric acid is poured in through the condenser, and the mixture heated to boiling. The contents are then transferred to a tubulated retort, and steam introduced. Carbon disulphide or petroleum ether, or any unchanged hydrocarbon distil over, leaving unchanged phthalic anhydride as phthalic acid and aluminium in solution. The benzoyl-benzoic acid collects as a thick heavy oil at the bottom, and after pouring off the superabundant liquid, it can be washed and purified, either by Meyer's method (*Ber.* **15**, 636) from the highly-crystalline ammonium salt, or by crystallisation from benzene, acetic acid, or alcohol. The yield is increased by use of excess of the hydrocarbon.

*β-methylanthracene*,  $C_6H_4 : (C_2H_5) : C_6H_3 \cdot CH_3$ , melting point  $199^\circ-200^\circ$ . Prepared by boiling *p*-xylylphenylketone for 12 hours, and then distilling. The distillate is stirred to thin consistency with alcohol, the alcohol drawn off, and the precipitate recrystallised from a mixture of benzene and alcohol or acetone and alcohol; also from *m*-xylylphenylketone or from *m*-xylylphenylcarbinol—from the latter easily, as shown:—



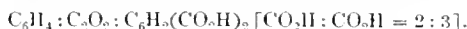
Or by reduction of the following compound—

*β-methylanthraquinone*,  $C_6H_4 : C_2O_2 : C_6H_3 \cdot CH_3$ , melting at  $176^\circ-177^\circ$ , by treating *p*-tolyl-*o*-benzoic acid with strong sulphuric acid, diluting with water, filtering, and drying the precipitate, and purifying it by sublimation.

*o*-dimethylanthracene,  $C_6H_4 : (C_2H_5) : C_6H_2 \cdot (CH_3)_2$  [ $CH_3 : CH_3 = 2 : 3$ ]. In yellow plates melting at  $246^\circ$ . Sublimes in bluish-green fluorescent plates. Made by reduction of (2:3) dimethylanthraquinone with zinc dust and ammonia. A picric acid derivative was formed in garnet red needles, which decomposed from  $108^\circ-125^\circ$ , but could not be prepared pure for analysis.

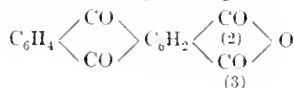
*o*-dimethylanthraquinone,  $C_6H_4 : C_2O_2 : C_6H_2 \cdot (CH_3)_2$  [ $CH_3 : CH_3 = 2 : 3$ ]. In long yellow needles melting at  $183^\circ$ . By treating *o*-xylyl-*o*-benzoic acid with strong sulphuric acid, diluting, allowing to stand 2—3 days to crystallise, and filtering through glass-wool. Purified by sublimation or by crystallising from boiling xylene.

A (3:4) *o*-dimethylanthraquinone could also be obtained from *o*-xylyl-*o*-benzoic acid. But the preparation of *o*-dimethylanthracene by reduction with zinc dust and ammonia shows the above-described to be the (2:3) compound.—*Anthraquinone-o*-dicarboxylic acid—



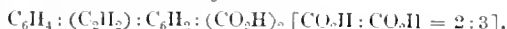
In yellow needles melting at  $340^\circ$ , insoluble in water, but soluble in cold alcohol. By oxidation of foregoing compound with dilute nitric acid by heating at  $220^\circ$ . On heating it sublimes, giving—

*Anthraquinone-o*-dicarboxylic Anhydride—



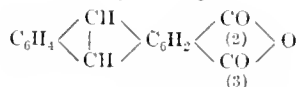
In yellow lustrous plates melting at  $280^\circ$ , insoluble in water, but soluble in alcohol and ammonia. The ammonium, calcium, lead, and silver salts, all red in colour, of the above acid are described.

*Anthracene-o*-dicarboxylic Acid—



In greenish-yellow crystals, melting at  $345^\circ$ , insoluble in water, but soluble in alcohol and ammonia. It gives, on subliming—

*Anthracene-o*-dicarboxylic Anhydride—



The calcium (brownish-green), lead (yellow), and silver (brownish-yellow) salts are described.

*m*-Dimethylanthracene—



By condensation of methylphenylketone (J. prakt. Chem. 35, 480) or by reduction of *m*-dimethylanthraquinone with zinc dust and ammonia (Ber. 20, 1365; see also Bull. 44, 167, and Ann. Chem. 234, 240), the picric acid derivative was similar to the *o*-derivative. A bromine derivative is also described.

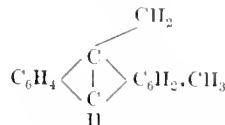
*m*-Dimethylanthraquinone—



melting at  $162^\circ$ , by treating recrystallised *m*-xylene-*o*-benzoic acid with strong sulphuric acid.

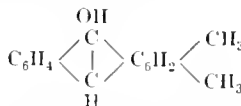
By reduction of *m*-dimethylanthraquinone with zinc dust and ammonia, a hydrocarbon was obtained in small colourless plates, melting at  $85^\circ$ , soluble in hot alcohol, darkening in presence of air and light. By treatment with a saturated alcoholic solution of picric acid a derivative was formed, melting at  $135^\circ$ , and soluble with difficulty in alcohol.

From the analysis of the hydrocarbon and the above derivative, the author concludes that it is *m*-dimethylanthracene,  $C_{16}H_{12}$ .



Generated from a pre-existing *m*-dimethylanthranol by abstraction of its hydroxyl and an atom of hydrogen (from its ortho-methyl group) as water.

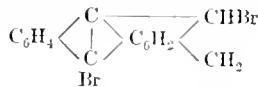
*m*-dimethylanthranol,  $C_{16}H_{14}O$



was obtained in varying quantities by the same reduction in small colourless needles, melting at  $155^\circ$ , darkening in air, insoluble in water, but soluble in alcohol, carbon disulphide or warm alkalis.

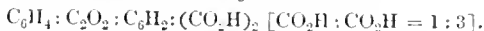
The bromination of *m*-dimethylanthracene gave no addition product, but a substitution one was formed, namely:—

*m*-Dimethylanthracene dibromide,  $C_{16}H_{10}Br_2$ —



In sulphur yellow prisms, melting at  $175^\circ$  and decomposing, giving off hydrobromic acid, soluble in carbon disulphide and acetone, and only with difficulty in ether and alcohol. The author confirms the above formula by an experiment to show the absence of a bromine atom in the benzene ring.

*Anthraquinone-m*-dicarboxylic Acid—



In yellow needles, melting at  $330^\circ$ , insoluble in water, and soluble only with difficulty in any of the usual solvents.

By oxidation of *m*-dimethylanthraquinone with dilute nitric acid.

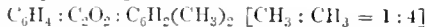
Its neutral salts of ammonium, potassium, sodium, calcium, barium, lead (all reddish coloured), copper (deep green), and silver (rose coloured) are described. The chloride is also described. The amide crystallised badly, and showed no characteristics.

*Anthracene-m*-dicarboxylic Acid—



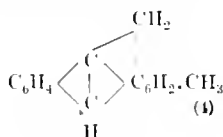
A brownish yellow crystalline powder, melting at  $330^\circ$ , and easily soluble in all solvents. By reduction of the foregoing acid with zinc dust and ammonia. The ammonium and copper (both green) and the silver (brownish yellow) salts are described, as is also the chloride.

*p*-dimethylanthraquinone—

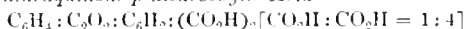


In yellow needles melting at  $118^\circ-119^\circ$ , soluble with difficulty in alcohol, but easily in benzene and xylene. From *p*-xylyl-*o*-benzoic acid by abstraction of water with strong sulphuric acid.

By reduction of the foregoing with zinc dust and ammonia a product crystallising badly was obtained. This yielded a *picric acid derivative* in deep red needles, melting at 129°. By decomposition of this by sodium hydrate a yellow powder was obtained, soluble in alcohol and crystallising in very small yellow plates, melting at 63°. This proved not to be *p*-dimethylantracene, but a hydrocarbon poorer in hydrogen, viz.,  $C_{16}H_{12}$ , and probably of the formula—

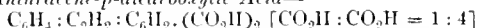


#### Anthraquinone-*p*-dicarboxylic Acid—



In yellow crystals, melting at 300°, soluble with difficulty in water, but easily in alcohol. By oxidation of *p*-dimethyl-anthraquinone with dilute nitric acid. The *alkaline salts* are reddish. The *calcium, lead and silver salts* are also all reddish coloured.

#### Anthracene-*p*-dicarboxylic Acid—



A clear brown crystalline powder, melting about 320°. The following neutral salts are described: The *alkaline salts*, blue or bluish green; the *calcium salt* (brown); the *lead salt* (yellowish green); and the *silver salt* (brownish yellow).—D. A. S.

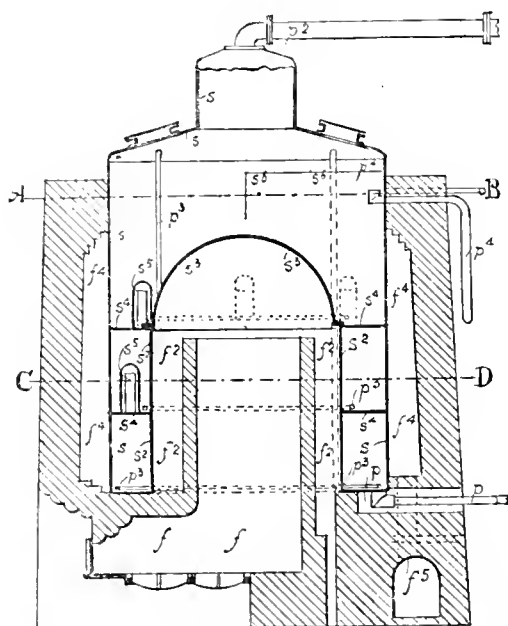
### PATENT.

*Improvements in the Distillation of Mineral Oils, and in the Apparatus employed therefor.* W. Young, Peebles, and G. T. Beilby, Slateford. Eng. Pat. 4597, March 16, 1889. 8d.

This is a process for continuous distillation, wherein it is claimed the more volatile oils assist in the distillation of the less volatile oils, and that by the construction of the still, increased rapidity of distillation is secured.

Fig. 1 shows an elevation of the still in section. Fig. 2 shows a horizontal section on the line A, B.

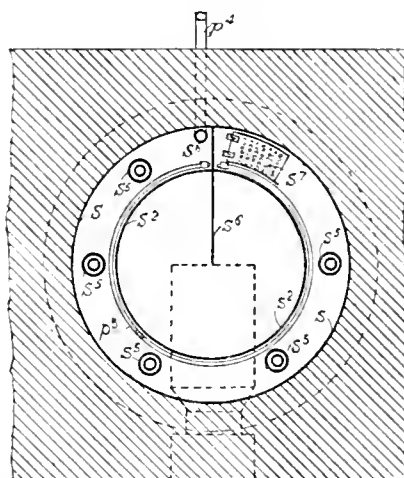
Fig. 1.



$s$  and  $s^2$  are the outer and inner shells of the still, of malleable iron.  $s^3$  is the crown of inner shell, of cast iron,

$s^4$  partitions dividing the still horizontally into three compartments,  $s^5$  tubular openings covered with loose caps,  $s^6$  vertical partitions to cause circulation of oil,  $s^7$  perforated hinged doors in the partitions,  $p$  oil feed pipe,  $p^2$  oil vapour

Fig. 2.



pipe leading to the regenerative condensers,  $p^3$  perforated steam pipes round each of three compartments,  $p^4$  residue or overflow pipes leading to the coking stills.

In working, oil is fed continuously in at the bottom, and in its progress upwards is subjected to an increasing temperature. The more volatile parts are at once converted into vapour, and in their passage through the still caused to bubble through or come intimately in contact with the less volatile oils, and so take up and carry over the less volatile vapours. Less steam is thus required than in the ordinary system.—D. A. S.

## IV.—COLOURING MATTERS AND DYES.

*The Action of Light in Colouring pure Aniline.* P. Werner. Bull. Soc. Ind. de Mulhouse, June—July 1889, 331—332.

It is well known that when aniline is exposed to air and light it becomes coloured after some time, first yellow, then red, and finally brown. It was deemed of interest to try to discover if this colouration was due to partial oxidation of the aniline, and to study the influence of light upon this colouration. To clear up the question the following trials were made. Pure colourless aniline had been preserved in vacuo, and in an atmosphere of air and of oxygen, one portion in the dark and the other exposed to diffused daylight for three months and a half.

### 1.

#### In Diffused Daylight.

1. *In vacuo.*—The aniline remains absolutely colourless.
2. *Air.*—Yellowish brown colouration. Absorption for 1 gm. of aniline 0.4 cc. on 9 cc. of air.
3. *Oxygen.*—Intense colouration, brownish red. Absorption for 1 gm. of aniline 1.1 cc. on 8 cc. of oxygen.

### II.

#### In the Dark.

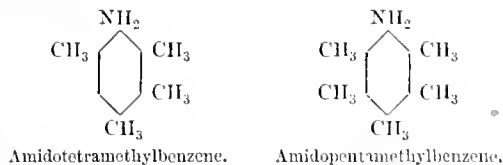
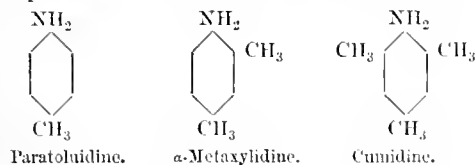
1. *In vacuo.*—Aniline remains absolutely clear.
2. *Air.*—Very slight colouration. Absorption inappreciable.
3. *Oxygen.*—Colouration feeble, yellowish. Absorption very slight.

The colouration is thus proved to be due to the action of light. In the light it is also shown that an absorption of oxygen takes place, i.e., oxidation, and so the effect is produced by the combined action of oxygen (air) and light.

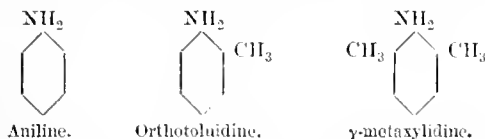
*On some Colouring Derivatives of Triphenylmethane.*  
E. Noelting. Bull. Soc. Ind. de Mulhouse, 1889, Aug.—Sept. 436—443.

The conditions under which magenta is formed have been particularly studied by Hoffmann, Rosenstiel, Emil, and Otto Fischer and Rosenstiel and Gerber. Rosenstiel and Gerber distinguish three classes, according to the manner in which the homologues of aniline behave towards oxidising agents.

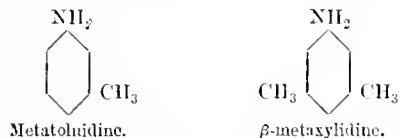
**First Class.**—Bases which when oxidised with arsenic acid by themselves do not yield colouring matters, but do so in presence of aniline—



**Second Class.**—Bases which on treatment with arsenic acid yield no colouring matters, but which do when oxidised in presence of a base of the first class—



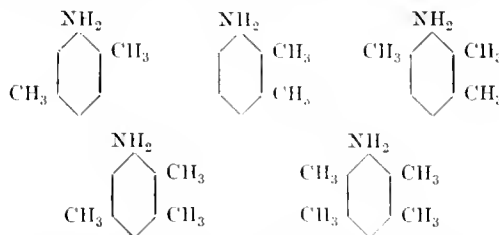
**Third Class.**—Bases which on oxidation with arsenic acid yield no colouring matters like magenta, neither by themselves nor in presence of any of the two previous classes—



In the first class one methyl group is in the para-position to the amido group, whilst in the bases of the second and third class the para-position is free. In the second class the methyl groups are in the ortho-position, and in the third class in the meta-position to the amido group. E. and O. Fischer's experiments have proved that in the simplest rosaniline the three amido groups are in the para-position to the fundamental carbon atom. The complete analogy of the homologous rosanilines with the first having been proved, it can be readily understood why the members of the first group when oxidised by themselves or with each other do not yield rosaniline, but do so when oxidised in presence of aniline.

In the bases of the second class the para-position is free, and one or two ortho-positions are occupied. It is clear that these bases cannot yield rosanilines, unless they are oxidised with a base of the first group. Neither can the bases of the first class produce magentas, but no reasons yet given can explain why they should not be able to produce rosanilines, when oxidised with the members of the first class. The experiments of Monnet, Reverdin, and

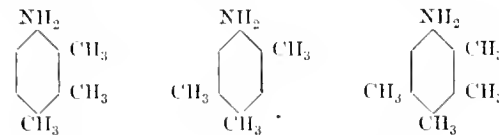
Noelting and of O. Fischer and Koek, have, however, confirmed this experience. Now the question arises whether amido compounds, in which the ortho- and meta-position is occupied, belong to the second or third class. Such amines are—



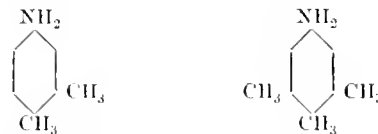
Experiments made with all these bases in their absolutely pure state prove that they belong to the third class. Not a trace of colouring matter was obtained when oxidised by themselves or in presence of para-toluidine,  $\alpha$ -metaxylydine, or mesidines. The presence of a methyl group in the meta-position to the amido group renders these bases incapable of forming rosanilines.

Another question arose, whether the presence of a methyl group in the meta-position in the bases of the first class would influence their capacity for forming rosanilines. In the first three bases of this class the meta-positions are free, in the last two the ortho- and one or two meta-positions are occupied. But how meta-methylated paratoluidines would act remained to be seen.

The following are ortho- and meta-methylated paratoluidines—



whilst—



are only meta-methylated.

All these bases yield rosanilines in abundance, when oxidised with aniline, orthotoluidine, and  $\gamma$ -metaxylydine.

The author further shows that there are 30 isomeric rosanilines possible, if 1 mol. of a para base be oxidised with 2 mols. of either aniline, orthotoluidine or  $\gamma$ -metaxylydine, but that this number may be increased to 60, if instead of taking 2 mols. of the same base of the second class, 1 mol. of aniline and 1 mol. of orthotoluidine, &c. be used. However, some of the latter 30 compounds are identical either among themselves or with bodies of the first 30; there exist in reality only 52 isomeric and homologous rosanilines.

It remains to be decided whether a rosaniline can be obtained by oxidising 1 mol. of a base of the first class with 1 mol. of a base of the second class, and 1 mol. of a base of the third class.—A. L.

*Researches on Colouring Matters of the Triphenylmethane Group.* E. Noelting. Bull. Soc. Ind. de Mulhouse, 1889, Aug.—Sept. 444—446.

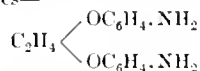
*Condensation of Tetramethyldiamidodiphenylcarbinol with Meta-substituted Amines.*—Kern has discovered that tetramethyldiamidodiphenylcarbinol can be condensed with amines, and that the compound yields on oxidation derivatives of rosaniline. He used as amines the primary, secondary, and tertiary derivatives of aniline, orthotoluidine, and  $\alpha$ -naphthylamine, but no meta-substituted compounds. The author prepared the condensation products from meta-toluidine, paraxylydine,  $\beta$ -orthoxylydine,  $\beta$ -metaxylydine



amidines of Edler and Mayer, and the crystallised duridine with tetramethylamidodiphenylcarbinol, all of which form on oxidation with chloranil blue-violet colouring matters. The secondary and tertiary compounds of these meta-substituted bases yield also colouring matters similar to those obtained from the primary amines.—A. L.

*Note on the Ethylenic Ethers of the three Amido-phenols and some Colouring Matters from them.* C. Krohn. Bull. Soc. Ind. de Mulhouse, 1889, Aug.—Sept. 16—17.

THE three amines—



are distinguished from each other by the colour with which they dye woody fibre (lignin). The para-compound dyes woody fibre orange, the meta- and ortho-compound dye it yellow. The azo-colouring matters prepared from these bases differ in their affinity for vegetable fibre. The para-compounds dye cotton directly very well, the meta derivatives badly, and the ortho-compounds not at all. By combining ethylene-para-amidophenol with  $\alpha$ -naphthol- $\alpha$ -sulphonic acid, a beautiful red, fast to acids, is obtained; with resorcinol, an orange; with salicylic acid, a fine yellow. With naphthionic acid it forms an orange, with  $\beta$ -naphthylamine- $\delta$ -sulphonic acid, a reddish orange, and with Brömer's  $\beta$ -naphthylamine sulphonic acid a still redder orange. These three colours are altered by acids.—A. L.

*The Synthesis of Metaphenylene-diamine from Resorcinol and Ammonia.* A. Seyewitz. Compt. Rend. 109, 814—817.

A MIXTURE of resorcinol and ammoniacal calcium chloride, obtained by saturating calcium chloride with dry ammonia, is heated in a sealed tube for three hours between 280° and 300° C. The mass is treated with cold water, and the aqueous solution repeatedly extracted with half its volume of ether. The ethereal solutions are saturated with hydrochloric acid gas, when the base crystallises out as the hydrochloride. The yield is 60 grms. per 100 grms. of resorcinol taken. The author gives details of variations in time and temperature of the reaction, but prefers the figures as given above.—S. G. R.

*Synthesis of Dihydroxydiphenylamine and of a Reddish-Brown Colouring Matter.* M. Seyewitz. Compt. Rend. 109, 946—949.

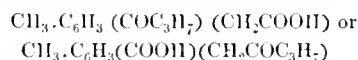
A MIXTURE of resorcinol with four times its weight of ammoniacal calcium chloride were heated for about 10 hours in sealed tubes from 190° to 260°. The tubes were found to contain a deep yellow colouring matter, which was powdered, digested with water, and filtered. The filtrate was neutralised with hydrochloric acid; this precipitated a further quantity of the same substance, which had remained on the filter. After drying in vacuo over sulphuric acid, the residue was dissolved in a little alcohol and filtered. On now adding water to the red solution, which also shows a fine green fluorescence, brown flakes separate out. They are purified by repeated crystallisations. An analysis led to results justifying the formula  $\text{HO}\cdot\text{C}_6\text{H}_3\cdot\text{NH}=\text{C}_6\text{H}_4\cdot\text{OH}$ , and this was corroborated by the behaviour of the body towards acids, alkalis, nitrite of soda, and diazo-bodies. Also, on heating with zinc powder, diphenylamine was obtained.

By heating the above-mentioned mixture of resorcinol and ammoniacal calcium chloride for eight hours at 300°, a reddish-brown dye is obtained. This is readily diazotised, and then reacts on phenols with formation of other dyes. On cotton mordanted with tannin and tartar emetic a fast brown is produced. Wool which has been treated with bichromate is coloured in good shades, which are fast to milling.—A. R.

*On a New Class of Diacetones.* A. Béhal and V. Auger. Compt. Rend. 109, 970—973.

MALONYL chloride, as well as ethyl- and methyl-malonyl chloride, react with aromatic hydrocarbons with formation of  $\beta$ -diacetones  $\text{R}\cdot\text{CO}\cdot\text{CHX}\cdot\text{CO}\cdot\text{R}$ . By using ethyl-malonic acid, substances are obtained giving characteristic blood-red solutions on adding an alkali or an alkaline carbonate. The best results have been obtained with *m*-xylene and the chloride of ethylmalonic acid. The diacetone obtained by the aid of aluminium chloride melts at 63°. Its formula is  $\text{C}_{13}\text{H}_{14}\text{O}_5$ . The red colouration produced by alkalis is due to the H-atom which is attached to the carbon atom situated between the two carbonyl groups. If the H-atom be replaced by ethyl, the diketone is no longer coloured red by alkalis. We may therefore regard these diketones as containing a new chromogen group. The tinctorial properties of these bodies are lost by oxidation with ferrocyanide, when tetraketones are produced.

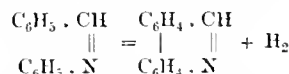
The diketone from ethylmalonic acid yields a dioxime melting at 235° with hydroxylamine, which confirms the presence of the two carbonyl groups. Heated with baryta water to 200°, methylbutyrylphenylacetic acid is produced, of the formula



On oxidising this acid, propionic acid and a dibasic acid  $\text{C}_6\text{H}_3(\text{COOH})(\text{CH}_2\text{COOH})$  result; the latter melts at 178° with loss of  $\text{CO}_2$ . These new diketones therefore possess the remarkable property that one of their carbonyl groups unites with water to form an acid of the same number of carbon atoms as the diketone itself.—A. R.

*Phenanthridine.* A. Pietet and H. I. Ankersmit. Ber. 22, 3339—3344.

HERETO only two of the five isomeric compounds, formed theoretically by the substitution of a nitrogen atom for a CH-group in phenanthrene, have been known. The authors have prepared a third of these, differing from the other two previously known in containing its nitrogen atom in the central ring, and give it the name, already suggested by Etard and Bernthsen, of phenanthridine. The new compound was obtained by passing the vapours of benzylidene-aniline through a tube heated to bright redness, the chemical change being represented by the following equation:—



A brown oil formed the chief product of the reaction. This was examined for the presence of phenanthridine by first submitting it to distillation, and testing the portion boiling above 250°. On treating it with warm dilute hydrochloric acid, and adding mercuric chloride to the solution so obtained, a voluminous precipitate was formed, which was recrystallised from hot water, and washed with boiling alcohol. The base was then liberated from the washed salt, and, after further purification by distillation with superheated steam and crystallisation from dilute alcohol, was analysed, and its chief properties examined, with the results shown in the following table:—

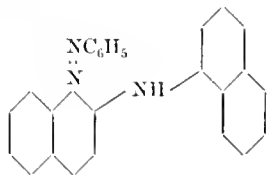
	Acridine.	Phenanthridine.
Melting point, .....	107°	104°
Platinum double salt	Small, stellate needles. Melting point, above 225°.	Small, aggregate needles. Melting point, above 225°.
Gold double salt ....	Long needles, sparingly soluble in hot water.	Long needles, insoluble in hot water.
Mercury double salt	Long stellate needles. Melting point, 225°.	Long stellate needles. Melting point, 130°.

	Acridine.	Phenanthridine.
Bichromate.....	Short needles, almost insoluble in hot water.	Long, fine needles, fairly-easily soluble in hot water.
Picrate.....	Prismatic needles. Decompose towards 200°.	Fine, thin needles. Undecomposed at 220°.
Nitrite.....	Small needles.	Floccy amorphous precipitate.
Sodium sulphite added to solution of the hydrochloride.	Precipitate consisting of small, brown-red needles.	No precipitate.
Potassium iodide added as the last.	Precipitate consisting of long yellow needles.	Yellow, granular precipitate.
Reduction with tin and hydrochloric acid.	Compound without basic properties.	Basic hydro-derivative, crystallising from alcohol in small white needles of melting point near 100°, converted by action of nitrous acid into a nitrosamine.

—E. B.

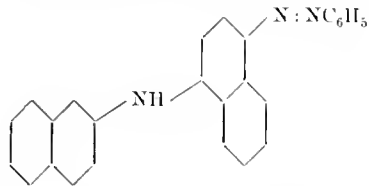
*The Combination of  $\alpha$ - $\beta$ -Dinaphthylamine with Diazo-benzene.* P. Matthes. Ber. 22, 3341—3348.

On adding a solution of diazobenzene to one of  $\alpha$ - $\beta$ -dinaphthylamine in acetone in the cold and in presence of sodium acetate, there is formed the azo-compound of the following constitution:—

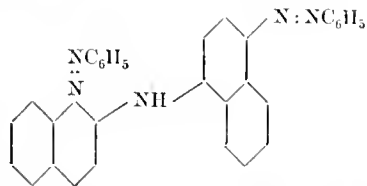


The diazo-nucleus enters the  $\alpha$ -position next to the substituted  $\beta$ -position.

But when the combination of the above compounds is effected in the presence of hydrochloric acid and at a temperature of 40°—50° three azo-compounds are produced. Not only does the diazo-rest enter the substituted  $\beta$ -naphthylamine molecule in the  $\alpha$ -position as above, but it enters the substituted  $\alpha$ -naphthylamine in the para-position, forming the compound represented below:—



In addition a small quantity of a disazo-compound is formed:—

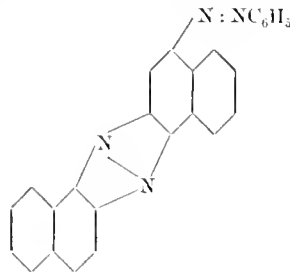


The disazo-compound being much less soluble in alcohol than the two azo-compounds, can be easily separated from the mixture. As the solubilities in most solvents of the two mono-azo-compounds are almost the same, the separation of these is more difficult. Benzene alone dissolves

more of the  $\alpha$ -compound than the  $\beta$ -compound and by repeatedly crystallising from this solvent the latter compound is obtained pure. It crystallises in long, floccy, deep-red needles, melting at 167°. On treatment with mineral acids it is changed into an azine. Advantage is taken of this in the preparation of the pure  $\alpha$ -compound. The acetic acid solution of the two mono-azo-compounds is treated with mineral acid, whereby the  $\beta$ -compound is converted into an azine, the  $\alpha$ -compound being merely converted into the corresponding salt. Both compounds are precipitated by water, then the  $\alpha$ -salt is separated by solution in alcohol in which the azine is practically insoluble. By addition of ammonia to the alcoholic solution the free  $\alpha$ -azo-compound is separated. After recrystallisation from hot alcohol it is obtained in the form of short, thick, orange prisms, melting at 137°.

The disazo-compound crystallises from benzene in long, floccy, violet needles, with a melting point of 238°.

It was further found that the disazo-compound was easily formed from either mono-azo-compound by treatment with diazobenzene chloride. Also, when a sufficient amount of solvent is present, the disazo-compound produced, remains dissolved in the cold, which explains the apparent non-formation of this body in the first reaction mentioned above. The violet disazo-body was boiled with acetic acid for a long time and a crystalline brown-yellow substance obtained therefrom, melting, after crystallisation from benzene, at 287°. On reduction with zinc dust and hydrochloric acid the latter substance was converted into a eurdiodine, which fact, in conjunction with the analytical figures, leaves no room for doubt that the brown-yellow substance is an azo-azine of the following constitution:—



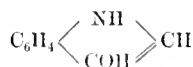
—E. B.

*Synthesis of Indigo from Monobromacetanilide.* W. Flimm. Ber. 23, 57—60.

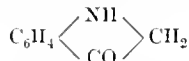
THE action of caustic alkalis on bromacetanilide differs according to the condition in which the two bodies react on each other. Thus aqueous potash causes the absolute breaking up of the molecule with formation of carbamine. By using alcoholic potash Abenius obtained diphenyldiketopiperazine. Again, if the calculated quantity of sodium be added to an absolute alcoholic solution of bromacetanilide, after some time there separates a thick yellowish oil, ethoxyacetanilide,  $C_6H_5NH(COCH_2OC_2H_5)$ .

Finally, the author has discovered that when a small quantity of monobromacetanilide is mixed with an equal weight of dry caustic potash, and the mixture rapidly heated to a high temperature until a homogeneous reddish-brown melt is obtained, then on dissolving the latter in water and a little ammonia, a greenish solution is obtained, which rapidly changes to blue and after a short time a blue colouring matter separates. If the melt be dissolved in dilute acid and ferric chloride added, indigo separates out at once. The fact of its really being indigo was proved by a great variety of characteristic reactions, including also an examination of the absorption spectrum, which absolutely agreed with that of indigo. The yield is only 4 per cent.

The chemical reactions whereby the formation of indigo is in this case brought about, are explained thus. Starting from bromacetanilide, indoxyl—

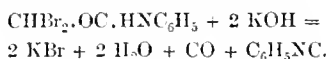


is first produced, or possibly a pseudo indoxyl—



and the latter is then oxidised to indigo. The syntheses of this colouring matter from *o*-amidoacetophenone which is brominated in the side-chain is a well known one. Now this substance is isomeric with bromoacetanilide. It is, however, not likely that the production of indigo from the latter body is preceded by the formation of the former, since it is evident that the first reaction that will take place is the splitting off of hydrobromic acid.

Attempts to obtain dyes similar to indigo from bromoaceto-*α*- or *β*-naphthalide or from bromoacetyldiphenylamine or from bromoacetoorthotoluide were unsuccessful. From bromoaceto-*para*-toluide, however, dimethyl indigo was readily prepared. In its outward properties the latter cannot be distinguished from indigo. By distilling it with excess of caustic alkali, pure *para*-toluidine distils over. The reason why indigo is not produced from the orthotoluide is evidently due to the fact that a methyl group occupies the ortho position in the benzene nucleus. The carbonyl group is consequently unable to enter at this point. This fact the author considers a further proof of the groups CO and NH in indigo being attached to adjoining carbon atoms. Di-bromoacetanilide when strongly heated with caustic potash or alcoholic potash yields no indigo, but considerable quantities of carbamine according to the equation—



—A. R.

*On α-Naphthylamine-ε-Disulphonic Acid.* G. Schnltz.  
Ber. 23, 77.

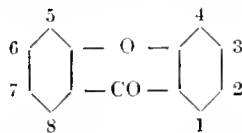
THE preparation of *α*-naphthylamine-*ε*-disulphonic acid has been patented by the three firms: the "Actiengesellschaft für Anilin fabrikation" (Ger. Pat. 45,776), "Ewer and Piek" (Ger. Pat. E. 2318), and the "Badische" (Ger. Pat. B. 9514). In each patent the method employed for the production of the above acid is identical, consisting in nitrating *α*-*β*-naphthalenedisulphonic acid and then reducing the resulting *α*-nitro-*α*-*β*-naphthalenedisulphonic acid. The only appreciable difference between the above three patents is that the first-named firm employs a mixture of *α*-*α*- with *α*-*β*-naphthalenedisulphonic acid, thereby finally obtaining both *α*-naphthylamine *δ*- as well as *ε*-disulphonic acid, about the same quantity of each being produced.

Besides these acids other sulphonic acids are obtained as by-products in the above processes, and amongst them the author was able to identify *β*-naphthylaminedisulphonic acid.

—A. R.

*The Euxanthone Group.* C. Graebe. Annalen, 254,  
265—303.

THESE compounds are derivatives of triphenylene-ketone oxide (xanthone) for which the subjoined constitutional formula is regarded by the author as definitely established:—



the numerals indicating the order of designation of the substituting groups, which he has adopted for the formulation of the derivatives.

The compounds are of special technical interest from the occurrence of a typical representative (Euxanthic Acid) in the well-known Indian Yellow or Purree. This colouring matter is a urinary secretion of cows fed upon mango leaves, and exclusively produced by a sect of the Gwalis in the Monghyr district of Western India. Upon this peculiar process of manufacture the author quotes in full

a report by T. N. Mukharji to the India Office of August 1883 (see also Jour. Soc. Arts, 32, 16). The Purree separates spontaneously from the urine, but the separation is promoted by applying heat. Each cow, on this diet, yields on the average 3.4 litres of urine daily, from which 50—60 grms. of the Purree are obtained. The physiology of the process has not been as yet elucidated. Euxanthic acid, the chief constituent of the yellow, has been shown by Kostanecki (Ber. 19, 2918; this Journal, 1887, 507) to be formed from euxanthone, in the passage of this compound through animal organisms; but investigation of mango leaves have so far failed to reveal the presence of any constituent resembling euxanthone.

A selected specimen of Purree gave the following results on analysis:—

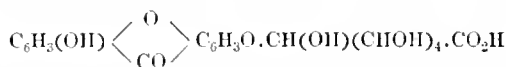
Euxanthic acid.....	51.0
Silica and alumina.....	1.5
Magnesium.....	4.2
Calcium.....	3.4
Water and volatile constituents (160°—170° C.)	39.0
	99.1

The firm of MM. Lefranc and Cie (Paris) supply several varieties of Purree, designated in order of inferiority by letters from A to G, the corresponding prices varying from 300 frs. to 50 frs. per kilo. Subjoined are the results of the author's analyses of these products (Compare Ber. 16, 864):—

	A.	B.	C.	D.	G.
Euxanthic acid .....	72.3	70.9	64.3	59.3	53.34
Euxanthone.....	0.0	1.1	2.8	7.4	31.0
Mg .....	5.4	4.9	4.9	4.6	3.7
Ca.....	1.8	2.4	2.6	3.3	3.7

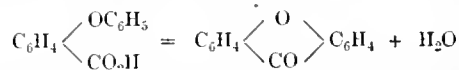
From these analyses it is evident that euxanthone is present in the inferior sorts only, in which also the ratio Ca : Mg is relatively high.

*Euxanthic Acid* is easily prepared in the pure state by first digesting a Purree of good quality with dilute hydrochloric acid and dissolving out the acid from the residue by treatment with ammonium carbonate. The solution is filtered and treated with an acid, when the compound separates. It forms two classes of salts,  $\text{C}_{19}\text{H}_{16}\text{O}_{11}\text{M}'$  and  $\text{C}_{19}\text{H}_{16}\text{O}_{11}\text{M}'_2$ . It is a condensed derivative of euxanthone and glycuronic acid, containing the COOH group of the latter unaltered, but only one of the OH groups of the euxanthone. The author proposes the subjoined constitutional formula (assuming Schmiedeberg and Meyer's formula for glycuronic acid)—



the glycuronic acid, when split off (by hydrolysis) contains the group  $\text{CH}(\text{OH})_2$  which is at once resolved into  $\text{COH} + \text{H}_2\text{O}$ , and this explains the fact that the hydrolysis of the euxanthic acid does not appear to involve combination with  $\text{H}_2\text{O}$ .

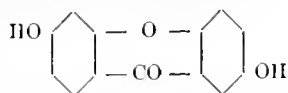
*Xanthone* (diphenyleneketone oxide) is the chief product (45 per cent.) of the action of heat upon salol, phenylsalicylic acid being first formed, then dehydrated.



Its characteristic reactions are (1) reduction to methylenediphenyl oxide by heating with zinc dust; and (2) conversion into dioxybenzophenone by heating with alcoholic potash at 180°, by which its constitution is established.

*Euxanthone* fused with potassium hydrate yields resorcinol in addition to quinol (hydroquinone), the two phenols in

approximately equal quantity, whence the author deduces the annexed constitutional formula—



Euxanthone, identical in every respect with that obtained from Indian Yellow, was obtained synthetically by heating  $\beta$ -resoreic acid and quinol carboxylic acid together with acetic anhydride.

*Euxanthonic Acid* was obtained by heating euxanthone with three times its weight of caustic potash and some water, so that gradually the temperature is brought up to  $260^{\circ}$ — $270^{\circ}$ . Euxanthonic acid fuses at  $200^{\circ}$ — $202^{\circ}$ , losing water, and gradually forming euxanthone.

The paper concludes with a tabular résumé of all the members of the group at present investigated.—C. F. C.

*Studies on the Constitution of the Tri-derivatives of Naphthalene. No. 1. The Constitution of Betanaphthol- and Betanaphthylaminedisulphonic Acids R and G. Naphthalenemetadisulphonic Acid.* H. E. Armstrong and W. P. Wynne. Proc. Chem. Soc. 1890 (77), 11—15.

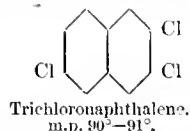
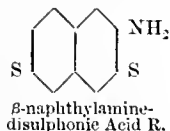
$\beta$ -NAPHTHOLDISULPHONIC acid G (Gelb), like the "Bayer" modification of  $\beta$ -naphtholmonosulphonic acid, interacts but slowly with diazo salts, while the corresponding  $\beta$ -naphthylaminedisulphonic acid, like the "Badische" modification of  $\beta$ -naphthylaminemonosulphonic acid, is incapable of forming azo dyes with the majority of diazo salts.

In converting the dichloronaphthalenes into sulphonic acids, the authors noticed that isomeric changes occurred in several instances (Proc. Chem. Soc. 1889, 52, 120). As a contribution to a study of the precise course of the change in sulphonation, it seemed desirable to make a comparative study of the influence exercised by radicles such as Cl, OH, and  $\text{NH}_2$  on the formation of disulphonic acids.

With regard to the dichloronaphthalenes, see Proc. Chem. Soc. 1888, 104. In the case of the trichloronaphthalenes, no fewer than 14 are possible, which will ultimately serve as reference compounds for tri-derivatives of naphthalene. It is very necessary to characterise each compound, owing to the close similarity which obtains between many of the isomerides; no fewer than four, for example, melt at nearly the same temperature, about  $90^{\circ}$ — $92^{\circ}$ .

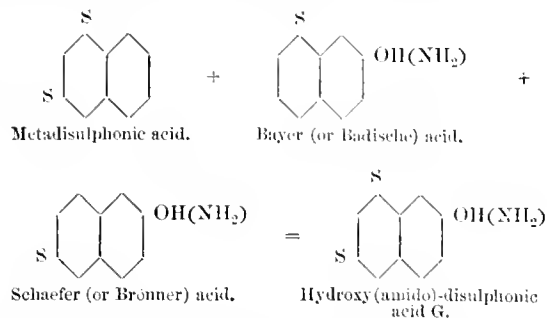
*Constitution of the  $\beta$ -naphthylaminedisulphonic Acid R.* —Duisberg and Pfitzinger inferred that this acid was derived from the  $\alpha$ -naphthalenedisulphonic acid of Ebert and Merz, as the disulphonic acid obtained from it on displacing the  $\text{NH}_2$  group by v. Bayer's hydrazine process gave the dihydroxynaphthalene corresponding to the  $\alpha$ -acid on fusion with potash. The authors thought it desirable to amplify the evidence in order to meet the possible objection that isomeric change may occur during the fusion, as in the case of benzene-derivatives. They find on directly comparing the disulphonic acid with the Ebert and Merz  $\alpha$ -acid that the two behave alike.

By adopting Sandmeyer's method, the  $\alpha$ -acid was converted into a chlorodisulphonic acid, and this on distillation with  $\text{PCl}_5$  yielded the corresponding trichloronaphthalene.

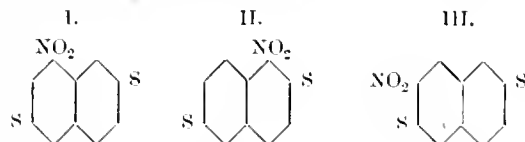


*Constitution of  $\beta$ -naphthylaminedisulphonic Acid G.* —A quantity of this acid was converted into the naphthalenedisulphonic acid by the hydrazine method and a new acid was thus obtained, which was proved to be the metadisulphonic acid of naphthalene. The relative position of the sulphonic radicles being thus determined, their positions relatively to the  $\beta$ - $\text{NH}_2$  radicle may be inferred from the facts that the hydroxy-G-acid is obtained as direct sulphonation-product of the Bayer modification of  $\beta$ -naphthol-

sulphonic acid, and that it yields, as Caro first proved, the isomeric Schaeffer acid when reduced by sodium amalgam. Although the sulphonic acids are usually reducible to naphthalene in the manner first pointed out by Otto in the case of naphthalene- $\alpha$ -sulphonic acid, the  $\beta$ -sulphonic derivatives are far less readily affected; the  $\text{NH}_2$  radicle also exercises a special protecting influence in many cases. Superposing the various results, the constitution of the G-acids is shown to be as follows:—



The G-acid is convertible by Sandmeyer's method into a chlorodisulphonic acid, the chloride of which crystallises from benzene in long radiate needles, melting at  $170^{\circ}$ ; this is converted by  $\text{PCl}_5$  into a trichloronaphthalene which crystallises in very slender needles melting at  $113^{\circ}$ , identical with that obtained by Alén (Ber. Referate, 1884, 437) from an  $\alpha$ -nitro acid prepared by nitrating the chloride of the  $\beta$ -disulphonic acid of Ebert and Merz. This result confirms the conclusion above arrived at, as of the three possible formulæ of Alén's acid:—



II. and III. are excluded by the fact that the trichloronaphthalene corresponding to III. melts at  $90^{\circ}$ — $91^{\circ}$  (*vide supra*), and that corresponding to II. also at about the same temperature (Proc. Chem. Soc. 1889, 52).

The results now obtained show that, as in the case of the Bayer and Badische monosulphonic acids, the action of diazo-salts on the G-disulphonic acids is either retarded or prevented by the "protecting influence" exercised by an  $\alpha$ -1'-sulphonic group.

The properties of the five known naphthalenedisulphonic acids are summarised in the appended table:—

(See top of next page.)

*Studies on the Constitution of Tri-derivatives of Naphthalene. No. 2.  $\alpha$ -Amido-1:3'-Naphthalenedisulphonic Acid.* H. E. Armstrong and W. P. Wynne. Proc. Chem. Soc. 1890 (77), 15—16.

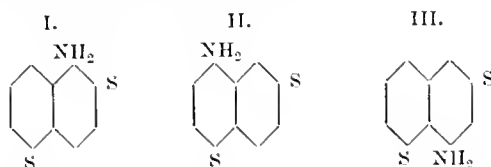
THE 1:3'-naphthalene- $\alpha$ - $\beta$ -disulphonic acid (Proc. Chem. Soc. 1886, 231; 1889, 10) has acquired considerable interest on account of the peculiar behaviour of the amido- and corresponding hydroxy-acid prepared from it and the dye-stuffs which the amido-acid affords (Bernthsen, Ber. 22, 3327). This amido-acid has been patented by the Aetiengesellschaft für Anilinfabrikation, in whose laboratory it was first prepared by Andresen (Schultz, Ber. 23, 77) by nitrating the  $\alpha$ - $\beta$ -disulphonic acid above referred to, and reducing the nitro-derivative. Technically, it has become known as  $\alpha$ -naphthylamine- $\epsilon$ -disulphonic acid.

The authors first satisfied themselves that the amido-acid had been prepared from their  $\alpha$ - $\beta$ -disulphonic acid by reducing it by the hydrazine method; the acid obtained gave a sulpho-chloride, melting at  $127^{\circ}$ , and a dichloronaphthalene, melting at  $48^{\circ}$ .

—	1 : 4.	2 : 2.	2 : 3.	1 : 3.	1 : 3.
$S = SO_3H$ .					
$(SO_3H)_2 \dots$	Armstrong ( <i>Chem. Soc. Jour.</i> , 1871, 173; <i>Ber.</i> 1882, 205); Armstrong and Wynne ( <i>C. S. Proc.</i> 1886, 231; 1887, 42); Ewer and Pick, Ger. Pat. 41,934.	Ebert and Merz ( <i>Ber.</i> 9, 592).	Ebert and Merz ( <i>Ber.</i> 9, 592).	Armstrong and Wynne ( <i>C. S. Proc.</i> 1886, 231); Armstrong ( <i>C. S. Proc.</i> 1889, 10); Ewer and Pick, Ger. Pat. 45,229.	Armstrong and Wynne.
$(SO_3)_2Ba \dots$	+ 4 $H_2O$ , granules consisting of microscopic needles.	+ 2 $H_2O$ , long, broad needles.	+ $H_2O$ , "crusts"	+ 4 $H_2O$ , granules consisting of microscopic needles.	+ 4 $H_2O$ , no definite form. Granular, very soluble.
$(SO_3K)_2 \dots$	+ 2 $H_2O$ , in prismatic needles	+ 2 $H_2O$ , in needles.	Needles.....	+ $H_2O$ , granules consisting of microscopic needles.	+ 2 $H_2O$ , small prismatic forms. Very soluble.
$(SO_3Na)_2 \dots$	+ 2 $H_2O$ , glistening scales ...	+ 6 $H_2O$ , in lustrous needles.	+ $H_2O$ , crystalline aggregates.	+ 7 $H_2O$ , long, broad needles	Very soluble in water.
$(SO_2Cl)_2 \dots$	Needles which become opaque. m.p. = $183^\circ$ .	Prisms which become opaque. m.p. = $158^\circ$ .	Needles. m.p. = $216^\circ$ .	Aggregates of small needles. m.p. = $127^\circ$ .	Prisms or prismatic needles. m.p. = $137^\circ$ .
$Cl_2 \dots$	Needles. m.p. = $107^\circ$ .....	Scales. m.p. = $114^\circ$ .	Flat needles. m.p. = $136^\circ$ .	Needles. m.p. = $49^\circ$ .....	Needles. m.p. = $61.5^\circ$ .

The corresponding chlorodisulphonic acid was then prepared by Sandmeyer's method; the disulphochloride of this acid melts at  $110^\circ$ ; on distillation with  $PCl_5$  it is converted into a trichloronaphthalene, which appears to be dimorphous, crystallising from alcohol either in long slender needles melting at  $87^\circ$ , or in large flat prisms melting at  $90^\circ$ .

As the amido-acid is known to yield  $\alpha$ -naphthylamine when deprived of its sulphonic groups, there are only three formulae which can be assigned to it:—



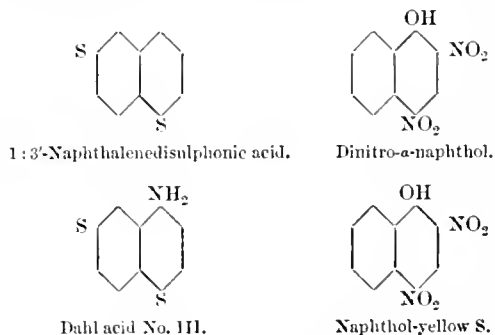
Formula I. is precluded, as the corresponding trichloronaphthalene melts at  $78^\circ$ ; formula II. is likewise precluded, as the corresponding trichloronaphthalene (*vide infra*) melts at  $66^\circ$  and  $56^\circ$ ; formula III. therefore expresses the constitution of the acid. This result is confirmed by the conclusions arrived at by Bernthsen, who finds that the corresponding hydroxy-acid exhibits the behaviour of the 1:1'-hydroxysulphonic acid, readily yielding a naphthalene-sulphonelactone-sulphonic acid.

*Studies on the Constitution of the Tri-derivatives of Naphthalene. No. 3.  $\alpha$ -naphthylaminedisulphonic Acid Dahl No. III. The Constitution of Naphthol-yellow S.* H. E. Armstrong and W. P. Wynne. *Proc. Chem. Soc.* 1890 (77), 16—18.

It is well known that when  $\alpha$ -naphthol is sulphonated by excess of sulphuric acid, it is readily converted into acids capable of yielding naphthol-yellow S, a most valuable dyestuff, the sulphonic acid of dinitro- $\alpha$ -naphthol, on treatment with nitric acid; although it is known that the Yellow is a heteronuclear derivative and that the sulphonic group is in a  $\beta$ -position, inasmuch as it yields 1:2:4-sulphophthalic acid on oxidation, the position of this group has not hitherto been determined. With the object of ascertaining the constitution of the yellow, the authors have examined the  $\alpha$ -naphthylaminedisulphonic acid No. III.

(Dahl's acid), prepared by sulphonating naphthionic acid with 35 per cent. anhydrosulphuric acid at a temperature not exceeding  $30^\circ$ , this acid being very readily converted into the yellow by diazotising and then heating with nitric acid.

When reduced by the hydrazine method, the Dahl No. III acid is found to yield the authors' 1:3'-naphthalene disulphonic acid; the sulphochloride actually prepared from the acid melted at  $127^\circ$ , and the corresponding dichloronaphthalene at  $49^\circ$ . Taking into account the fact that naphthol-yellow S is a heteronuclear  $\beta$ -sulphonic derivative, this result is alone sufficient to determine the constitution of the yellow and of the amido-acid, thus—



The chlorodisulphonic acid prepared from the Dahl acid yields a sulphochloride very soluble in benzene, from which it crystallises in small prisms; it separates from petroleum spirit in rosettes of small, apparently rectangular prisms, melting at  $107^\circ$ . The corresponding trichloronaphthalene affords a remarkable case of dimorphism: it is sparingly soluble in hot alcohol, from which it crystallises in slender needles, melting at  $66^\circ$ ; if the melting point be redetermined as soon as solidification has taken place, it is found to be  $56^\circ$ , but if determined after a longer interval,  $66^\circ$ , as in the first instance. This trichloronaphthalene should be identical with that prepared by Widman from dichloronaphthalene- $\beta$ -sulphonic chloride, though the authors have shown to be a derivative of 1:4-dichloronaphthalene, but Widman's product is said to melt at  $56^\circ$ ; the authors find, however, on preparing it according to Widman's direction,

that it also melts at 66° or 56°. By nitrating 1:3'- $\eta$ -dichloronaphthalene, Cleve obtained a nitrodichloronaphthalene, yielding a trichloronaphthalene, melting at 65°; the authors' observations show that this is in reality identical with the Widman trichloronaphthalene, and, therefore, that the behaviour of 1:3'-dichloronaphthalene on nitration is perfectly "normal," and strictly comparable with that of both  $\alpha$ - and  $\beta$ -monochloronaphthalene.

By sulphonating 1:4-chloronaphthalenesulphonic acid in the form of potassium salt by means of the theoretical proportion of sulphuric anhydride employed as 20 per cent. anhydrosulphuric acid at 100°, the authors have obtained a chlorodisulphonic acid identical with that prepared from the Dahl amido acid: showing that the  $\alpha$ -chloro-,  $\alpha$ -amido-, and  $\alpha$ -hydroxy-monosulphonic compounds behave similarly on further sulphonation; but it remains to be ascertained whether the series of changes leading up to the production of the final stable compounds is identical in the several cases.

*New Colouring Matters.* J. Soc. Dyers and Colourists, 1890, 32.

#### I.—DYES INTENDED PRINCIPALLY FOR WOOL AND SILK.

"*Clayton Cloth Red*" is the Ammonium salt of Naphthol primuline. The commercial product is a dark red granular powder, which dissolves in hot water with a clear scarlet colour. Dilute sulphuric acid completely separates the colour as an orange precipitate. It is also completely precipitated by excess of caustic soda. Stannite of soda, or hyposulphite of soda, reduces the colour, producing a yellow solution. Cotton boiled in this, passed through a bath of nitrous acid, and then into an alkaline solution of  $\beta$ -naphthol, is dyed red. *Clayton cloth red* is dyed on wool in an acid bath, and produces a full blue shade of scarlet, which bleeds slightly when milled with soap, but does not go into the white. Dyed on wool, it gives the following reactions: Boiled with dilute sulphuric acid the colour is not changed; by ammonia it is partly removed. Concentrated sulphuric acid turns the fibre dark violet.

*Auroline* is Tetra-nitro-phenolphthalein. The commercial product is a yellow paste, which dissolves in water with an orange-red colour, which is rendered clearer and somewhat darker on the addition of caustic soda. Dilute sulphuric acid completely precipitates the dye. The most characteristic reaction is that with stannite of soda, in presence of excess of caustic soda, by which the yellow colour of the solution rapidly changes to an intense indigo blue. The dried paste gives, with strong sulphuric acid, a brown-orange solution. *Auroline* is dyed on wool in a bath acidulated with acetic acid, and produces orange-yellow shades resembling those obtained with Tartrazine. The colours are fast to milling, and do not bleed into the white. Dyed on wool, *Auroline* gives the following reactions: Boiled with dilute sulphuric acid, the colour goes, but is restored by ammonia. Stannite of soda and caustic soda turn the fibre blue. By concentrated sulphuric acid the fibre is turned somewhat redder.

#### II.—DYE INTENDED PRINCIPALLY FOR COTTON.

*Thiazol Yellow* dissolves in water with a yellow colour. Dilute sulphuric acid produces an orange precipitate having a yellow solution. Caustic soda produces a scarlet precipitate, which dissolves on boiling. By stannite of soda the orange colour of the hot alkaline solution is not affected. Calico is dyed with 12 per cent. of the colour, 5 per cent. of common salt, 5 per cent. of sodium phosphate, and 2 per cent. of soap, a bright canary yellow, which bleeds very slightly to soap water and does not go into the white. Dyed on cotton, *Thiazol yellow* gives the following reactions: Boiled with dilute sulphuric acid, the fibre is turned orange, with caustic soda a blue shade of red. Stannite of soda produces an intense orange; strong sulphuric acid no change.

#### PATENTS.

*Improvements in or connected with the Manufacture and Production of Unsymmetrical Acid Green.* B. Willecox, London. From "The Farbenfabriken vormals Fr. Bayer and Co.," Elberfeld, Germany. Eng. Pat. 1046, January 19, 1889. 6d.

A PROCESS for the production of new acid green dyestuffs by condensing benzaldehyde, dimethylaniline and methylbenzyl-aniline in molecular proportions in presence of a dehydrating agent. The unsymmetrically substituted di-amido-triphenyl-methane derivative thus obtained is sulphonated and the leucosulphonic acid on oxidation yields a green dyestuff. The working details are as follows: About 22 kilos. of sulphuric acid of 66 per cent. strength are gradually poured into an enamelled vessel cooled externally containing 12.1 kilos. of dimethylaniline and 19.7 kilos. of methylbenzyl-aniline and 10.6 kilos. of benzaldehyde are then slowly added at the ordinary temperature. The mixture is afterwards heated to 120°—130° C. and kept at this temperature for some hours. The melt is dissolved in water when a small quantity of symmetrical dimethyl-dibenzyl-diamido-triphenyl methane which is formed separates out. The tri-methylbenzyl-diamido-triphenylmethane is precipitated by adding sodium sulphate solution, leaving the small quantity of tetra-methyl-diamidotriphenylmethane formed in solution. The precipitate is boiled up several times with water, and forms a resinous mass of a pale-green colour. It is dissolved in cooled sulphuric acid (about five parts by weight) containing 20 per cent. of SO<sub>3</sub> and allowed to stand at the ordinary temperature until a sample dissolves in dilute alkali to a clear solution. The melt is then poured into water, the sulphonic acid converted into its sodium salt and oxidised with the calculated quantities of lead peroxide and dilute sulphuric acid. 10 kilos. of the sodium salt are dissolved in about 250 litres of water. The bluish-green solution of the dyestuff which results is filtered from the sulphate of lead and evaporated to dryness. The dyestuff obtained gives a bluish-green shade, and dyes more slowly on wool than do the ordinary acid greens. Different shades are obtained by using other tertiary amines, and in place of benzaldehyde, *m*-nitrobenzaldehyde may be used (Eng. Pat. 15,329, of 1885).—T. A. L.

*A New or Improved Manufacture of Para-Amido-Phenol Ethers.* B. Willecox, London. From "The Farbenfabriken vormals Fr. Bayer and Co.," Elberfeld, Germany. Eng. Pat. 1771, January 31, 1889. 6d.

See under XX., page 324.

*A New Extract and its Utilisation as a Dye, Ink, Stain, Tanning Liquor, and the like.* E. Brauser, London, and J. H. Knowles, Hull. Eng. Pat. 2360, February 9, 1889. 4d.

THE fibrous plant or material known as "*Bauhinia Vahlä*" is boiled in an alkaline solution, and afterwards squeezed by passing through rollers by which a colouring matter is extracted. The solution can be concentrated and the colouring matter may, according to the specification, be employed for the variety of purposes mentioned in the title.—T. A. L.

*The Manufacture or Production of a Colouring Matter or Dyestuff from Phenolphthalein.* C. Dreyfus, H. Bull, and J. Hall, Manchester. Eng. Pat. 3441, February 26, 1889. 6d.

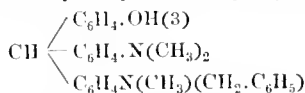
YELLOW dyestuffs are obtained by the action of nitric acid on phenolphthalein dissolved in sulphuric acid or glacial acetic acid or in a mixture of the two. A dinitro compound melting at 196° C. containing 6.91 per cent. of nitrogen (calculated 6.86 per cent.) and a tetra-nitro-derivative melting at 244.5° C. containing 10.89 per cent. of nitrogen (calculated for C<sub>20</sub>H<sub>10</sub>N<sub>4</sub>O<sub>12</sub>, N = 11.2 per cent.) have been obtained. The salts of the latter are soluble in water and dye animal fibres deep yellow from a neutral bath.—T. A. L.

*Improvements in the Manufacture or Production of Indulines or Induline-like Dyestuffs.* B. Willeox, London. From "The Farbenfabriken vormals Fr. Bayer and Co.," Elberfeld, Germany. Eng. Pat. 3529, February 27, 1889. 6d.

THE specification describes several methods for the production of indulines soluble in water. Most of these consist in fusing spirit-soluble dyestuffs technically known as nigrosine, aniline black, and safranine with *p*-phenylenediamine at temperatures about 190° C. until the melt is completely soluble in dilute hydrochloric acid. The whole melt is then so dissolved and the dyestuff salted out. Toluylenediamine may be substituted for the phenylenediamine. Azobenzene, azotoluene, azoxy- and hydrazo-compounds also yield soluble dyestuffs when heated with *p*-phenylenediamine in presence of sal-ammoniac. Greenish-blue to blue-black induline-like dyestuffs are obtained by oxidising *m*- and *p*-phenylenediamines with weak organic oxidising agents such as chloranile, dichloronaphthoquinone, naphthoquinone and naphthoquinone chlorimide. Aniline hydrochloride and its homologues when heated to 120°, and finally to 165° C. with *m*- or *p*-nitraniline, together with chloride of iron and benzoic acid or ammonium chloride, also form similar colours. The aniline may be replaced by a secondary or tertiary amine, a good result being obtained if a small quantity of aniline or toluidine hydrochloride be added to the melt.—T. A. L.

*Improved Manufacture of Green and Bluish-Green Colouring Matters.* O. Inray, London. From "The Farbwerke vormals Meister, Lucius and Brüning," Höchst, Germany. Eng. Pat. 5549, April 1, 1889. 6d.

THIS is an extension of Eng. Pat. 12,796 of 1888 (this Journal, 1889, 701) and describes the preparation of the *m*-hydroxybenzoic bases of the malachite green series of which *m*-hydroxytrimethylbenzylidiamidotriphenylmethane—



may serve as a type. These bodies may be obtained as already described by the action of nitrous acid on the corresponding amido compounds, or with regard to the compound here named by condensing equal molecular parts of *m*-hydroxybenzaldehyde, dimethylaniline, and methylbenzylaniline. The products are converted into sulphonic acids and on oxidation yield colouring matters which form copper-red powders of metallic lustre easily soluble in water. They dye wool and silk bluish-green shades fast to soap and light.—T. A. L.

*Improvements in the Manufacture and Production of Colouring Matters for Dyeing and Printing.* C. Dreyfus, Clayton, Manchester. Eng. Pat. 18,901, November 29, 1889. 4d.

DEHYDROTHIOPARATOLUIDINE sulphonic acid (see this Journal, 1889, 980; and 1890, 49) is converted into its diazo compound and combined with  $\beta$ -naphthol in an alkaline solution. The colouring matter after precipitation with an acid is converted into the ammonium salt. It dyes wool from an acid bath fast to fulling and can be used for printing cotton when applied with basic sulphate of alumina, the goods being subsequently steamed.—T. A. L.

#### ERRATUM.

In February issue, 1890, page 173, col. 1, 31 lines from the top, for "toluidine sulphate" read "tolidine sulphate."

Also *ibid*, same page and column, 8 lines from the bottom, for "diamido-azobenzene" read "diamido-azoxybenzene."

## V.—TEXTILES: COTTON, WOOL, SILK, Etc.

### PATENTS.

*A New Composition, forming a sort of Artificial Silk, for the Production of Threads or Filaments and Films, and for Coating Threads, Fabrics, or Objects, and Apparatus Employed in the Manufacture of said Composition.* I. H. du Vivier, Paris, France. Eng. Pat. 2570, February 13, 1889. 8d.

THE basis of this invention is a solution of tri-nitrocellulose in glacial acetic acid. The tri-nitrocellulose is prepared as follows. Cotton is steeped for three days, with occasional stirring, in a cold solution of caustic soda and ammonia. It is then washed, dried and carded. The nitrating bath is made by mixing 20 parts of potassium nitrate with 30 parts of sulphuric acid at 66° B. The temperature of the bath should be about 85° C. One part of cotton is introduced in small tufts, and the vessel containing it is revolved for five or six minutes, when the nitrated cotton is thoroughly washed and dried. Three solutions are prepared: (1) a 12·5 per cent. solution of gutta-percha in carbon disulphide; (2) a 5 per cent. solution of isinglass in glacial acetic acid; (3) a 7 per cent. solution of the tri-nitrocellulose in glacial acetic acid. These solutions are mixed together so that the mixture contains 4 parts of tri-nitrocellulose, 1 part of isinglass, and  $\frac{1}{2}$  part of gutta-percha; small quantities of glycerin and castor oil are then added and the whole kneaded together. The resulting viscous fluid is filtered and forced through narrow orifices under water. The thread so produced is first led into a bath of soda to remove the acetic acid, then into one of albumen, then into one of bichloride of mercury, and from thence into an atmosphere of carbon dioxide. In order to lessen its combustibility it is treated with ammonia and sulphate of alumina. The necessary apparatus is fully described. (Compare this Journal, 1889, 539.)—E. J. B.

*Improvements in Apparatus for the Manufacture of Textile Threads from Viscous or Semi-fluid Matters.* I. H. du Vivier, Paris, France. Eng. Pat. 2571, February 13, 1889. 8d.

THIS specification relates to apparatus for forming the threads described in Eng. Pat. 2570 (preceding abstract). Also to apparatus for drawing the threads through baths, drying and winding on bobbins, &c.—E. J. B.

*An Improved Textile Fabric or Material adapted to be worn next to the Skin.* C. I. Schott, Bradford. Eng. Pat. 2619, February 14, 1889. 4d.

THE fabric is formed of a mixture of hard spun yarn with a soft spun yarn, the manufacture being so conducted that the former projects in kinks beyond the surface of the fabric. Such articles produce a slight exciting action upon the skin without being irritating.—E. J. B.

## VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

*Notes on Mordants applicable in Wool-Dyeing.* (From the Dyeing Department of the Yorkshire College.) W. M. Gardner. J. Soc. Dyers and Colourists, 1890, 37—40.

WHEN dyeing wool with what have been termed the "polygenetic" colouring matters, it is necessary to use as mordant some metallic compound. The number of such mordants in practical use at the present time is very limited, some half-dozen completing the list.



*Bichromate of potash* (or soda) is probably the most largely used, but *aluminium sulphate* and *ferrous sulphate* are also employed for this purpose in enormous quantities. Somewhat less important are *stannous chloride* and *copper sulphate*, while a few other metallic salts find application in special cases.

From a purely chemical standpoint the few substances enumerated above possess no qualifications to act as mordants for wool which do not also characterise a large number of metallic compounds. Chromium, iron, copper, aluminium, and tin combine, *e.g.*, with alizarin, each metal producing a different colour. We should expect, therefore, by using compounds of other metals as mordants that still other colours would be obtained from the same dyestuffs.

The introduction of a successful new mordant is of great practical importance, and since there is the possibility of it yielding a new shade with many of the class of dyestuffs above referred to, it may prove of equal value to the introduction of several new colouring matters. The latter are being continually brought forward, but of late years few, if any, new metals have been used as mordants in wool-dyeing, although several new compounds of the metals at present in use have been introduced into the trade.

The question may here occur, are the particular compounds of chromium, iron, &c., now used, the best possible form in which to apply those metals? It is thought that for general purposes they may be considered satisfactory, but in many cases a distinct advantage would be gained by using other salts.

With respect to chromium, it is usually employed as a mordant for wool in the form of potassium or sodium bichromate. This being a strong oxidising agent it may, under certain circumstances, oxidise the wool or the colouring matter employed, and more or less destroy it. Hence the desirability of a chromium salt not possessing this defect. In view of this, those chromium salts in which the chromium forms the base—*e.g.*, chromic sulphate, chrome alum, &c., are naturally turned to. The last-named salt has long ago been proposed as an efficient substitute for potassium bichromate. As a by-product in the manufacture of alizarin it is comparatively cheap, and one might have expected that it would frequently be employed in practice, and this no doubt would be the case if it had been found superior to bichromate of potash.

It is well known that chromium sulphate, acetate, &c., are not satisfactorily fixed upon cotton by the methods which are used with the corresponding aluminium salts, and it would not be surprising if chromium and aluminium salts showed similar differences when used as mordants for wool. From experiments the author has made, however, it appears that chrome alum may be applied in the same manner as common alum—*i.e.*, with an addition of cream of tartar. But whereas with 1 molecule common alum an addition of 4 molecules cream of tartar is sufficient, with the molecule of chrome alum it is found necessary to use 12—18 molecules cream of tartar. The great expense of this large addition of cream of tartar is probably the reason why chrome alum is not practically employed.

A *Fluoride of chromium* ( $\text{CrF}_3 \cdot 4 \text{H}_2\text{O}$ ) has recently been put upon the market under the name of "*fluorchrome*." This substance, like chrome alum and oxalic acid, is a non-oxidising mordant, and also gives the green ( $\text{Cr}_2\text{O}_3$ ) mordanted wool, but attempts to obtain with it such full bright shades as with the use of potassium bichromate and tartaric acid were not successful. It appears also to give somewhat uneven shades, but possibly these defects may be overcome on further trials. The defects noticed are possibly due to the hydrofluoric acid liberated in the mordanting operation. "*Fluorchrome*" is considerably more expensive than chrome alum and oxalic acid.

Compared with other metals, chromium appears to be the most generally useful as a mordant for wool, since it gives both red, yellow, and blue colours with different colouring matters, so that by using a mixture of colouring matters a very large variety of compound shades may be obtained. Of the various chromium salts with which experiments have been made, potassium or sodium bichromate is the best for general use, being cheap, easily applied, and apparently not materially affecting the fibre, although the last point is

much questioned. Experiments on this latter point are in progress.

The shades produced by chromium mordant are, as a rule, dark, brilliant, and very fast against light and alkalis.

With respect to the *aluminium* mordants, it is worthy of remark that for most purposes the long-cherished alum is gradually giving way to the more rational aluminium sulphate, which can now be obtained sufficiently free from iron, and which, considering the percentage of aluminium it contains, is much cheaper.

Cream of tartar is the best assistant to use in conjunction with aluminium sulphate or alum for most colouring matters, but in some cases oxalic acid or binoxalate of potash gives much better results.

The chief use of the aluminium mordant is for the production of such light colours as cannot be obtained with chromium or iron. Speaking generally, the colours are more fugitive to light and to alkalis than those of chromium, copper, or iron, although not so easily affected as the tin compounds. Aluminium sulphate and alum crystals (potassium or ammonium alum) are the only salts of the metal which are used as mordants for wool.

The usual iron mordant is *ferrous sulphate*. In experimenting with this mordant it has been found that in almost every case an addition of oxalic acid gives much better results than the usual assistant—cream of tartar. It is also much cheaper. With many colouring matters some lime salt must be added to the dye-bath when using this mordant.

As to the comparative behaviour of *ferrous* and *ferrie* sulphate as mordants in wool dyeing, experiments showed that the two salts behave very similarly with respect to the assistants required, best proportions, &c., as well as in regard to the shade produced. Still the ferrie salt is sometimes to be preferred to the usual ferrous sulphate.

The remarkable action of an addition of oxalic acid when mordanting with ferrous sulphate has been already noticed, and this assistant has an equally good effect when ferrie sulphate is employed.

An amount of ferrie sulphate equivalent to from 4—10 per cent.  $\text{FeSO}_4 \cdot 7 \text{H}_2\text{O}$ , with an addition of 2—5 per cent. oxalic acid, should be used, the best amount varying within those limits according to the particular dyestuff and the percentage of it employed. As with ferrous sulphate, an addition of chalk or calcium acetate to the dye-bath is often very beneficial.

A most noteworthy point in connexion with the iron mordant, both ferrous and ferrie, is that the concentration of the mordant-bath has in many cases a very marked influence upon the shade produced. Good results are only obtained when using comparatively dilute solutions, *e.g.*, 0.3—0.6 gm.  $\text{FeSO}_4$  per litre; that is to say, in order to have the best percentage of mordant (6—9 per cent.  $\text{FeSO}_4$  and 3—4 per cent. oxalic acid), no more than 5—10 grms. of wool should be mordanted per litre of water.

If the solutions are concentrated—*e.g.*, 1.5—2 grms.  $\text{FeSO}_4$  per litre of water—it may happen that no colour is produced at all, or at most a dirty grey is obtained. In our experiments 1 litre of water and 10 grms. of wool were employed, but in practice the proportion of water to wool would be much less than this. Much more concentrated solutions would therefore be employed if the percentages given above were adhered to, and in order to prevent the injurious effect of too great concentration the amounts must be modified.

*Stannous chloride* ( $\text{SnCl}_2$ ) is the usual tin mordant, but the more acid *stannic chloride* ( $\text{SnCl}_4$ ) is sometimes useful. The great disadvantage of the tin mordant is that it has a very injurious action upon the wool fibre, tending to destroy its milling properties, and making it rough and harsh to the feel. This action is less marked with the stannic than with the stannous salt, the strong reducing action of the latter possibly having an influence. There is no doubt that some new mordant which would give as bright shades as tin, but without this very serious defect, would be of great value.

As is the case with aluminium mordant, the best assistant to use with tin mordant varies with the colouring matter employed. In some cases cream of tartar, in others oxalic acid, gives the best result.

The copper mordants are not of first-rate importance. The cuprous salts are too unstable for use as wool mordants, and cupric sulphate is the only salt employed for the purpose. With most colouring matters copper sulphate produces very similar shades to potassium bichromate, and as the latter is much cheaper, it is naturally employed. Usually, cream of tartar is a better assistant than oxalic acid or binoxalate of potash with the copper mordant.

A general consideration of the metals already in use as mordants shows that in some cases cream of tartar is the best assistant to use, while in others oxalic acid or binoxalate of potash is much to be preferred. It would naturally be expected that when a certain assistant proves to be the best with any given mordant, that it would be so, irrespective of the colouring matter employed. Experiment, however, proves that this is not the case.

The new mordants considered in this paper are the sulphates of manganese, nickel, cobalt, and uranium. The first three of these, together with iron, form a natural group possessing similar properties. They all form compounds more or less analogous to the ferrous and ferric salts, and it appeared interesting to determine by experiment whether the behaviour of their salts as mordants showed a like similarity. Manganese being a cheap product, it would be within the range of practical application if it was found to give useful colours, and even the other metals, though more expensive, might still be used if they produced colours possessing specially useful properties.

A careful series of experiments was made with these mordants in conjunction with anthrapurpurin, the effect of various amounts of mordant, assistants to mordant-bath or dye-bath, methods of dyeing, &c., being determined.

#### MANGANESE SULPHATE.

With anthrapurpurin this mordant yields reddish-purple shades, much less blue than those produced by iron mordant, but bluer than the chromium shades. The best amount to use is 4—6 per cent.  $MnSO_4$ , but, unlike the iron mordant, an addition of cream of tartar or oxalic acid does not improve the shade. The presence of chalk in the dye-bath, which may be considered essential with iron mordant, has comparatively little effect, although 1—2 per cent. makes the shade slightly fuller. The single bath method may also be used, although the shades are not so full and bright as those dyed by the two-bath method; it is best to use 4—6 per cent.  $MnSO_4$  without addition.

*Alizarin S S powder*, probably anthrapurpurin sulphonic acid, gives exactly the same results as anthrapurpurin.

With manganese mordant, *Nitro-alizarin* gives brownish purple shades, very similar to those produced by madder with this mordant. The shades show a greater similarity to the chromium and copper than to the iron shades.

*Alizarin blue*, when dyed upon cloth mordanted with manganous sulphate, gives blue shades, which are lighter than those dyed with the same percentage of dyestuff upon chromium mordant. The colour is also made purer—i.e., less purple than the chromium shade.

*Alizarin black* gives very similar shades whatever mordant is employed, although the manganese colour has a reddish tone.

In the case of *Gallocyanin* (*Solid violet*) manganese gives very similar shades to aluminium—viz., reddish purples; while *Gallein* with this mordant produces bluish purples, almost identical with the chromium shade.

*Carulein* yields green with all mordants, which, however vary in tone. The manganese shade is greyish green.

The behaviour of *Resorcinol green* ("Dark green") is interesting. The iron compound of this colouring matter possesses a dark green colour, but with no other mordant is a green obtained. Manganese produces a greyish-brown shade. This is noteworthy, since it is a striking instance of the fact that two metals as closely related to each other as iron and manganese may produce totally different colours when combined with the same dyestuff.

*Anthracene brown* ("Anthrakoll") with manganese produces bluish brown shades, which are similar to those

obtained when bichromate of potash + tartaric acid (green mordant) is the mordant employed. Bichromate of potash alone gives much yellower shades.

With the natural colouring matters (dyewoods) the following results were obtained with manganese mordant:—

*Logwood* produces purplish browns, which are not nearly so blue as the iron shades. *Peachwood* gives brownish reds, less blue than the chromium shade, and much less blue than with iron. *Cumwood* gives dull reds, not unlike the aluminium shade, but duller.

The *Cochineal* manganese colour may be extensively employed if cochineal continues to decrease in price. It is a capital imitation of the shade produced by orchil, and, of course, very much faster. By suitable mixtures of iron, manganese, aluminium, and tin, the whole range of shades from bluish purple to scarlet can be obtained from this dyestuff alone, and if its price is reduced it would, no doubt, be extensively employed, not for scarlets alone, but also for the production of other shades not easily obtainable from the alizarin colours.

The yellow dyestuffs, *Old fustic*, *Quercitron bark*, *Flavin*, *Held*, and *Young fustic*, give very dull yellow shades with manganese mordant. *Persian berries* produce very green shades of yellow, greener than the weld-aluminium shade, and therefore the greenest possible shade obtainable from the natural dyestuffs. The shade, however, is not so bright as the weld colour above mentioned.

A general survey of the results of the experiments shows that there is a distinct similarity in the shades produced by the group of metals Mn, Fe, Ni, and Co. At the same time, several great differences in their behaviour are noticed. Oxalic acid has been spoken of as a very beneficial assistant with many mordants, but in no case is its good effect so marked as with iron mordant. It is interesting to note, therefore, that manganese, nickel, and cobalt are better without that addition. With iron mordant again, the presence of a lime salt in the dye-bath is often essential to the development of the colour, whereas it is found injurious with the same colouring matter when manganese, nickel and cobalt is the mordant.

With certain dyestuffs—e.g., Resorcinol green and the yellow woods—the three mordants above-mentioned yielded in the experiments an entirely different shade to iron.

Uranium, of course, belongs to a different group of metals to manganese, cobalt, and nickel, and it is not surprising, therefore, that it gives shades which differ entirely from any of those above described. This metal forms one of the chromium group, and we find that in many cases it gives shades more or less similar to those produced by chromium. With many other dyestuffs, however, the two metals produce very different shades.

With *Anthrapurpurin*, uranium sulphate yields very pleasing bluish grey shades. Mordant with 4 to 6 per cent.  $U(SO_4)_2$ , with a small addition of tartar or oxalic acid, and dye with 2 to 5 per cent. anthrapurpurin (20 per cent. paste). The greys may also be produced in a single bath, 4 to 6 per cent. of uranium sulphate or 6 to 8 per cent. of uranium oxalate being employed. The water used should be as pure as possible, since the presence of lime salts makes the shade redder, and thus greatly detracts from the excellence of the shades. This mordant has been used to a small extent for the production of greys by certain dyers on the Continent.

*Alizarin* is to be preferred to *anthrapurpurin* for the production of uranium greys, because it produces slightly bluer shades. *Flavopurpurin*, on the other hand, is not so satisfactory, since redder shades are obtained.

Uranium in conjunction with *Nitro-alizarin* produces purplish greys, and with *Alizarin black* purplish browns. Neither of these shades are particularly pleasing. With *Alizarin blue* very blue shades of grey are obtained. *Gallocyanin* with uranium gives similar shades to Alizarin blue and chromium.

With *gallein*, uranium yields a bright reddish violet shade, approaching that given by a red shade of Methyl violet, but not quite so bright.

*Carulein* produces yellower shades with uranium than with any other mordant.

Turning to the natural colouring matters, it is found that uranium and *logwood* produce a blue grey very similar to the alizarin-uranium shade, but somewhat duller, and as the cost of the dyestuff in dyeing these greys is very slight, it is certainly better to use the purer colouring matter, alizarin.

*Cochineal* gives similar shades to anthrapurpurin, and this is another point of similarity between this most interesting dyestuff and the alizarin colours.

With *peachwood* and *camwood* uranium gives similar shades to aluminium.

Rather curious results have been obtained with the natural yellow dyestuffs in conjunction with uranium mordant. In some cases the shades are similar to those produced by chromium, in others they are quite different.

With *Weld*, *Persian berries*, and *Flavin* the two metals give similar shades, *i.e.*, with *Weld* an old gold shade and with *Persian berries* and *Flavin* a bright reddish-brown. This reddish-brown shade is also obtained with uranium and *Old fustic*, while the chromium shade is old gold. The order is reversed with *Young fustic*, chromium producing a reddish-brown, uranium a dull olive.

From a general consideration of the results of the experiments with these new mordants, it appears that uranium gives shades which it is difficult to obtain as *fast* shades in any other manner; and although a somewhat expensive metal, it may well find a more extensive use as a mordant for the production of the grey shades before mentioned. Of the group manganese, nickel, and cobalt, the latter gives generally the best result; but since in all cases they give similar shades to some one or other of the usual mordants, only some special excellence in the properties of the colour they produce would warrant their application. It has already been found that cobalt with Alizarin orange produces an extremely fast colour.

The experiments showed there is often a great similarity in the shades produced by metals belonging to one group, *e.g.*, manganese, iron, nickel, and cobalt, and also chromium and uranium. The cases in which quite different shades are obtained are, however, almost as frequent, and it is impossible from present knowledge to formulate any theory upon the subject.

#### PATENTS.

*Improvements in the Bleaching of Fibre.* A. G. Salamon. London. Eng. Pat. 415, January 9, 1889. 6d.

THE scoured vegetable fibre is bleached by treating first with permanganate of soda, and then, after rinsing, with a solution of borax more or less saturated with sulphurous acid. About 11 lb. of permanganate of soda are, it is stated, required for a ton of the raw material.—E. B.

*Improvements in or Connected with the Dyeing and Printing of Animal and Vegetable Fibres.* B. Willeox, London. From "The Farbenfabriken vormals Fr. Bayer and Co.," Elberfeld, Germany. Eng. Pat. 3934, March 6, 1889. 6d.

THE patentees have discovered that the so-called substantive dyes have the property of forming with the oxides of the earthy and heavy metals insoluble lakes, which in general are very fast. The results obtained vary according to the nature of the metallic salt and colouring matter employed, but in all cases a lake is produced, which is faster to light, boiling water and fulling than the original colour. In certain cases the metallic salt has the effect of changing the ordinary shade of the dye. Salts of iron, for example, render the shade duller; salts of nickel generally make it redder; others, such as those of zinc, have no effect on the shade. A striking result is obtained when salts of copper are caused to act upon the azo-dyes obtained from diamidodiphenol-ether, such as Benzoazurine 3 G and Azo-violet, the former rather reddish-blue being converted into a beautiful fast greenish-blue, the latter changing from a violet to a reddish-blue. Moreover the shades obtained on cotton by this treatment with the copper salt, no longer have the objectionable property of reddening under the

influence of heat. Such shades, however, resist the action of tin salts, so that discharge effects cannot be obtained by this means.

Zinc sulphate is found to be most suitable for use in treating dyed wool and silk, the dyed material being boiled for a few minutes with a quantity of the zinc salt corresponding to the depth of shade. In this way the colour on the animal fibre is rendered perfectly fast to fulling and milling, and is unaffected by boiling water.

The colour-lakes formed on cotton goods are not, as a rule, quite so fast as those obtained on wool and silk, but the resistance of the colour is increased as is shown by the fact that dyed cotton goods which have been treated with copper or nickel sulphate, can be soaped at 40° C., whereas the ordinary shades suffer even with soaping in the cold.

Salts of chromium, again, yield fast lakes with the various dyestuffs resulting from the action of the tetrazo compounds of benzidine, tolidine, diamido-carbazol, &c., upon salicylic acid and its homologues, and with the mixed dyestuffs made in part with the latter. On wool the lakes obtained are perfectly fast to fulling or milling and, on cotton, very fast to soaping. In printing, the chromium salt, for instance, the acetate, is added to the colour-mixture, which is printed and well steamed, or the printed goods can be passed through a solution of a chromium salt. In dyeing the goods may be previously mordanted with chromium, or treated with a chromium salt after dyeing.—E. B.

*A New or Improved System or Mode and Means applicable for Producing Marble Pattern and such like Papers.* W. B. Blaikie, T. S. Harper, and J. Campbell, Edinburgh. Eng. Pat. 4598, March 16, 1889. 6d.

MOLTEN metal, such as is employed in stereotype work, is cast in a layer of nearly-uniform thickness on a flat metallic or other surface. This surface may be either smooth or grained and, in some instances, preferably moistened with water, oil, wax, or other substances before casting the metal upon it. The metallic plates so cast are suitable for mounting with the level face upwards on blocks or may be bent round and secured to the circumference of a roller. Also, stereotype or electrotypes copies and reverses may be prepared, so as to give greater diversity in the designs and colourings of the patterns obtained.—E. B.

*Improvements in Open Soapers for Dunging and Soaping Fabrics.* J. Yates, Broadbottom, and G. Kay, Prestwich. Eng. Pat. 4991, March 22, 1889. 6d.

THE fabric to be treated passes into the first tank between a pair of india-rubber batters revolving on the surface and in a direction opposed to that of the fabric. Upon emerging from the tank the fabric passes between a similar pair of batters, but revolving in the same direction as that of the cloth. The cloth is then rinsed with water by means of a squirt-pipe, and passes over a steam-heated cylinder before entering the next tank of the series, which, like all the succeeding ones, is fitted similarly to the first.—E. B.

*Improvements in Yarn-dyeing Machines.* L. Weldon, Amsterdam, U.S.A. Eng. Pat. 18,971, November 26, 1889. 6d.

THE object of this invention is stated to be the provision of a machine, capacious enough to carry a large quantity of yarn and yet occupying a minimum space. The machine comprises a dye-vat, vertical rotary discs above the same, endless chains or bands passing round the discs and dipping into the dye-vat, and yarn-supporting bars carried on the bands or chains at the inner and outer sides of the latter. For exact details of the mechanism reference must be made to the drawings accompanying the specification.—E. B.

*Improvements in the Process of Treating and Scouring Wool and other Materials containing Greasy, Oily, or Fatty Matters, and in Machinery applicable thereto.* G. W. P. Kyre, London, and T. I. Hopkins, Trowbridge. Eng. Pat. 17,429, November 29, 1888. 11d.

THE material to be treated is placed in a cage inside a specially constructed centrifugal machine furnished with a water seal. Carbon disulphide or other suitable solvent is then run in, the enclosed air escaping through a vent. The machine is then rotated and the solvent allowed to run off into a tank. Water is then run in to cleanse the material and to wash out any remaining solvent. It runs into an intermediate tank, where any solvent carried with it settles to the bottom.

The rotation of the machine is continued until the material is dry. The drying may be assisted by means of air forced in by a fan.—E. J. B.

*Improvements in Dyeing and Scouring Machines.* J. P. Delahunt, West Pittston, U.S.A. Eng. Pat. 20,715, December 24, 1889. 8d.

THIS invention is an improvement upon a dyeing and scouring machine previously patented by the same inventor. The chief innovations are as follow:—The cylinder or cage is made with solid sides and heavy cross-bars at the periphery, and with perforated sheet metal or wire-work secured upon the inner faces of the cross-bars; instead of the straight pins formerly used to lift the raw material under treatment, there are now employed curved fingers fixed in rows and of different sizes. Two sets of such curved fingers may be fixed in each row, so that the machine may operate in either direction of rotation instead of in only one, as heretofore.

The mechanical arrangements in the original specification are described with the aid of drawings.—E. B.

## VII.—ACIDS, ALKALIS, AND SALTS.

*The Simultaneous Solubility of Potassium and Sodium Chlorides.* A. Etard. Compt. Rend. 109, 740—743.

THE accompanying diagram gives the solubility of potassium chloride and sodium chloride present in the same solution. The ordinates represent the percentage of salt in 100 parts of solution, and the abscissæ the temperature.

The conclusions to be drawn are as follow:—1. The sum of the salts dissolved is represented by a continuous straight line. (7.) From  $-20^{\circ}$  to  $+180^{\circ}$   $y \pm \frac{180}{20} = 27 + \cdot 0962 t$ . Some qualitative determinations lead the author to believe that the straight line is prolonged beyond  $180^{\circ}$ , but that at a certain point the quantity of water would practically be nothing; and he calculates that this "limit of solubility" would be  $738^{\circ}$ , which temperature is given by Carnelley as the fusing point of potassium chloride.

The different action of the mixed salts would not, however, be apparent from their solubilities.

The solubility of a mixture of the two salts is represented by a straight line. The slight inflections shown in the lines (1) and (4) are only passing, and may be taken as curves having points of inflection. These curves show, however, that change of solubility of either constituent may occur without the total solubility being apparent.

From the line giving the solubility of the mixture of potassium and sodium chlorides, the point of fusion would be  $738^{\circ}$ . At this point the calculated quantity of the two salts present would be 16.7 of NaCl and 83.3 of KCl. The sum of the metals and non-metal is then equal; that is to say, there are present the same number of molecules of chlorine and of the metals.—J. B. C.

*Isolation of a Tetrahydrate of Sulphuric Acid existing in Solution.* S. U. Pickering. Proc. Chem. Soc. 1889 (72), 128—130.

THE freezing points of mixtures of sulphuric acid and water form three distinct curves, representing the crystallisation

of water, of  $\text{H}_2\text{SO}_4 \cdot \text{H}_2\text{O}$  and of sulphuric acid respectively: in each of them the highest point is in exact correspondence with the composition of the substance which crystallises out. No solutions containing between 75 and 40 per cent. of acid had yet been frozen, but it appeared to the author that if his former deductions from the irregularities in the curves representing the densities and other properties of solutions of the acid were correct, an independent curve representing the crystallisation of a new hydrate should occupy this interval, and that this new hydrate could only be either  $\text{H}_2\text{SO}_4 \cdot 5 \cdot 5 \text{H}_2\text{O}$  or  $\text{H}_2\text{SO}_4 \cdot 4 \text{H}_2\text{O}$ . Experiment proved that it is the latter. The two branches of the new curve obtained rise from about  $-80^{\circ}$ , and meet in a sharply marked angle at a point exactly corresponding to the composition of the tetrahydrate, the temperature at which this point is reached being  $-25^{\circ}$ . The tetrahydrate forms large, well defined, hard crystals.

Apart from the consideration that the isolation of this hydrate was the direct outcome of his having previously recognised its existence in the dissolved state, the author maintains that the crystallisation of a substance from a liquid necessitates the recognition of its existence in that liquid. The various possibilities are (1) that the solid constituents solidify separately in the proper proportions, and subsequently combine: this view is obviously untenable; (2) that the molecular aggregates constituting the solid are formed directly from the acid and water: this means that a hydrate has no fundamental molecule, and besides leaves the impossibility of explaining how these solid aggregates can hunt each other out, and congregate to form large crystals; (3) that fundamental molecules of the hydrate are formed in solution, but cannot remain there, and consequently separate out almost immediately: this is tantamount to saying that the hydrate is an insoluble substance, a proposition which could scarcely be maintained even if the substance were not crystalline; (4) that the molecules of the hydrate are formed in the liquid, and exist there till the temperature is lowered below its freezing point. This, in the author's opinion, is the only tenable hypothesis.

When a liquid freezes and melts at a definite temperature; when the temperature remains constant till the whole of the liquid is frozen; when any alteration in its composition lowers its freezing point: we have the most certain evidence yet adduced for regarding that liquid to be a definite though possibly somewhat dissociated compound. If we deny this argument as applied to the hydrate in question, we must deny it in all cases, and conclude that no liquid is a definite substance.

The author believes that the dihydrate may yet be obtained as a solid, since there is a reach of 7 per cent. in its vicinity where freezing points have not yet been attained. He has also isolated another hydrate which crystallises in small quantities with the monohydrate, but has not succeeded in determining its composition.

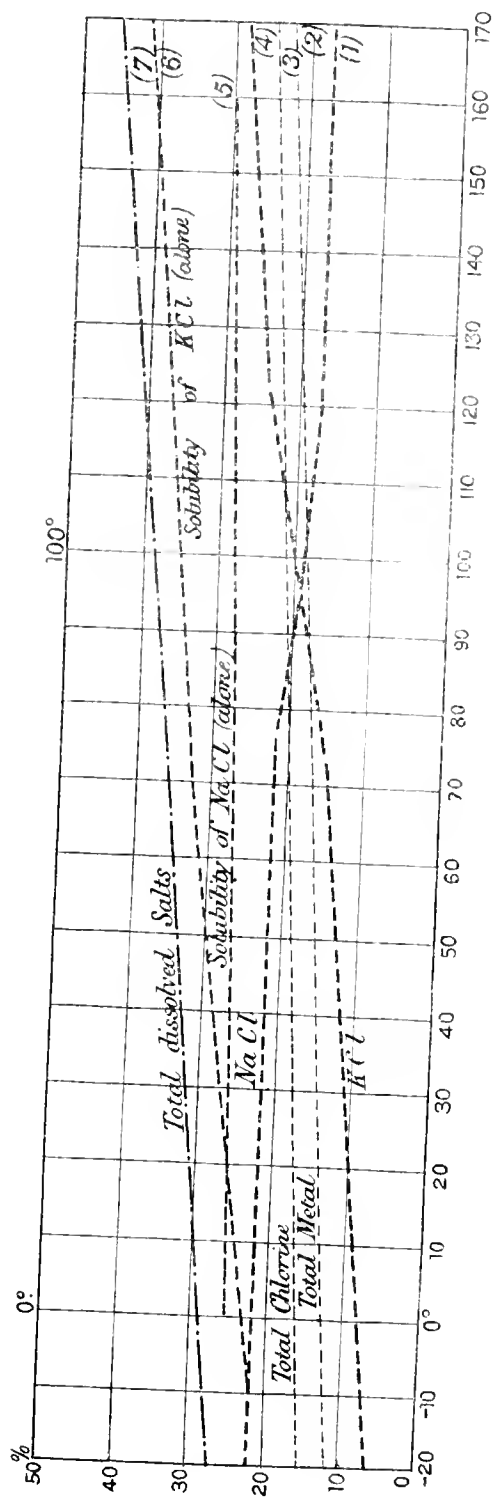
## PATENTS.

*Improvements in the Manufacture of Ammonia, and in Apparatus therefor.* A. Feldmann, Bremen, Germany. Eng. Pat. 3643, August 1, 1882. (Second Edition.) 8d.

Already described in this Journal, 1883, 380.—F. S.

*Improvements in the Treatment of Lime Refuse from Alkali Works, to render it suitable for the Manufacture of Cement, and in Apparatus employed therein.* James Hargreaves, T. Robinson, and John Hargreaves, Widnes. Eng. Pat. 94, January 3, 1889. 8d.

IN order to remove the sulphur and sulphur compounds present in carbonated alkali-waste, air, or some oxidising agent such as chlorine or bleaching-powder, &c., is blown through the mass made up into a cream with water. When the waste is only moist or semi-dry, air is blown through the heap when exposed to the atmosphere by perforated pipes passing in at the bottom. During the oxidation part of the sulphur is liberated as such, and this is dissolved by adding lime or lime sludge from the caustic soda manufacture. Another convenient solvent is the solution of calcium hydrosulphide obtained by steaming ordinary alkali

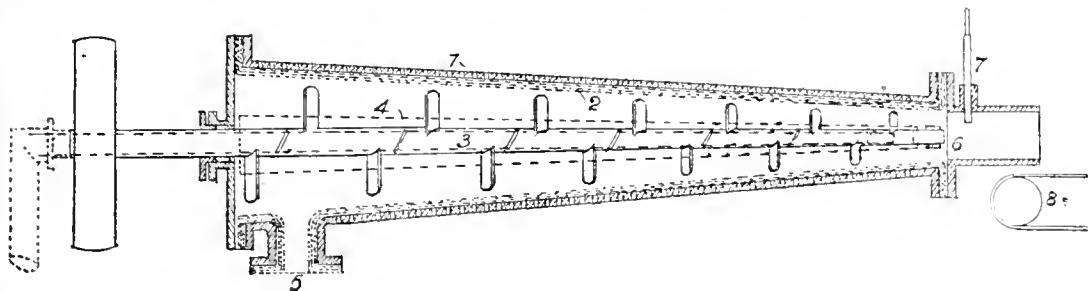




waste under pressure, but the best solvent of all is a solution of caustic soda or carbonate. On washing, all the sulphur is removed. Lime sludge is similarly treated by a preliminary oxidation, and the sulphur separated, dissolved in the alkali which is always present and then washed out. To filter the magma a special form of filter is used.

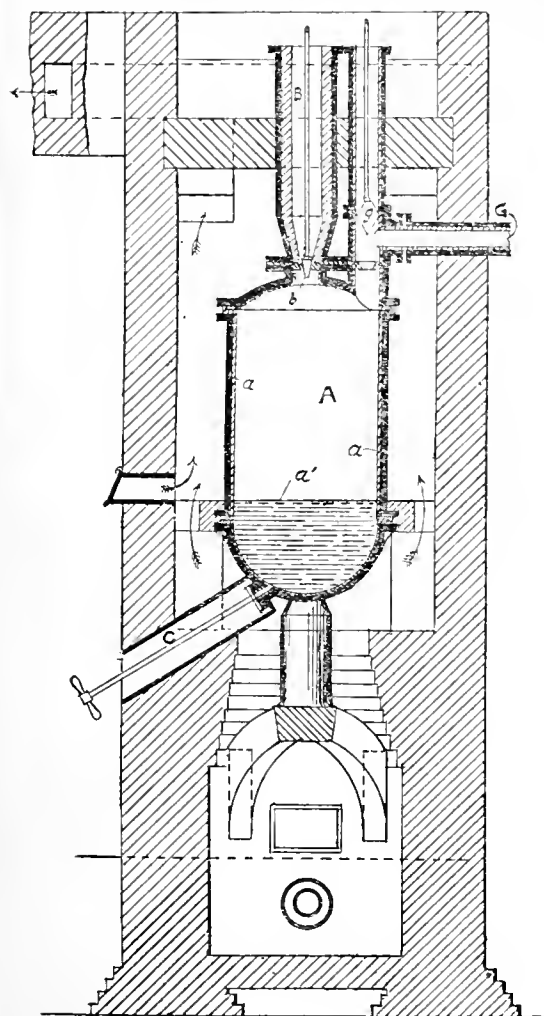
It consists of an outer perforated casing 1, lined with cloth placed between two layers of wire gauze, 2. There is an inner hollow shaft 3, enclosed in a perforated cloth-covered tube 4, the filtration thus proceeding both outwardly

and inwardly. The slurry enters at 5, and issues at 6; the slide for regulating the outlet is seen at 7, whilst 8 is the band for removing the filtered material. If required, the carbonated alkali waste, or lime sludge, or a mixture of the two, may be mixed with clay in the form of "slip," and oxidised, treated with a solvent to remove the sulphur, washed and filtered. The residue after drying is roasted and ground to form cement. The operations for removing sulphur and alkali are hastened by steaming, agitating and heating the mass.—S. G. R.



*Improvements in Apparatus for Volatilising Ammonium Chloride.* L. Mond, Northwich. Eng. Pat. 2160 February 6, 1889. 6d.

IN volatilising ammonium chloride, great difficulty has been found in procuring vessels able to withstand the corrosive



action of its vapours. Vessels of nickel or cobalt and their alloys, or iron vessels lined with these metals or their alloys, and of sufficiently large size being difficult to obtain, the patentee uses instead iron vessels lined with antimony, or an alloy of antimony. As antimony melts at a somewhat low temperature, the vessel is filled with melted zinc chloride to a level higher than that which the outside heating flame reaches. The apparatus employed is of the following description:—A represents the retort or chamber lined with antimony *a*, and filled up to the level *a'* with zinc chloride. The ammonium chloride is fed in through B, which is plugged by the rod *b*. C is the valve for washing out and emptying the vessel A. The ammonium chloride vapours are led away by the flue G. (See this Journal, 1887, 289; and 1888, 626.)—S. G. R.

*Improvements in the Manufacture of Cyanogen Compounds from Sulphocyanides.* T. Parker and A. E. Robinson, Wolverhampton. Eng. Pat. 2383, February 11, 1889. 4d.

THE solution containing the sulphocyanide is precipitated by sulphate of copper and a suitable reducing agent, such as sulphate of iron or sulphurous acid. The precipitated cuprous sulphocyanide is treated in the moist state with carbonic anhydride or with sulphuretted hydrogen under pressure so as to obtain carbonate or sulphide of copper and sulphocyanic acid. The sulphocyanic acid thus brought into solution is separated from the insoluble copper salts by filtration. The filtrate is subjected to electrolysis for the separation of the hydrocyanic acid which is condensed in water, to form the hydrocyanic acid of commerce, or in a solution of an alkali or of an alkaline earth to form a cyanide.—F. S.

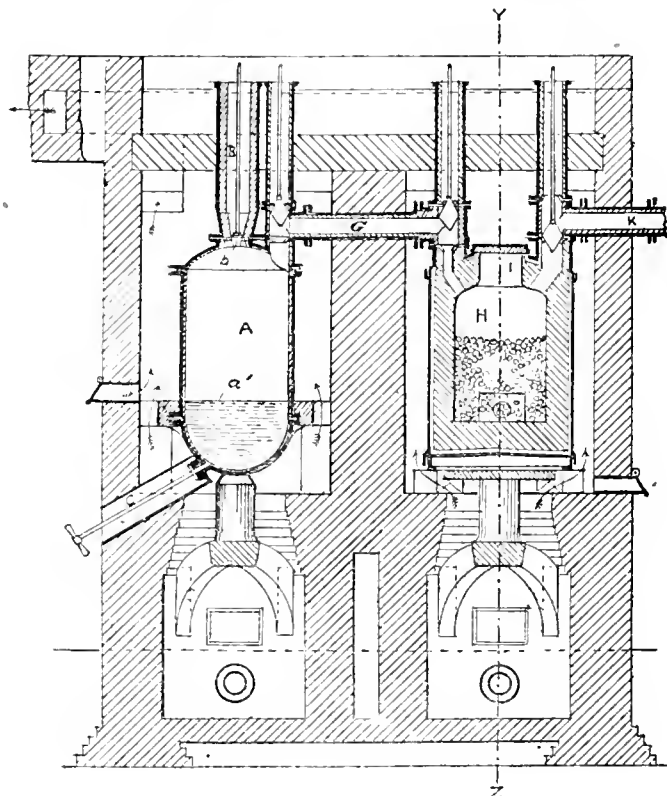
*Improvements in Volatilising Ammonium Chloride, and in obtaining Ammonia, Chlorine, and Hydrochloric Acid therefrom, also in obtaining Chlorine from Hydrochloric Acid.* L. Mond, Northwich. Eng. Pat. 2575, February 13, 1889. 8d.

THE vessel A is made of iron lined with antimony, it having been found that this metal resists the corrosive action of the ammonium chloride vapour better than any others. (For further details of the vessel A see adjoining Figure.) The vessel H is made of bricks or tiles of fireclay set in a cement which may be made of barium sulphate and sodium silicate, and is closed both at top and bottom. Outside the tiles is fitted an iron jacket. There are various inlet and outlet tubes, two of which are shown at G and K. Through the opening I are fed in the "pills" consisting of a mixture of 100 parts of magnesia, 75 parts of china clay, and 6 parts of lime made into a stiff paste with potassium chloride solution, and moulded into pellets of about half



an inch in diameter. The magnesia is preferably precipitated from sea-water, carefully washed, and ignited. The working of the apparatus, so far as concerns the vessel H, depends on the gases passed between these pellets and

upon the temperature employed. The vessel H is heated to  $350^{\circ}$ — $400^{\circ}$  C., and the vaporised ammonium chloride introduced through the flue G from the vessel A, the ammonia produced being led away to an absorption



apparatus. The supply of ammonium chloride is then cut off and the temperature raised by passing in heated inert gases, free from oxygen and aqueous vapour, from a limekiln or other source. A little more ammonia is at first evolved and is removed. When the temperature has risen to about  $500^{\circ}$  C. hydrochloric acid is given off and is taken away in its turn to the condensing plant. Carefully dried air at a temperature of from  $800^{\circ}$ — $1,000^{\circ}$  C. is now passed in and chlorine produced by the decomposition of the magnesium chloride. When the percentage of chlorine in the gases evolved falls below seven, the air containing this small amount of chlorine is again heated up to  $1,000^{\circ}$  C. and passed into another of the vessels ready for the evolution of chlorine. Cool air is then allowed to traverse the vessel H to reduce the temperature to  $400^{\circ}$  C. while the air, thus partially heated, passes through a Cowper's stove and thence to another chlorine-evolving apparatus. More ammonium chloride vapour is passed into H and the operations begin afresh. By using gases containing hydrochloric acid in the above apparatus instead of vaporised ammonium chloride, chlorine can be advantageously obtained. (See this Journal, 1887, 140, 216, 217, and 289; also 1888, 626.)

—S. G. R.

*Improvements in the Method or Process of Treating or Utilising the Spent or Residual Liquid formed in the Process of Cleaning Iron or Steel with Hydrochloric Acid.* T. B. Saunders, Cleckheaton. Eng. Pat. 3757, March 4, 1889. 6d.

SCRAP-IRON is added to the residual liquors to neutralise all the free acid, and the solution is evaporated to dryness. The chloride of iron is then treated with sulphuric acid, the hydrochloric acid evolved being condensed by suitable means. The sulphate of iron remaining may be either crystallised

or converted by roasting into the oxide. The process is carried out as follows:—

The spent liquor is run into the tank *a* containing scrap-iron and thence pumped to the tank *b*. This is in communication by *C* with the boiling-down pan *e*. After concentration, sulphuric acid is run in by the pipe *m* from the tank *o*. The hydrochloric acid evolved passes away through the flue *r* of the roaster *l* to the condenser *t*. The pasty mass of sulphate is then pushed over into the chamber *k*, and there dried by the waste heat from the furnace *F* which traverses the flues *u* and *v*.—S. G. R.

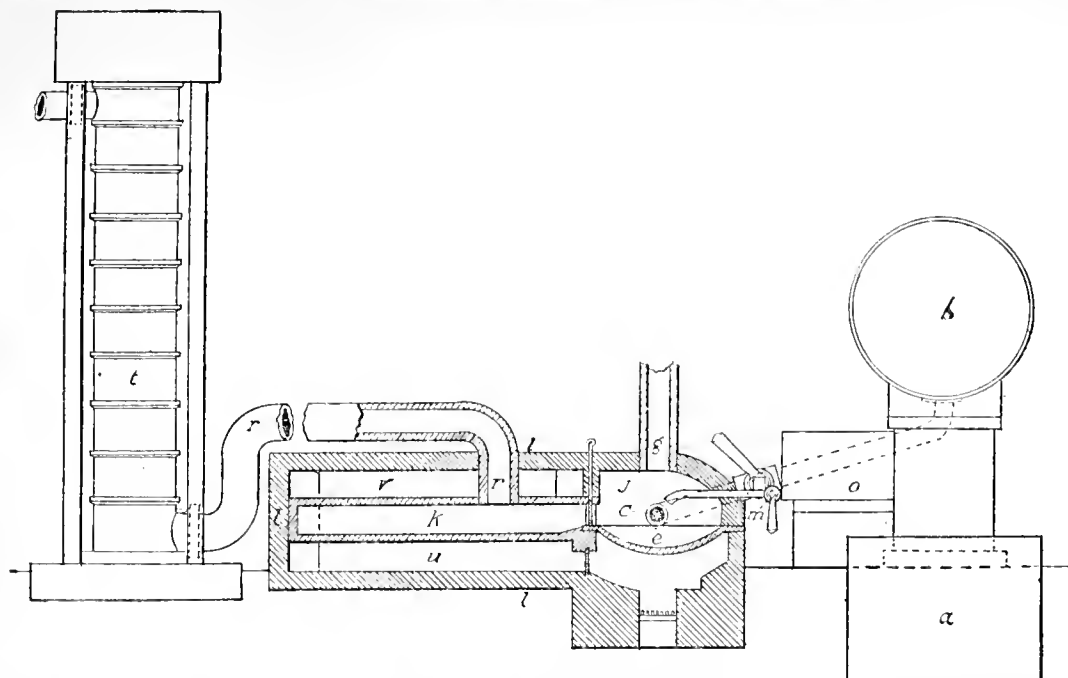
(For illustration, see next page.)

*Improvements in the Manufacture of Ammonium Nitrate.* E. Carez, Brussels, Belgium. Eng. Pat. 4316, March 12, 1889. 4d.

BARUM sulphate by roasting with charcoal and resin oil is converted into the sulphide, which boiled with water and sulphur forms a solution of polysulphide. The solution, when mixed with sodium nitrate and concentrated, yields barium nitrate, which by double decomposition with ammonium sulphate forms ammonium nitrate, while barium sulphate is precipitated.—S. G. R.

*Process of Manufacture of Ammoniacal Calcic Phosphate.* G. A. H. Gabet, Paris, France. Eng. Pat. 5340, March 28, 1889. 6d.

AN acid solution of calcium phosphate is treated with either gaseous ammonia led directly into the solution, or else with ammoniacal liquor. The solution gradually becomes pasty, and the mass can be easily dried. It is claimed that simul-



taneously with the formation of a new industrial product a method for fixing ammonia, not previously in use, has been devised. As a manure it is stated to be richer in nitrogen and phosphoric acid than any other known.—S. G. R.

*An Improved Process of Producing Ammonium Nitrate.*

A. W. and J. A. Wahlenberg, Stockholm, Sweden. Eng. Pat. 12,451, August 6, 1889. 4d.

A SUITABLE nitrate is decomposed by a salt of ammonia such as ammonium sulphate, ammonium carbonate, or ammonium chloride. The ammonium nitrate formed is separated from the other salts and impurities by means of alcohol.—F. S.

*Improvements in the Manufacture of Acetic Acid, Soluble*

*Acetates, and Calcium Sulphite.* J. E. Johnson-Johnson, Stratford. Eng. Pat. 17,348, November 1, 1889. 4d.

ACETIC acid is prepared from calcium acetate by heating equal weights of calcium acetate and water to about 100° F., and passing sulphur dioxide gas through the semi-liquid mass, which is kept in motion by a stirrer, until fully saturated with the gas. The sulphite of calcium is separated by decantation and filtration from the acetic acid, which may be purified by any suitable and well known process, but preferably by that described in Eng. Pat. 13,336 of 1887 (this Journal, 1888, 628). Acetate of soda is prepared by treating calcium acetate in a semi-fluid mass, and heated to 100° F. with sulphite of soda, or by decomposing the calcium acetate, before or after the addition of sodium hydrate and carbonate, by sulphur dioxide.

Weak and impure acetic acid (pyroligneous acid) can be treated by the above process.—F. S.

*Improvements in the Manufacture of Acetic Acid and*

*Soluble Acetates.* J. E. Johnson-Johnson, Stratford. Eng. Pat. 20,135, December 14, 1889. 4d.

WHEN acetic acid is to be prepared the salt employed, preferably "lime salt" or crude calcium acetate, is treated with hydrochloric acid. On distillation acetic acid comes over, calcium chloride remaining behind in the still. If a soluble acetate, such as the sodium salt, be required, then calcium acetate in a semi-liquid state may be mixed with sodium chloride and the whole thoroughly stirred, sodium

acetate and calcium chloride being the products of the reaction. Similarly the sodium salt can be obtained by treating a mixture of calcium acetate and sodium carbonate or caustic soda with hydrochloric acid. Crude or weak acetic acid such as pyroligneous acid can be neutralised with sodium or calcium salts, and then treated by one of the above methods, producing a pure acid.—S. G. R.

## VIII.—GLASS, POTTERY, AND EARTHENWARE.

*On the Devitrification of the Ordinary Glass of Commerce.*

Appert and Henrivaux. *Compt. Rend.* **109**, 827—829.

By heating glass at a temperature near to that at which it begins to solidify, crystals of varying composition are obtained according to the composition of the glass. The smallness of the crystals and their intimate admixture with the residual glass only permits of their examination microscopically. A glass rich in soda and lime gives crystals of wollastonite distinguished by their occurrence in the monoclinic system in flattened prisms produced in a direction parallel to the ortho-diagonal. Their optical properties also correspond with those of wollastonite. When the bases present, as in common bottle glass, are magnesia and protoxide of iron, then pyroxene is the predominant form, the crystals exactly resembling diopside, with the same crystalline form, cleavages, &c. They can be distinguished from wollastonite by their faint green colour, their refraction and their angle of optical extinction. If the glass contains alumina as well as these bases then feldspars of different varieties are the principal results.—S. G. R.

*On the Composition of the Minerals ("Roches") made use of in China for the Manufacture of Porcelain.* G. Vogt. *Compt. Rend.* **110**, 43—45.

ANALYSES made in former years by Ebelmen and Salvétat led them to suppose that the difference in composition between the materials used in China, termed "yeou-ko"

and "petun-tsé," and the pegmatite of Limousin, was but small. If these substances be fused with sodium carbonate, and treated with hydrofluoric acid, then the percentage compositions of them all closely agree with one another. Treatment with hot concentrated sulphuric acid, however, brings great differences to light. Whilst pegmatite only yields up about 3 per cent. of soluble matter to the acid, "yeon-ko" dissolves to the extent of 34.15 per cent., whilst of the remaining 66.36 per cent., as determined by actual experiment, 52.95 per cent. is quartz and 13.41 per cent. soda felspar. The soluble portion corresponds to a formula  $6 \text{SiO}_2 \cdot 3 \text{Al}_2\text{O}_3 \cdot \text{K}_2\text{O} \cdot 6 \text{H}_2\text{O}$ , which is the formula of muscovite. Hence "yeon-ko" has the following composition:—

Quartz .....	52.9
Muscovite or white mica .....	31.3
Soda felspar .....	13.4
Calcium carbonate .....	2.0
Hydrated silica .....	1.0
	<hr/> 100.6

whilst the composition of pegmatite is—

Quartz .....	23.87
Felspar .....	72.83
Matter soluble in sulphuric acid .....	3.30
	<hr/> 100.00

The presence of muscovite or white mica in many of the Chinese products is shown by the following analyses:—

	Petun from			
	Cheo-Ki.	Yu-Kan.	Ki-Men.	Sang-Pao-Pong.
Muscovite, per 100 parts.	40.6	37.3	31.1	18.6

Muscovite was also detected by the microscopical examination. Although the percentage compositions of pegmatite and these Chinese minerals are much the same, yet they are in reality formed of very different substances, and the presence of such large quantities, about 20 per cent., of mica will probably exert a very appreciable influence on the qualities of the porcelain made from such sources.—S. G. R.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

### PATENTS.

*Improvements in the Treatment of Lime Refuse from Alkali Works, to render it suitable for the Manufacture of Cement, and in Apparatus Employed therein.* Jas. Hargreaves, T. Robinson, and John Hargreaves, Widnes. Eng. Pat. 94, January 3, 1889. 8d.

See under VII., page 290.

*Improvements in the Manufacture and Utilisation of Cement and Plaster, and in the Machinery and Appliances therefor.* R. Stone, London. Eng. Pat. 116, January 3, 1889. 8d.

The patentee adds to a mixture, composed of about 25 per cent. of river mud with about 75 per cent. of chalk, about 10 per cent. of "sawdust or other suitable fibrous material," saturated with petroleum, petroleum spirit, sulphuric acid

or oil, in the proportion of about 6 galls. of the liquid to 1 ton of the absorbent. The mixture is then burnt, and it is claimed that cement of high quality is produced, and that "all the sulphur is removed." Another material patented is composed approximately of 25 per cent. of flints, 25 per cent. of river mud, and 50 per cent. of chalk, burnt and ground together; it may be moulded by plain or grooved rolls and moulds. Plant for the manufacture of cement is also claimed; it includes several grinding arrangements, consisting of rolls, cylindrical or barrel shaped, run in contact with others of corresponding shape, at different speeds, so as to enhance the grinding action, a cylindrical vertical furnace, with or without a blast, and a horizontal furnace, through which pass endless bands for effecting the loading, burning and withdrawal of the cement continuously.

—B. B.

*Improvements in the Manufacture of Artificial Stone.* A. C. Ponton, Parkstone, B. L. Mosely and C. Chambers, Hastings. Eng. Pat. 2780, February 16, 1889. 4d.

SAND, gravel, pebbles, or similar siliceous material is mixed with a solution of an alkaline silicate and (optionally) with powdered burnt flint or sand, separately or together, moulded by pressure, and fired.—B. B.

*Improvements in or relating to the Manufacture of Artificial Siliceous Stone.* A. C. Ponton, Parkstone, B. L. Mosely and C. Chambers, Hastings. Eng. Pat. 2781, February 16, 1889. 4d.

Siliceous materials, such as sand, gravel and flints, are heated until the silica they contain is converted into the amorphous variety termed by the patentees "Tridymite" (this Journal, 1890, 190), the completion of the change being defined by the specific gravity of the material ceasing to alter. The product is cemented by a solution of soluble glass, moulded and fired.—B. B.

*Improvements in Arrangements for Drying Slurry by the Waste Heat from Cement Kilns.* G. Batchelor, Frindsbury. Eng. Pat. 4333, March 12, 1889. 8d.

At the back of a cement-burning kiln are built two arched chambers, one above the other, of such size that the total floor space they afford suffices for the reception of a full charge of slurry for the kiln. The products of combustion from the kiln are led into the slurry-drying chambers, circulated through them, and discharged into a chimney shaft at the end remote from the kiln. Both floors of the chambers are inclined slightly towards the kiln (about 1 in 300) so as to have the greatest thickness of slurry at the hottest part, and in them openings, with a brick edging to confine the slurry, are provided, to allow the flow of hot gases from one to the other. The slurry to be dried is contained in tanks formed in the depressions between the arches that compose the drying chambers, and is led thence through shafts or passages on to the floors of both upper and lower drying chambers. The arrangement is applicable for a group of kilns built side by side as well as those standing by themselves.—B. B.

*Improvements in the Manufacture of Cement.* J. W. H. James, London. Eng. Pat. 5157, March 26, 1889. 8d.

THE patentee asserts that in the modern method of making cement by the use of revolving furnaces, sufficient time is not given for the complete reaction of the materials on each other, and accordingly provides "maturing chambers," into which the clinker is passed as it leaves the furnace, and in which it is maintained at a high temperature for the requisite period. In the arrangement illustrated in the specification the maturing chamber is set vertically below the mouth of the revolver, from which the clinker either falls directly into the chamber down a shoot, with a door at the point of entrance to regulate its admission, or is carried thither by

an endless band, revolving table, or similar device. The maturing chamber tapers towards the bottom, and the finished clinker is removed by a worm and transferred to the mill. The chambers are worked in sets of four, one being charged while another is being discharged, and the remaining two in action and full of clinker.—B. B.

*Improvements in the Manufacture of Artificial Stone or of Articles formed of the same.* W. P. Thompson, Liverpool. From C. Kohn, Teschen, Austria. Eng. Pat. 17,640, November 6, 1889. 6d.

"EQUIVALENT parts" of powdered quartz and calcined magnesite, or "a double portion of quartz and one portion of calcined magnesite" are mixed with diluted soluble potash glass to form a stiff paste, moulded, and "freed from the superfluous amount of water at a temperature of about 15° C." The moulded blocks are subjected to a vacuum, and then to carbonic acid under a pressure of 1–10 atmospheres, the result being "that the hydrate of oxide of magnesium is completely converted into carbonate of magnesium, which in its turn combines with the quartz to form carbo-silicate of magnesium."

The product is said to be hard, unaffected by weather, and of high compression strength. The method of carbonation described may be applied to blocks of artificial stone already made by the known process of treating magnesite with magnesium chloride. An artificial stone not liable to fracture on being strongly heated may be made by inserting a network or perforated sheets of asbestos or similar material during the process of manufacture.—B. B.

*Improved Manufacture of Artificial Granite.* P. de Kristoffovitch, Paris, France. Eng. Pat. 18,911, November 25, 1889. 6d.

THE raw materials used are two kinds of clay possessed of the following characteristics:—

(1.) A less fusible clay containing less than 7.75 per cent. of ferric oxide, and not less than 42 per cent. of silica, and losing on ignition not more than 10 per cent.; its "coefficient of infusibility" (Bischoff) should be not less than 3.

(2.) A more fusible clay with as much as 14 per cent. of ferric oxide, silica varying from 42 to 73 per cent., and losing on ignition not more than 8 per cent.; its "coefficient of infusibility" may vary from 0.5 to 1.

One to two parts of (1), ground and sifted, are mixed with two to one parts of (2), previously baked, ground and sifted, and 6 to 20 per cent. of water is added. The mass is moulded under a pressure of from 200–1,000 kilos. per sq. cm., or where the use of pressure is impracticable from the shape of the articles to be made, moulded after adding more water to form a paste. The objects thus shaped are dried and ignited at a temperature above the melting point of (2), and about 150° C. below that of (1), becoming agglomerated and capable of receiving a polish. The cooling should be slow to avoid cracking. Granite may be imitated by using the more fusible clay in granules instead of powder. The product may be used as a building material generally, and is to be called "pyrogranite."—B. B.

## X.—METALLURGY.

*The Use of Bichloride of Mercury in the Saving of Fine Gold.* B. F. Wilson, jun. Eng. and Min. J. 1890, 60.

A SOLUTION of bichloride of mercury is made by the action of an electric current on a solution of common salt, metallic mercury at the bottom of the trough containing the salt solution being one pole and suspended carbon slabs the other. A trace of bichloride of mercury is formed in the

solution while the lower chloride is precipitated. The calomel is taken from the bottom of the generating boxes and treated with nitro-hydrochloric acid. The solution is concentrated and siphoned off, and its strength tested by its ability to coat copper wire. It is then passed from the tank to the grinder pan, where it permeates the ground-up particles of ore, and amalgamates with the very fine particles of gold which might otherwise escape and not be touched. The mercury in the pans serves the double purpose of amalgamating with the coarser gold and collecting the amalgamated particles which the bichloride attacks. If the solution be too strong it comes over with the tailings, thereby causing a loss. "The problem remains: how are we to use a strong solution and not lose any of it in the tailing pan? When that problem is solved the use of bichloride of mercury in gold amalgamation will be an established fact." "As it is, it is a great helper."—A. J. S.

*Comments on the Chemical Reactions in the Blast Furnace.* A. D. Elbers. Eng. and Min. J. 49, 227.

THE author discusses the nature of the chemical changes taking place in the blast furnace, and draws the following conclusions:—

"1st. The reduction and complete saturation with carbon of  $\text{Fe}_2\text{O}_3$  requires theoretically one pound of CO for each pound of Fe, and that of  $\text{Fe}_3\text{O}_4$ : one-twelfth less.

"2nd. The proportion of the quantity of CO that is required for actual carburisation increases much more rapidly with the degree of saturation than the theoretical proportion.

"3rd. The more completely the reducing ore becomes carburised the less heat is required for its fusion; in other words, 75 pounds of Fe and 30.36 pounds of  $\text{FeC}$  (= 105.36 pounds of  $\text{Fe}_3\text{C}$ ) require less heat for fusion than 100 pounds of Fe,  $\text{Fe}_3\text{C}$ ,  $\text{Fe}_3\text{C}$ , &c."—O. H.

*Mining Industry in Siam.* Board of Trade Journal, 8, 1890, 340–342.

TIN is found principally in the Malay Peninsula. Great Britain and Siam now divide this invaluable peninsula between them, but whereas in the British portion of the country companies are being rapidly formed to work mines, in the Siamese districts, or to speak properly, in the native Malay States, over which Siam claims suzerainty, the numerous mines to all practical purposes still lie awaiting the enterprising hand to come and throw their abundant treasures open to the world. The most productive mines are at a place called Lampang, which is situated at the foot of a hill from 3,000 to 4,000 feet high. Jala, which is a considerable way in the interior, is completely encircled by hills, and the surrounding country is rough and hilly. The tin ore is found here in small grains, and lies in patches in the alluvium of the valleys and on the hillsides close to porphyritic granite rocks. Tin quarries, more than a mile long in many cases, extend along the valleys and up the hillsides, while copper, when found, has been described as existing in almost solid isolated blocks.

In the island of Junk Ceylon, on the west coast of the peninsula, it is reliably stated that under such primitive conditions as described above an annual output of three million dollars' worth of tin is produced. This is much less than what it used to be before the administration of the island was taken from the semi-independent Rajah and handed over to a commissioner at Bangkok, and is indubitably only a tithe of what might be produced under properly-organised management.

Lakon and the island of Junk Ceylon are, however, not the only places where tin and copper are found, for, as already remarked, Chinese are engaged in tin-mining all over the peninsula, and there is no reason to doubt that the range of hills separating Siam from British Burmah has been upheaved under the same geological conditions as the continuation of it which passes right down the Malay Peninsula, and now shown to be so rich in metals. Mouhot,

the celebrated naturalist and traveller in Siam, in 1862 describes the district of Petchabourne to the north-east of Bangkok, near the great bend of the Mekong River, as the richest mineral district in Siam. Besides argentiferous copper and tin, he found traces of magnetic iron and antimony. Of these only iron was worked to any extent, and knives and tools of native manufacture were supplied to all the north-eastern part of Siam.

Gold is found in different places in Siam, and since time immemorial has been produced in the imperfect native fashion. Bangtaphan, which lies on the east side of the Malay Peninsula, about 10° N. lat., has for centuries been the most celebrated gold district in the kingdom. That gold exists there in large quantities is undoubted, but the British company which now works these mines has laboured under several initial disadvantages, of which the most damaging is the unhealthy nature of the surrounding jungle.

As regards the presence of gold and precious stones, Korat, which is situate to the north-east of Bangkok, may be described as having the same reputation as Kabin, and gold, to a more limited extent, has been found in the low hills which separate Siam from Cambodia.

There are no silver mines worthy of the name anywhere in Siam; but that the metal exists in many districts is fully established, and only proper working would furnish reliable estimates as to the wealth of the country in silver.

Coal has been discovered cropping up through the surface on the western coast of the Malay Peninsula, and ironstone has been found in the same place. As previously mentioned, iron ore is produced in the northern part of the kingdom.

#### *The Manufacture of Steel by the Direct Process.*

An ironworks in Pennsylvania is said to have been making steel by the direct process for a year past. Rhode Island graphite was formerly used to absorb the impurities of the iron ore. The graphite was a success, but the freight on it amounted to a considerable figure, and the company looked around for something with which to replace it. They experimented with coke, and soon found that it would answer all purposes when treated with milk of lime. When coke was broken into small pieces and soaked in thin milk of lime all the impurities of the ore were fused, the oxygen of the iron joined with the carbon of the coke, passing off as carbonic acid gas, leaving the impurities in such a shape that they could be eliminated. By this means the blast furnaces are done away with, and a great deal of expense avoided. The coke has now been in use for over a year, and as a result, the carbon works are turning out some of the finest bridge-plates made in the United States, and steel is being produced which is very low in phosphorus.

#### *A New Alloy.*

A new alloy has been discovered by Herr Rieth, Bockenheim, Germany, which is said to practically resist the attack of most acid and alkaline solutions. Its composition is as follows:—Copper, 15 parts; tin, 2·34 parts; lead, 1·82 parts; antimony, 1 part. This alloy is, therefore, a bronze, with the addition of lead and antimony. The inventor claims that it can be very advantageously used in the laboratory to replace vessels or fittings of ebonite, vulcanite, or porcelain.

#### PATENTS.

*Improvements in the Method or Process of Treating or Utilising the Spent or Residual Liquid formed in the Process of Cleaning Iron or Steel with Hydrochloric Acid.* T. B. Saunders, Cleckheaton. Eng. Pat. 3757, March 4, 1889. 6d.

See under VII., page 292.

*Improvements in the Manufacture of Copper.* P. C. Gilchrist. Eng. Pat. 4882, March 20, 1889. 6d.

The improvements relate to the separation of copper from impurities, more especially arsenic, antimony and tin. In the roasting of white or pimple metal for the production of blister copper, in the treatment of metallic bottoms for the removal of arsenic and conversion into blister or into refined copper, and also in the toughening and refining of blister copper, reverberatory furnaces are used lined with shrunk dolomite, magnesite, chrome iron ore, or other basic or neutral lining, by which means it is possible to maintain during the operation of refining a basic slag instead of the acid slag as hitherto. A purer product is thus obtained, together with a larger yield and increased output. It has been found that a suitable amount of lime to add when charging white metal containing 75 per cent. of copper, is from two to three cwt. per seven tons of white metal charged. The metal should be melted down slowly under air. Much less slag will be formed than is usual in sand-lined furnaces. A good heat should be kept on the furnace throughout the charge. It is often advantageous to add a few shovels full of lime to the slag shortly before tapping the charge. Care must be taken not to form too thick a slag or the operation will be retarded. It is often convenient to charge in also some copper oxides or slags, and to subject the surface of the bath to the action of an air blast, the oxidising action of which materially assists the diminution of the arsenic. The object of the smelter should be to obtain as little slag as possible, and with a low percentage of copper in it. Skimmings of slag should be made three or four times as desirable. When the sample begins to show a blister fracture a few shovels of lime are to be added once or twice until the bath is ready for tapping. The slags obtained when working as described will not average more than 30 per cent. of copper, whereas when working with the ordinary sand bottoms the slag usually averages 55 per cent. of copper, besides weighing considerably more per ton of blister produced. It was found when treating 400 tons of metallic bottoms in a basic-lined furnace that there was obtained 323 tons of blister and 107 tons of slag, averaging 25 per cent. of copper, and that when treating an equal weight of metallic bottoms in a sand-lined furnace there was obtained 191 tons of blister only, and 221 tons of slag, averaging 55 per cent. of copper. A slag in which there is no more silica than 20 per cent. should be worked with, as with more siliceous slags the elimination of the impurities takes place more slowly.—A. J. S.

*Improvements in and relating to a Process of and Apparatus for Producing Steel direct from the Ore.* W. F. M. McCarty, W. H. Ashton, and H. D. Walbridge, Washington, U.S.A. Eng. Pat. 9289, June 4, 1889. 8d.

The ore and flux are passed through a blast furnace by falling from one to another of a series of shelves built out from the walls, and during the downward passage are subjected to a current of hydrogen gas, which reduces the ore; after which the metal is melted by introducing hydrogen and carbonaceous matter with air or gas, which converts it into steel. The specification contains drawings of the furnace and eight claims.—A. W.

*Improvements in or relating to Amalgamators for Extracting Precious Metals from the Various Impurities combined with them in Ores and the like.* W. P. Thompson, Liverpool. From J. B. Brewster, New York, U.S.A. Eng. Pat. 16,396, October 17, 1889. 8d.

This apparatus, which has for its object the "working of ores of a friable or semi-fluid, sticky nature," consists of a deep vertical "pocket," an inclined screen, and a stepped incline terminating in another smaller pocket. Mercury is placed in these two pockets, and the pulp is fed into the larger one and lifted out again on to the screen by means of a row of endless revolving chains. The larger particles are caught by the screen and roll down to its side, while

the finer pulp is carried through the screen over the stepped incline to the smaller pocket, where the amalgamation is completed.—A. W.

*An Improvement in the Art of Treating Copper and its Alloys.* G. G. Mullins, Los Angeles, California. Eng. Pat. 16,649, October 22, 1889. 4d.

"THE object of this invention is to render more fusible, refine and improve copper and other metals in admixture to form alloys; to remove the dross, scoria, or slag; to prevent porosity; to harden and toughen the metal, and greatly increase its tensile and resilient strength." On the bottom of the crucible, or on the bed of a reverberatory furnace, from 1 to 10 per cent. of a flux consisting of 4 parts of silica, 3 parts of fluorspar, 2 parts of carbonate of potassium, and 1 part of tungstate of sodium is placed, according to character and weight of metal charged. When all is molten the bath is thoroughly stirred, allowed to remain quiet for a few moments, the dross skimmed off, and the process proceeded with as usual. Or from 1 to 10 per cent. of the flux, carried in paper bags, is placed in the already molten metal, thoroughly stirred, and the process carried on as usual.—A. J. S.

## XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

### PATENTS.

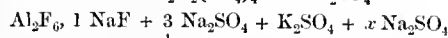
*Improvements in Processes for the Extraction and Electro Deposition of Aluminium and Aluminium Alloys.* P. Marino, Paris, France. Eng. Pat. 14,445, October 8, 1888. 6d.

SALTS of ammonium and salts of aluminium are electrically decomposed, oxygen and acid appearing at the "positive pole" and hydrogen and the aluminium base at the "negative pole." Five baths are described in which different salts of aluminium are used and in which the ammonium is obtained from the chlorhydrate of ammonia. Several other baths are described in which sodium, potassium, &c. are employed. The salt of aluminium preferably used is hauxite. By placing at the positive pole of one of the above baths a metal such as gold, tin, nickel, &c., any desired alloy can be obtained by the simultaneous deposit of aluminium and the particular metal made use of. Before the articles to be coated are put in the bath, they are thoroughly cleaned and treated with nitrate of mercury. The method is applied to the coating of glass with aluminium.

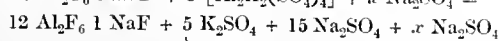
—B. T.

*Improvements in the Manufacture of Aluminium and its Alloys.* W. Diehl, Berlin, Germany. Eng. Pat. 813, January 16, 1889. 8d.

FOR the separation of aluminium and its alloys by electrolysis, a bath is prepared of the fluorides of alkali metals, or the compound fluorides of the alkali metals and anhydrous alum, a sulphate of an alkali and chloride of sodium. The substances are taken in the proportions given in the following equation, viz. :—



or

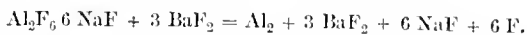


and melted together in a crucible. When melted, they are allowed to cool down, and are then ground and thoroughly washed in water to get rid of all traces of the sulphate. The fluoride thus formed will contain, for 1 mol. of aluminium fluoride, 1 mol. of alkaline fluoride,  $\text{Al}_2\text{F}_6, 1 \text{ NaF}$ . This

compound fluoride is now melted with an alkaline chloride, e.g., chloride of sodium, in proportion about the chemical equivalent of the aluminium fluoride, with as much fluorspar as the molten mass will take up, and while in a molten state the mass is submitted to a current of electricity, aluminium being collected on the negative pole, and chlorine liberated at the positive pole. In addition, a certain amount of alkali fluoride will be formed in the molten mass, as indicated in the following equation—



This sodium fluoride, if melted with anhydrous alum, will form the double fluoride,  $\text{Al}_2\text{F}_6, 1 \text{ NaF}$ , and will be in a condition to be subjected to electrolysis, as before explained, after the mixture has been thoroughly washed and the alkaline chloride added thereto. In place of the anhydrous alum an aqueous solution of nitrate of aluminium,  $\text{Al}_2(\text{NO}_3)_6$ , may be used for the purpose of producing the compound fluoride of aluminium to be treated. The compound fluorides of aluminium and alkali may also be taken and melted with fluoride of barium, without any chloride. Thus—



The various compounds obtained by the above means are subjected to the action of the electric current in a crucible heated in a furnace. When employing a retort carbon as cathode, the desired quality of the aluminium is not obtained, nor the desired output, as compared with the electric power expended; it is therefore preferable to produce, first, an alloy by an electrolytic process, from which the aluminium can be afterwards separated by means of commonly known processes. If either of the metals—iron, copper, cobalt, or nickel—he employed as the cathode, the current must be so regulated that an alloy rich in aluminium shall be obtained from the cathode. This easily fusible alloy is separated by careful fusion, into a crystalline alloy difficult of fusion and containing little aluminium, and a pure or nearly pure aluminium, comparatively easily fusible. This separation proceeds more effectually when it takes place in combination with certain salts which will not undergo chemical change, but only facilitate the separation: alkaline chlorides in union with alkaline nitrates; earthy alkali chlorides and alkali nitrates, &c.—A. J. S.

*Improvements in Secondary Batteries.* F. King, London. Eng. Pat. 1420, January 25, 1889. 8d.

EACH plate is provided with two lugs diagonally opposite each other, all the bottom lugs of one set, say the negative set, being burned into a strip of connecting metal which serves as a support, and all the bottom lugs of the other set being burned into a similar connecting strip which also serves as a support, but these two connecting strips are kept on opposite sides of the containing vessel. Notched distance-pieces of insulating material are inserted at each of the four corners and held in position by suitable lugs on the framework.—B. T.

*A Process for the Production of Pure Double Chlorides of Aluminium.* H. Y. Castner, London. Eng. Pat. 1989, February 4, 1889. 6d.

THE double chloride of aluminium or sodium prepared in the ordinary way contains small quantities of the chlorides of iron, which latter, when the material is used for making aluminium, become reduced, an impure aluminium being the result. To purify the double chloride it is melted and subjected to the action of the necessary quantity of aluminium, sodium, or other convenient metal, to decompose the chlorides of iron. The metallic iron which is produced sinks through the molten mass, while the colourless purified double chloride is drawn off or distilled in any convenient manner.

When an alkali metal is used it reduces some of the iron together with some aluminium, the latter in turn reacting with the remainder of the impurity.—A. W.

*Improvements in the Manufacture of Cyanogen Compounds from Sulphocyanides.* T. Parker and A. E. Robinson, Wolverhampton. Eng. Pat. 2383, February 11, 1889. 4d.

See under VII., page 291.

*Improvements in or Connected with Processes of Manufacturing Iron wherein the Iron is Subjected to an Electric Treatment.* A. G. Greenway, West Derby, and B. F. Babcock and B. Radcliffe, Liverpool. Eng. Pat. 3502, February 27, 1889. 8d.

THIS invention relates to improved methods of subjecting molten iron to electric currents in order to purify it and increase its strength. For this purpose electrodes of carbon are placed in the runners or gutters of the furnace or in a depression or chamber in the path of the same. Or, if desired, the electrodes may be placed in a receiver such as those used to convey metal to Bessemer converters. A current of air may be blown through the molten metal to aid in the process of purification.—E. T.

*An Improved Process for the Production of Fixed or Detachable Deposits of Homogeneous Iron in any Form, Size, or Thickness, from Iron Ores or Steel, or Iron in any Form.* A. E. Barthel, London. Eng. Pat. 5078, March 23, 1889. 6d.

THE object is to produce iron of greatly improved quality by electro-deposition. A bath is made up by precipitating ferrous oxide from ferrous sulphate by sodium carbonate and dissolving in strong sulphuric acid, using definite proportions of all ingredients. Anodes may be of cast or wrought iron, steel, or iron ore. A bath made by adding sodium sulphate to ferrous sulphate may also be used, but is not so good as that first described. Temperature should be from 14° to 40° R.—E. T.

*Improvements in Electric Batteries.* H. H. Lake, London. From Lacombe and Co., Paris, France. Eng. Pat. 5563, April 1, 1889. 8d.

A TUBE of carbon, forming one electrode, is placed in the position of the porous cell of an ordinary Leclanché cell. Inside this is a small porous tube, the space between the two being filled with the mixture of carbon and manganese peroxide. In the centre of all is the zinc rod. The cell is filled with the usual electrolyte, that in the porous tube being in communication with that which is outside the carbon tube.—E. T.

*Improvements in the Production of Aluminium.* R. E. Green, Southall. Eng. Pat. 5914, April 6, 1889. 4d.

A SUITABLE retort, crucible or furnace is provided having an inlet at its extreme lowest part, to which a porcelain or other pipe which will resist the heat of the furnace is attached and fixed, the joints being well luted up with a fire-resisting substance, such as a paste made of plumbago. This pipe is then connected to a supply of coal gas or other carburetted hydrogen or hydrogen gas. The size of this pipe for a charge of material of a quantity up to 2 cwt. need not be more than half an inch diameter. Broken bits of charcoal, coke, or other refractory substance are then placed at the bottom so as to cover the opening of the gas inlet and prevent its being stopped up during the subsequent operations. Upon this charcoal is placed fluoride of aluminium or the double fluoride of aluminium and sodium or potassium mixed with silica, silicate of soda or potash, or other compound of silicon, and finely powdered charcoal, and upon the top of this mixture is placed a layer of freshly burnt lime, though this is not absolutely necessary to the success of the process. The furnace is subjected to a red heat for an hour with the gas supply turned on. The decomposition being completed, the gas supply is shut off

and disconnected. The slag being removed, the whole is allowed to cool, when the greater part of the aluminium will be found mixed with and absorbed by the charcoal or coke at the bottom of the furnace. A quantity of aluminium of inferior quality remains mixed with the slag, which may be separated by grinding the slag and subjecting it to a second heating under the same conditions as above described.

—A. J. S.

*Improvements in Thermo-Electric and Furnace Batteries.* C. Hudscr, London. From H. Mestern, Munich, Germany. Eng. Pat. 11,661, July 22, 1889. 8d.

THE negative metal of each element consists of a short cylinder of cast iron, furnished at one end with a square base and at the other with a metal tube screwed on it. This tube is conical and holds in place a hollow asbestos block, into the interior of which, and in close contact with the iron, is poured the positive metal (antimony, zinc or alloys) in a molten state. A conducting strip passes out of this positive metal at the top, and curves round to join the next element at its iron end. The latter may be split, the conducting strip being placed in a channel between the two halves, which are then fastened together by screws passing through a common base piece. Rows of elements may be held by their square bases in a suitable frame, with the help of a fireproof cement. Two such frames are placed parallel with the iron ends facing, and heated at these ends by hot furnace gases passing between them to suitable flues. The antimony ends of the elements are kept cool by currents of cold air circulating over the reverse faces of the frames.

—E. T.

*Improvements in "Secondary or Electric Storage Batteries."* H. H. Carpenter, New York, U.S.A. Eng. Pat. 16,241, October 15, 1889. 6d.

THE electrode resembles a broad machine belt passing over two long rollers of small diameter, one at top and one at bottom, the space between the two sides—which are perforated with numerous holes to allow the liquid to permeate—being occupied by tablets of the active material. Cerussite is preferred for the active material for cheapness' sake, being found native, in a pure state. The plates are held in position by a central bolt passing through all, washers being interposed between the separate plates.—E. T.

*Improvements in and connected with Plates for Primary and Secondary Batteries.* I. B. Entz and W. A. Phillips, Schenectady, U.S.A. Eng. Pat. 18,365, November 18, 1889. 6d.

THESE plates are formed by weaving around a flexible copper wire a network of finer copper wire, in the meshes of which a metallic oxide or salt may be embedded. Any suitable length of these netted wires may then be bent round into the required shape.—B. T.

*An Improved Electrode for Secondary Batteries or Accumulators.* G. M. Cruikshank, Glasgow, Scotland. From W. A. Johnson and J. N. Smith, Toronto, Canada. Eng. Pat. 18,953, November 26, 1889. 6d.

EACH electrode is composed of a number of narrow lead plates, dovetailed together at the edges and fastened at their ends to suitable end-pieces. Each plate is provided on each face with a number of longitudinal grooves, preferably of J-shape, like the grooves in the beds of planing and other machines. The edges are burred inwards at intervals so as to divide each groove into short lengths. The plates are pasted as usual. They may be supported from the top of the cell by projections on the end-pieces.

—E. T.



*Improvements in Primary or Galvanic Batteries.* O. A. Enholm, New York, U.S.A. Eng. Pat. 18,966, November 26, 1889. 6d.

Porous partitions are used of a conducting material such as dense carbon in order to lower the resistance of the battery. Positive electrodes of zinc are used, and negative ones of carbon, these latter being made in the form of cups, in which the strong acid is placed, which percolates through the cup into the surrounding liquid to replenish it.—B. T.

*Improvements in Secondary or Storage Batteries.* W. F. Smith, Philadelphia, U.S.A. Eng. Pat. 18,989, November 26, 1889. 6d.

THE stout metallic border of the plate is cut through at places to allow of expansion of the active material, the cut portions being joined by metallic loops (resembling in shape the handles of carpet bags). The containing cells are provided with internal ridges, on which the plates rest by some of the above-mentioned loops.—E. T.

*Improvements in Electrical Secondary Batteries.* J. K. Pumpelly and F. D. Thomason, Chicago, U.S.A. Eng. Pat. 19,883, December 10, 1889. 8d.

HORIZONTAL electrodes are employed, preferably perforated and "pasted" in the following way:—A porous pad is temporarily placed below the plate, the perforations are then filled with dry commercial lead peroxide, and the plate charged in a suitable electrolyte. The time of preparation and forming is said to be in this way much reduced. The plates, with the porous pads removed, are placed in position in their cell, with cellulose fibre filling all spaces round and between the plates, to prevent the electrolyte from easily spilling and to keep the plates free from buckling.—E. T.

*Improvements in or relating to Electrical Batteries.* W. P. Thompson, Liverpool. From M. W. Parrish, Detroit, U.S.A. Eng. Pat. 19,889, December 10, 1889. 6d.

THE materials employed are a lump of crude graphite and zinc as the two electrodes, and salt and water as the electrolyte. It is stated that the cell is very powerful, and that "the zinc electrode cannot be depolarised, even if it comes into direct contact with the graphite."—E. T.

## XII.—FATS, OILS, AND SOAP MANUFACTURE.

*The Examination of Commercial Glycerin.* J. H. Wainwright. J. Amer. Chem. Soc. **11**, 125—130.

See under XXIII., page 331.

*Analysis of Marine Oils.* R. W. Moore. J. Amer. Chem. Soc. **11**, 155—158.

See under XXIII., page 331.

### PATENTS.

*Improvements in the Manufacture of Soft Soap for Laundry, Cleansing, and other Purposes.* J. Gott, Manchester. Eng. Pat. 16,845, November 20, 1888. 4d.

To ordinary soap dissolved in water, ordinary petroleum lamp-oil and borax dry soap are added.—W. L. C.

*Improvements in the Manufacture of Soaps and Cleansing Compositions.* R. Stone, London. Eng. Pat. 18,782, December 22, 1888. 4d.

SLATE-REFUSE, marble, or shale, ground to a very fine powder, is mixed with tallow, soap, oil, or other greasy substance, or even with Devonshire or china clay.—W. L. C.

*Improvements relating to the Purification of Fatty Substances, and to Apparatus therefor.* H. H. Lake, London. From "La Société Anonyme des Parfums Naturels de Cannes," Paris, France. Eng. Pat. 18,852, December 24, 1888. 6d.

IN the case of fatty substances extracted by volatile solvents, there is usually much difficulty in completely driving off the last traces of solvent from the fat. The patentees claim to effect this very completely by injecting steam in small jets through the fatty matter, and connecting the condensing apparatus at the same time with an air-pump, so as to maintain a good vacuum.—W. L. C.

*Improvements relating to the Purification of Fatty Substances and to Apparatus therefor.* H. H. Lake, London. From La Société Anonyme des Parfums Naturels de Cannes, Paris, France. Eng. Pat. 18,857, December 24, 1888. 6d.

THE last portions of solvents used in extracting fatty substances are difficult to remove from the fatty matters themselves, and the following method is claimed to facilitate this separation. A vertical boiler with a double bottom, through which steam can be passed, is rendered partially vacuum, and has introduced into it a quantity of water varying with the requirements of each case. It is then heated by turning on the steam, and the mixture of fat and solvent to be separated is injected through a horizontal coil pierced with numerous small holes, situated at its upper end. The steam arising from the heated water mixes with the spray thus produced, and carries away the solvent in the direction of the exhausting pump, on the way to which it passes through a worm and is condensed.—B. B.

*Improvements in Lubricants.* W. McLay, Wandsworth. Eng. Pat. 1501, January 28, 1889. 4d.

LUBRICANTS containing mineral wax are rendered more efficient and more economical in use by being incorporated with a strong aqueous solution of soap, e.g., one part of soap, containing 63 per cent. of fat, with three parts of water.

—W. L. C.

*Improvement in the Manufacture of Lubricants for Machinery.* O. Jeyes, Birmingham. Eng. Pat. 1857, February 2, 1889. 4d.

"REFINED naphthaline" is added to tallow alone or to a mixture of tallow and mineral oil.—W. L. C.

*Improvements in Lubricating Compounds.* H. Morgan, Blairstown. Eng. Pat. 2136, February 12, 1889. 4d.

PARAFFIN wax and French chalk are heated with any mixture of animal, vegetable or mineral oils.—W. L. C.

*A New Ointment.* J. Taylor, Isle of Wight. Eng. Pat. 2471, February 12, 1889. 4d.

IN addition to the usual lard, sweet oil, and beeswax, this ointment contains palm oil, oils of the liver of cod and sting-ray, and red oxide of mercury, all in specified proportions.—W. L. C.

*Improvements in the Treatment of Used Soapy Liquors.* W. G. Reid, Galashiels. Eng. Pat. 4367, March 13, 1889. 6d.

THE waste soapy liquors from the washing of wool or similar material are run into a settling tank for the deposition of the suspended matter, and thence into a vessel where they are treated with lime, and sometimes with a small quantity of some magnesium salt, such as the carbonate (dissolved by excess of carbonic acid) or oxalate, and an alkaline aluminate. The mixture is agitated by blowing in air or furnace gases, and if the lime be in excess it is removed from solution by adding a sufficiency of the original soapy liquor. The desired result is to leave all the alkalis in solution as hydrates, so that the clear liquid can be used for scouring once more, either direct or after treatment with carbonic acid, to avoid injury to the materials to be scoured, by caustic alkali, while the lime is left in the sludge combined with fatty acids. This residue can be utilised for manure, or for making gas, oil or soap. If the recovered liquors become too concentrated, owing to the alkali introduced into them by the wool itself, a portion may be evaporated, and the alkali obtained. In the presence of ammonium salts the ammonia may be recovered by distillation. Sulphates, if in quantity deemed injurious, may be precipitated by a barium salt.—B. B.

*Improvements in or appertaining to the Manufacture of Soap.* W. G. Smith, Kingston-on-Thames. Eng. Pat. 5353, March 28, 1889. 4d.

TURPENTINE and ammonia are incorporated with yellow soap on its removal from the copper.—W. L. C.

*Improvements in Treating Cotton-Seed Oil Mucilage to obtain Soap.* L. Pearson, Glasgow. Eng. Pat. 5622, April 2, 1889. 4d.

IN refining cotton-seed oil by means of alkali, a mucilaginous residue, formed by the action of the alkali on the impurities present in the oil, is obtained. The patentee heats this with close steam, and when liquid, adds a strong solution of caustic soda, or solid caustic soda if the mucilage be dilute, to the extent of about 100 lb. per ton. Open steam is then blown into the mixture for a short time. After shutting off the steam and allowing the liquid to remain at rest, a crude soap collects at the surface, being thrown up by the strong caustic alkali. Should this effect be delayed, the addition of more caustic soda (solid or in concentrated solution) will determine its occurrence. The soap may be bleached with a hypochlorite, and finished by any usual process.—B. B.

*An Improved Dry Soap or Washing Powder.* W. Hepworth-Collins and W. J. Smith, Bolton-le-Moors. Eng. Pat. 17,741, November 7, 1889. 4d.

TO the ordinary dry soap or washing powder of commerce, salts of sodium and potassium are added, the latter in the form of carbonate, nitrate, sulphate or chlorate.—W. L. C.

*A Disinfectant and Detergent Dry Soap or Washing Powder, or Soap Extract or Essence.* W. Hepworth-Collins and W. J. Smith, Bolton-le-Moors. Eng. Pat. 17,742, November 7, 1889. 4d.

MANGANATE of potassium, sodium, or calcium is incorporated with dry soap, or with any solid or liquid preparation thereof.—W. L. C.

*A New Method of Thickening Linseed Oil.* C. H. Robinson, Philadelphia, U.S.A. Eng. Pat. 18,046, November 12, 1889. 4d.

THE reagent employed is the thick gummy mass produced when linseed oil is boiled with litharge and exposed in films to air, in the first stages of the manufacture of linoleum.

One part of this is added to eight of raw oil, and the whole heated to 350° F., until the gummy mass is completely dissolved. The temperature may then be raised to about 630°, and if quick drying be required, 1 per cent. of litharge may be added.—W. L. C.

*Improvements in the Treatment or Preparation for Consumption of Soft Soaps and other like Materials.* J. V. Johnson, London. From L. Genevoix, Naples, Italy. Eng. Pat. 18,592, November 20, 1889. 4d.

TO prevent waste, the soap is wrapped in a covering or bag of some very porous material, usually woven, the size of the meshes or interstices of which depends on the thickness or fluidity of the soap. The covering may even be wholly or partially metallic; in fact, any material may be used which will retain the soap-paste, and yet allow it to exude as required. The invention may be applied also to powders.

—W. L. C.

*A New or Improved Method for Sweetening Margarine, Butter, and other Oils and Fats.* P. Heinz, Frankfurt-on-the-Main, Germany. Eng. Pat. 19,158, November 28, 1889. 4d.

See under XVIII. (A.), page 319.

*An Improved Process for the Extraction of Fats and Oils from Substances containing the same by Means of Sulphurous Acid.* W. Grills and M. Schröder, Neunhüll-Hamborn, Germany. Eng. Pat. 19,948, December 11, 1889. 4d.

THE patentees have found that liquid sulphur dioxide is a good solvent for fats and oils, quite comparable with carbon disulphide and light petroleum. It is true that it does not mix with fats in all proportions, but if the fat be in excess thorough mixture is obtained; otherwise, the mixture separates into two layers, the upper consisting of the oil with defect of sulphur dioxide, the lower of the heavier sulphur dioxide relatively poor in oil. The process of extraction is similar to that generally adopted, and is carried out at 30°–40° C., the chief difference being that to retain sulphur dioxide in the liquid condition at this temperature a pressure of about six atmospheres is needed. A systematic method of extraction may be adopted, or the following simpler plan:—The material to be extracted is placed on a grating or perforated plate in an iron cylinder, at the bottom of which is a steam coil. Liquid sulphur dioxide is now admitted below the grating and heated by the steam coil, the whole apparatus being closed gas-tight. The vaporised sulphur dioxide rises through the material to be extracted, partly condensing there, and partly in a worm above it (which is of course closed at its upper end), and drips back into the lower part of the cylinder charged with the fatty material it has dissolved. This process is continued for hours or days as may be necessary, when the exhausted material is removed, and the sulphur dioxide distilled off from the fat it holds in solution, the last portions being removed by blowing in air or steam; in the latter case the apparatus must be lead-lined, wet sulphur dioxide acting on the iron.—B. B.

*Improvements in and relating to the Manufacture of Soap.* W. Rödiger, Magdeburg, Germany. Eng. Pat. 20,237, December 16, 1889. 6d.

THE patentee states that the difficulties hitherto experienced in making a hard soap from rosin or from mixtures rich in rosin, arise from the varying and considerable affinities for water possessed by resinate of soda made from rosins of different qualities. He partially dehydrates and hardens rosin soaps by the addition of soda-ash, the proportions and conditions of application varying with the circumstances of

each case. As an example of his process the following will suffice:—To 40 parts of a solution of caustic soda of about 30° B., 100 parts of rosin are added, and soda ash in quantity determined by experiment (e.g. 10 per cent.) thrown in to effect the hardening spoken of above; if the action prove sluggish it can be hastened by raising the temperature, and in the contrary case retarded by cooling. The mass is well stirred, put into small "forms" holding about 200 lb., and the stirring continued until the hardening is complete, as is the case in from a few minutes to three-quarters of an hour according to the quality of the rosin. The addition of about 5 per cent. of salt promotes the hardening. The process is applicable to soaps containing fat as well as rosin if the proportion of the latter exceed 50 per cent.—B. B.

#### Improvements in Self-Lubricating Compositions for the Manufacture of Bearings, Cog-Teeth, and other Articles.

P. H. Holmes, Gardiner, Maine, U.S.A. Eng. Pat. 20,433, December 19, 1889. 4d.

WOOD or vegetable fibre is mixed with plumbago made into a paste with water; in some cases a little sulphuric acid and size are added. The product is moulded into eggs, journal boxes, or other articles subject to friction in use.

—B. B.

### XIII.—PAINTS, PIGMENTS, VARNISHES, AND RESINS.

#### The Future Supply of India-Rubber. Industries, 8, 187.

THE work of collecting the crude rubber juice has been left till recently to ignorant natives, who have destroyed millions of trees either by cutting them down or by their injudicious system of tapping. Latterly, however, the province of Amazonas has passed laws to regulate the season for tapping the trees, and awarded premiums to encourage the planting of new forests.

Some twenty years ago the importance of the question of the future supply of rubber was recognised by our Government, and James Collins, of Edinburgh, was instructed by the India Office to draw up a report concerning the rubber trees of America, and to ascertain whether they could be grown in India. The results of his investigations were published in 1872. In 1875 Robert Cross, of Liverpool, in concert with C. R. Markham, of Assam, went to Panama to collect seeds and cuttings of the Central American rubber tree (*Castilloa*). He was also sent by the India Office in the following year to Pará to collect information about the Pará (*Hevea*) and Ceara (*Manihot*) trees, which produce the best rubber. The descriptions given in his official report show that he met with formidable difficulties in the course of his undertakings, but he succeeded in his main object. The seeds and cuttings which he obtained were duly sown and planted at Kew, and in due time dispersed over various botanical gardens in India and the Colonies. As trees, they are now flourishing at Madras, Calcutta and Zanzibar, also in Burma and Ceylon. The great superiority of the Pará rubber (*Hevea*) over all other kinds has ensured for it the most attention, but its cultivation has been very much of a failure, notably in Assam and Northern India, though not equally so in Burma and Ceylon. It requires an equable moist climate without extremes of temperature. The *Castilloa* tree was reported a few years ago to be doing well in Burliar and other places in Southern India. Some attempts have been made to grow the West African tree (*Landolphia*) in India. It is a hardy shrub but takes up a good deal of room and the quality of its products leads to some doubt whether it would pay for cultivation. The following is a short list of plants producing india-rubber.

Nat. Order.	Genus.	Species.	Locality.
<i>Euphorbiaceæ</i>	<i>Hevea</i> .....	<i>H. Braziliensis</i>	Para.
	<i>Manihot</i> .....	<i>M. Glaziovii</i> ..	Ceara.
<i>Artocarpacææ</i>	<i>Castilloa</i> ....	<i>C. Elastica</i> ....	Central America, West Indies.
	<i>Ficus</i> .....	<i>F. Elastica</i> ...	Assam, Rangoon, Java.
<i>Apocynacææ</i> ..	<i>Hancornia</i> ..	<i>H. Speciosa</i> ...	Pernambuco,
	<i>Urceola</i> .....	<i>U. Elastica</i> ....	Borneo.
	<i>Vahia</i> .....	<i>V. Gummifera</i> ..	Madagascar.
	<i>Landolphia</i> ..	Various.....	West Africa, Mozambique.

The above trees are to be met with in a broad zone parallel to the equator, where there is an isotherm of 70° F.

Turning from the consideration of trees as they grow in untended nature to those that have been acclimatised, a report in 1881, by Dr. King, of the Calcutta Botanical Gardens, stated that the Ceara tree was the only one that promised good results, while the Pará and the Madagascar had utterly failed. This experience has been confirmed by Sir John Kirk, Consul at Zanzibar, who, in one of his official reports in 1882, said that the Ceara (*Manihot*) plants grew very well even in the worst soil, needed no attention, and commenced to yield rubber in five years, though not in a way to be remunerative. In October 1882, Dr. Trimen, of the Ceylon Botanical Gardens at Peradenya, sent to Kew a sample of the Central American rubber (*Castilloa*) produced from a cutting sent from Kew in 1876. This was examined by an expert, and reported to be of good quality, only deficient in tensile strength, owing perhaps to the immaturity of the tree from which it was taken. Colonial rubber is often being received at Kew, and submitted for examination, showing that this branch of economic botany is far from being neglected. Of Pará rubber from a Kew cutting, grown at Mergui, the first sample was received last season at Kew. As in the above case, it was found wanting in tensile strength, and fell short of the best qualities of ordinary smoked Pará. It remains to be seen whether the excess of its viscous over its fibrous constituents will be corrected in future tappings. It is not only in acclimatising foreign trees in India that work has been done, but, acting on Collins' report, the India Office has caused denuded districts to be replanted with the indigenous *Ficus elastica*. Already in 1870 there was a plantation of these trees at Chardnar, in the district of Darrang, which covered a space of eighty square miles. This particular area has since been annually increased, and there has been planting in other districts besides. It therefore appears that the wanton destruction of rubber trees has practically ceased, and that the importance of securing an adequate supply of rubber for future needs is fully recognised.

The financial side of the question can hardly be discussed till more reliable data have come to hand, but they are hardly likely to prove that undertakings in forestry bring any immediate or very early return. Dr. Trimen, for his part, feels confident that produce from Ceara trees will furnish an important item of revenue to Ceylon. It must be added that, in view of the competition in the case, other interested parties are not so sanguine of commercial success as he is.—O. H.

#### The Use of Coal-Tar Colours in the Manufacture of Pigments for Painters, &c. George H. Hurst. J. Soc. Dyers and Colourists, 1890, 32—36.

FOR some years past there have gradually come into extended use among painters, paper stainers, and calico printers, a class of pigments known as aniline lakes. Many of them find a large sale under a variety of names, such as Vermilionette, Royal reds, Post Office red, Victoria red, Emerald green, Non-poisonous greens, &c.

Perkin, in 1862, patented a process for making pigments from such colours as were then known, by precipitating them with arsenite of alumina, and boiling the precipitate with water. Messrs. Roberts, Dale, & Co. have also patented a process for preparing aniline lakes. They first make a

decoction of fustic, catechu, or quercitron; precipitate this with tin or alumina, and then mix the precipitate with aniline colour as long as it is absorbed—thus, for maroon they give the following: 100 gallons quercitron bark liquor at 1.06 (12° Tw.), 100 gallons of water, and 1 gallon perchloride of tin at 1.6 (equal 120° Tw.). The precipitate is then mixed with 8 lb. of aniline red. Other colours can be made by the same process, substituting other colouring matters for the red.

A lake is a pigment produced from natural colouring matters, such as cochineal, Brazil wood, and similar products, by extracting the colouring principle and combining this with a metallic base like alumina, oxide of tin, &c., so as to form an insoluble coloured pigment. Most of these lakes are true chemical compounds of the colouring principle, and the base—as, for example, Carmine, which is made from cochineal by precipitating the colouring principle, carminic acid, by means of alumina, so that carmine may be regarded as carminate of alumina. Crimson and scarlet lakes are similar in their composition. These lakes are, however, expensive bodies, and some cheaper lakes were made—as, for example, Rose pink and Dutch pink, by adding a quantity of white base, such as whiting or barytes. The shade of the lakes was paler and the tone duller, but they were cheap, and hence were largely used, especially by paper stainers.

The aniline lakes possess certain analogies to the lakes proper, inasmuch as they are or should be insoluble compounds of the colouring matter with a reagent; but they are not true lakes, inasmuch as some diluting agent is always present. The aniline lakes that were first made were not lakes at all, but simply a white base stained with colouring matter. Now, such a pigment cannot be satisfactory in use, for various reasons, one being that the colour, which is not properly fixed, is easily washed out.

A pigment must be insoluble in water and other bodies which are likely to be used as paint vehicles. Now, the most essential property of a coal-tar dye is that it must be soluble in water, and the problem to be solved is to find a body which will form, with the colour or dye, a compound which is insoluble in water. Such a compound could, without any impropriety, be called an aniline lake, but such a body is not suitable for use as a pigment, inasmuch as it has not sufficient body or covering power, and, moreover, would be costly. To get over these defects it is necessary to add some cheap white "base," such as barytes, China clay, or gypsum, which at once gives the desired body and cheapness.

The best method of producing this coloured aniline lake is by a process of precipitation, and all commercial pigments made from coal-tar colours are thus produced.

Necessarily the precipitating agent will vary with the properties of the colouring matters themselves. As a matter of convenience, those colouring matters derived from coal tar which are available for the manufacture of lakes, are divisible into three groups, as follows:—

I. *Basic Colouring Matters*, such as Magenta, Brilliant green, Violet, &c. The members of this class of colouring matters are precipitated by weak organic acids, such as tannic acid and picric acid; as a rule they are not precipitated by mineral acids or salts.

II. *Acid Colouring Matters*, such as the azo dyes derived from naphthols, naphthylamine, &c., which are precipitated by acids and various metallic salts from their solutions. The best precipitating agents for this class of dyes are lead acetate, barium chloride, and aluminium sulphate.

III. *Adjective Colouring Matters*, such as alizarin, gallein, cerulein. These approach more nearly the natural colouring matters in their properties, and they require metallic oxides, such as those of aluminium, chromium, iron, &c., to form lakes, and with these oxides they enter into true chemical combination, forming the coloured body. As a rule, the members of this class of dyestuffs have no, or but little, colour of themselves, and they produce different colours according to the metallic oxide with which they are combined. It is difficult to make very good lakes with them.

An aniline lake consists essentially of three constituents:—1st, the colouring matter; 2nd, the base; 3rd, the precipitating agent.

The Base is a most important constituent. A great deal of the usefulness of the finished product depends upon its quality. As bases the following may be used:—Barytes, China clay, gypsum, and zinc oxide. Starch has been used, but it is not satisfactory. The base should be quite neutral in its properties, and have good body or covering power. Barytes is mostly used, but it is not wholly satisfactory; the common qualities are usually rather gritty. The precipitated varieties known as "blanc fixe" are better and finer, but more costly. The best lakes the author ever made were those with precipitated barytes as base formed in the process of manufacture, but the cost was an item of consideration and would prevent this process from being much used. Gypsum is in some respects a better base than barytes, although it suffers from not possessing sufficient body. China clay gives good results, but is apt to give chalky lakes. Zinc oxide gives good results, but its cost is against it.

The best base for aniline lakes is a mixture of barytes, gypsum, and China clay. Weight for weight, barytes gives the deepest lakes, or, in other words, it takes less colouring matter to produce a lake of a given shade with barytes than it does with any of the other bases; and hence it costs less to make a given weight of a barytes lake than it does for a China clay lake, but on the other hand its covering power is also less.

As precipitating agents may be used tannic acid, picric acid, lead acetate, barium chloride, and aluminium sulphate. The following table gives the behaviour of a number of colouring matters with these agents:—

1. *Colours Precipitated by Tannic Acid*.—Magenta, Aniline blue, Brilliant green, Methyl green, New green, Nile blue, Rhodamine, Safranine, Phosphine, Chrysoidine, Bismarck brown, Paris violet, Methyl violet, Hofmann violet, Quinoline yellow, Auramine.

These belong to what are known as the basic aniline colours. The author found it best to use tartar emetic along with the tannic acid, as its use made the resulting pigment brighter in shade and more insoluble than if the tannin were used alone. One fault with the pigments made with tannic acid is that they do not dry well when mixed with oil; the cause lies with the tannic acid, which has the curious property of retarding, if not entirely preventing, the proper drying of the oil. Tannic pigments are therefore not suitable for oil paints; but for water paints, such as are used by paper stainers and calico printers, they are quite suitable.

2. *Colours Precipitated by Picric Acid*.—Auramine, Night blue, Methyl blue, Methyl green, Brilliant green.

Picric acid lakes are not suitable for oil colours; they dry even more slowly than tannic acid lakes.

3. *Colours Precipitated by Lead Acetate*.—Scarlet BB (precipitate of a poor shade), Mandarin G, Orange G, Orange IV. (gives brownish shade), Fast scarlet 3 R, Acid mauve B, Yellow N (very good precipitate), Croceine orange, Fast red T, Alkali blue, Citronine O, Methyl yellow, Citronine A, Tropaeoline OO (very pale shade), New yellow, Scarlet G, Ponceau 2 R, Scarlet R, Fast violet (this is not of a good shade), Orchil brown B, Fast red A, Azo-yellow, Double brilliant scarlet 2 R, Indian yellow, Crocein 3 B, Quinoline yellow, Crystal scarlet 6 R, Phosphine scarlet OO, Scarlet GT (very good precipitate), Vermilline scarlet KK, Bordeaux S (very good precipitate), Eosines.

(Since different makers use the same name for different colouring matters, it does not follow that the ponceau, for example, of one maker will be precipitated equally well as the ponceau of another maker.)

4. *Colours Precipitated by Sulphate of Alumina*.—Scarlet BB (gives rather brownish precipitate), Mandarin G, Orange IV., Fast scarlet 3 R, Croceine orange, Alkali blue, Victoria blue B, Citronine O, Scarlet G, Fast violet (gives very poor shade), Orchil brown B, Fast red A (too poor a shade to use), Azo yellow, Indian yellow, Scarlet GT.

5. *Colours Precipitated by Barium Chloride.*—Searlet BB, Ponceau, Orange G (fine shade), Orange IV., Fast searlet 3 R (very good result), Acid mauve B, Claret red B, Yellow N, Croceine orange, Crocein, Fast red T, Alkali blue, Victoria blue B, Citronine O, Methyl yellow, Citronine A, Tropaeoline OO (pale colour), New yellow, Searlet G, Ponceau 2 R, Searlet R, Resorein yellow, Fast violet, Orchil brown B, Fast red A, Azo yellow, Double brilliant searlet 2 R, Indian yellow, Crocein 3 B, Quinoline yellow, Crystal searlet 6 R (not a satisfactory shade), Naphthol yellow S, Phosphine, Searlet 2 R, Searlet (GT), Vermilline searlet KK, Searlet 2 RJ (gives fine shades), Searlet 3 R (gives good results).

Of the three last precipitating agents, barium chloride gives the most satisfactory results and the most permanent pigments; then comes sulphate of alumina, although this does not precipitate so many colours; lead acetate precipitates many colouring matters, but lead is rather objectionable in pigments, since if it comes in contact with sulphur it forms the black sulphide, and thus leads to discolouration of the pigment.

The pigments made with acetate of lead, aluminium sulphate, and barium chloride are very satisfactory to use either in oil painting, paper staining, or in calico printing.

The process of making these aniline pigments is comparatively simple. The base barytes, blane fixe, China clay, &c., is diffused through a quantity of water; this requires care, as if not properly done it leads to the formation of white chalky lumps in the finished product. The colouring matter is then added, having been previously dissolved in water; the whole is heated to from 80°–100° C., and the precipitating agent added, the whole being vigorously stirred. The pigment then forms, and on allowing to rest a while settles to the bottom; the top liquor is run off (this should be colourless, or at most have only a faint tint), clean warm water is added, the pigment stirred up, again allowed to settle, the clear top liquor run off, and the washing repeated. The pigment is now put on filter cloths to drain, and is then either sold in paste form for paper stainers and calico printers, or it may be dried for use by painters. The drying must be done at a low temperature. Too high a temperature spoils the shade.

#### VERMILIONETTES AND ROYAL REDS.

These pigments are made with eosines, and were at first used as substitutes for vermilion, hence their name, but they have gradually acquired a position of their own. Vermilionettes at first contained orange lead, but now this enters but little into their composition. Royal reds contain orange lead as their base; this is put in to give them weight and body. The following recipes will be found to give good results:—

	Vermilionettes.			Royal Reds.	
	I. Pale.	II. Deep.	III. Deep.	I. Pale.	II. Deep.
Barytes....	Lb. 100	Lb. 100	Lb. 100	Lb. ..	Lb. ..
Eosine.....	2	6	8	4	8
Orange lead	30	30	..	100	100
Lead acetate	6	16	20	10	20

The yellow shades of eosine give the best results. There are great differences in the quality of eosines; some kinds require more acetate of lead to precipitate them than others. Too great an excess of acetate of lead should be avoided, and every colour maker should ascertain by experiment the proper quantity to be used for the eosine he is working with. By varying the proportions and kind of eosine, a great variety of shades of these pigments can be obtained.

#### SCARLET PIGMENTS.

An immense variety of scarlet pigments may be made by using different scarlets and by mixing these in various

proportions and with other colouring matters, such as oranges and yellows. Hydrate of alumina makes a very good precipitating agent for these scarlets, and gives very fine pigments. The temperature at which they are made should not be allowed to exceed 50° C.

Where blane fixe is used as the base, this may be precipitated concurrently with the colouring matter, and by so doing a finer pigment is the result than if the base were simply diffused through the solution of the colouring matter.

This method may be further extended and hydrate of alumina used in conjunction, whereby very brilliant and deep pigments may be obtained.

*Scarlet Lake.*—110 lb. of sulphate of alumina are dissolved in 100 gallons of water and mixed with a solution of 25 lb. Ponceau RR. The mixture is heated to 60° C., and a solution of 100 lb. barium chloride added; the whole is then heated to the boil, cooled to 60° C., stirring all the time, and then a solution of 60 lb. soda crystals are added.

Care must be taken to avoid an excess of soda crystals as this would affect the shade of the pigment. The addition of the sulphate of alumina to the colouring matter must be made at a temperature not exceeding 60° C., and more especially is this important where those colouring matters are used which are precipitated by sulphate of alumina.

*Yellow Lakes.*—The third class of colouring matters—the so-called adjective colours—such as alizarin, gallein, Resorein green, &c., offer many and special difficulties to the production of lakes. The only one the author has been at all successful with is alizarin, and even with this the lakes made lacked brilliancy. He had not yet succeeded in making a lake from alizarin of the same shade and brilliancy as Turkey-red on cloth. Most of the so-called Turkey-reds that are being sold now are made from the Azo-scarlets.

Dark red lakes of good staining power can be obtained by the following processes:—

1. *Pure Alizarin Lake.*—20 parts of 20 per cent. alizarin are mixed with 250 parts of water, 10 parts of sulphate of alumina previously dissolved in water added, and 2 parts of calcium acetate, the mixture is then boiled for one hour, and 10 parts of soda crystals dissolved in water are added. Boiling is then resumed for some hours, followed by standing for 24 hours, filtering, washing, and drying of the precipitated lake. This yields a fine pure lake.

2. *Alizarin Lake.*—100 parts of barytes are diffused in 500 parts of water, and 20 parts of 20 per cent. alizarin, 10 parts sulphate of alumina, 2 parts of calcium acetate added; the mixture is then boiled well for two hours, stirring thoroughly. After this must be added the solution of 10 parts of soda crystals. Boiling follows for one hour longer, succeeded by standing for the rest of the day. The precipitate is then filtered, washed, and dried. This gives a lake of good colouring power and of a bright ruby tone. If caustic soda be used instead of carbonate of soda as a precipitating agent, the lake assumes a violet tone. By using a little oleine a rather brighter lake can be got, but an excess must be avoided.

Better results are got by the following process, but the time occupied is much longer:—

Take 100 parts of barytes and diffuse through 500 parts of water, add 10 parts of oleine, 10 parts of alizarin, and boil for one hour, then allow to stand for 24 hours, stirring at intervals; then add 20 parts of acetate of alumina (12° Tw.) and 2 parts of acetate of lime, stir well, allow to stand for two days, then boil vigorously for two hours, stirring well all the time; finally add, while still boiling, 5 parts of soda crystals. Filter, wash, and dry the precipitated lake.

Any other base than barytes can be used if thought desirable. Satisfactory lakes have not so far been obtained from other adjective colouring matters; as a rule these produce shades of rather a dull character not wanted in pigment colours.

The pigments thus described are mostly used in the painting trade. A few are used, to a limited extent, in calico printing, but the author is of opinion that in this department there is a wide field for their use in pigment styles, for producing brilliant shades unobtainable from other pigments.

In the discussion which followed, Dr. Knecht, referring to the precipitation of the eosin lakes by means of lead salts, asked whether—seeing that the presence of lead might sometimes be objectionable on account of its blackening in presence of sulphuretted hydrogen—no other substance had been used instead. He had himself succeeded in producing an erythrosine lake in the following manner:—A solution of erythrosine was added to a solution of wool in caustic soda, and then just sufficient sulphuric acid for neutralisation. In this manner a brilliant crimson lake was formed, but he had not tried its fastness to light nor its practical applicability. As precipitants for the basic colouring matters, Mr. Hurst had only referred to tannic acids and picric acids. There was another large class of precipitants which might be used, viz., the direct cotton dyes generally known as the benzidine dyes. A very wide range of shades might be obtained by using them. He understood that the same principle was employed pretty extensively in cotton dyeing, viz., topping colours produced with direct dyes, with basic ones, either for the purpose of intensifying the shade or altering it. Liechi and Suida, he believed, had succeeded in producing a brilliant Turkey-red lake by combining in suitable proportions, alizarin, alumina, lime, and Turkey-red oil. The red lake obtained by Müller-Jacobs' method ("Resinate lakes") was not likely to be brilliant, since the lime which is an essential constituent of Turkey-red was wanting (this Journal, 1889, 770).

Mr. H. Crossley thought that some of these coal-tar lakes were well worth the attention of designers in place of those produced by lead acetate.

Mr. Hurst, in reply, said that he had found that alum would precipitate some colouring matters which lead would not. He had found that the temperature of 180° F. was the best to use in producing these lakes, and the one at which the brightest colours were obtained. The reason why he had given empirical quantities of lead acetate for precipitating the eosins, instead of the theoretical ones, was because the eosins of commerce varied so much in their strength. Experience had shown that alum was not a good precipitant for the eosins, and he regarded the sulphuric acid contained in it as the reason, it being too strong an acid. He agreed with Dr. Knecht that the reducing action of the tannic acid was probably the cause of the slow drying of lakes produced from basic colouring matters. He was not favourably impressed with the use of the benzidine colours as precipitants; only some of them—viz., the yellow—could be used, because the others were not fast to light. Vermilionettes stood light well as oil paints if varnished over. The Manchester Tramway Company used them for their cars, and had found them to stand very well. The author agreed that the term "diluent" was preferable to that of "base," but used the term "base" because it was better understood by the practical painter.

#### PATENTS.

*Improvements in the Manufacture of Waterproof Material for Bags and other Articles.* R. Stone, London. Eng. Pat. 16,400, November 12, 1888. 4d.

BAGS made of hemp, flax, tow, cotton, or any fibrous or other material, or wire netting, are coated with a mixture of shale, tar, china clay, boiled linseed oil, terebine, and oak varnish.—E. J. B.

*Improvements in the Manufacture of Linoleum and like Fabrics.* H. W. Godfrey, C. F. Leake, and C. E. Lucas, Staines. Eng. Pat. 18,110, December 11, 1888. 8d.

This invention relates to a process for manufacturing linoleum and similar fabrics in such a way that the pattern extends throughout the thickness of the fabric. The process consists in "preparing a block of tesserae arranged in accordance with the pattern and laid one over the other; pushing forward such tesserae layer by layer on to a receiving

surface; advancing the said surface with the tesserae upon it step by step through a press, and consolidating the whole by an intermittent pressure."

The necessary apparatus, which is described, is also claimed.—E. J. B.

*A New or Improved Process and Compositions for Preparing and Coating Canvas, Floorcloth, Linoleum, and other Textile Fabrics.* J. Heyde, Dundee. Eng. Pat. 1758, January 31, 1889. 4d.

THE patent refers to a composition for first coating the fabric under treatment, to a composition for the back, and to another for the front or face of the fabric.

Composition No. 1 is as follows:—

9 parts by weight of extract of Irish moss in a thick gelatinous mass.

2½ parts by weight of strong starch.

7½ parts by weight of clay or whitening.

½ part by weight of brown glycerin.

½ part by weight of glucose.

The whole to be mixed with one-third by volume of water or skim milk; 10 to 12 lb. of alum per ton of above mixture and a little sulphuric acid may be added.

Composition No. 2 for backing the fabric is as follows:—

One part of No. 1, without clay or whitening, for which colouring matter such as oxide, ochre, &c. may be used.

Composition No. 3 for facing the fabric is as follows:—

Same as No. 2, but instead of the backing colour the ground colour must be used.—A. J. K.

*A Process for Waterproofing and Preserving Textures and other Materials.* C. F. Hime, London, and I. H. Noad, East Ham. Eng. Pat. 7715, May 8, 1889. 4d.

THE process consists in treating fabrics with a solution of cellulose in "ammonia-zinc." The latter is obtained as a colourless solution by treating an "ammonia-copper solution of cellulose" with metallic zinc.—E. J. B.

*Improved Materials and Process for Staining and Mottling Wood.* F. T. Prosser and W. Carter, London. Eng. Pat. 19,852, December 10, 1889. 4d.

THE wooden article to be stained, for instance a tennis racket, having had its surface properly smoothed and prepared, is sponged over with a mixture of 1 volume of nitric acid and 2½ volumes of water, and then heated in the flame of a gas-burner till the tint is fully produced. After "papering," the article is sprinkled with whiting wherever it is desired to produce a mottled effect, and is then sponged with a mixture of nitric and sulphuric acids somewhat diluted with water, and heated as before.—E. B.

*Improvements in Preparing the Surface of Iron and Steel in the Form of Flat or Corrugated Sheets, Hoops, Wrought or Cast Plates, Bars, or Manufactured Articles, for the Purpose of Coating it, and in Coating it with Waterproof Material for the Prevention of Oxidation or Rust.* R. C. Jay, London. Eng. Pat. 20,263, December 17, 1889. 6d.

THE iron or steel is exposed to the moisture of the atmosphere for a sufficient length of time to bring the scale off and form a coat of oxide sufficient to eat into the surface of the metal. A quicker method is to place the iron or steel in a bath of muriatic acid in the proportion of one part of acid to five parts of water. After taking out and washing off the acid, the plate is exposed to the action of the atmosphere until a sufficient coat of rust is formed, the process of rusting being assisted by moistening the surface from time to time with water. When the coating of rust is formed the iron or steel is dried at a temperature insufficient to destroy the coating of rust. At this temperature it is then coated with ordinary "gas tar, pitch, or a mixture of the two." Where pitch is employed an addition of treacle or molasses may be made so as to temper the pitch



and render it less brittle. Colours or paint mixed in the ordinary manner with boiled oil or dryers may be used. In some work Aspinall's enamel is used. Tar or pitch, either separately or combined with treacle or molasses will, when mixed with the colours, produce darker shades, which differ considerably from the colours originally employed. A few minutes in the bath is sufficient, when heating in an oven follows, beginning with a low temperature and rising to temperatures depending on the nature of the coating materials, but, as a rule, not falling below 150° F., so that the heat of the sun at least may be easily withstood.

—A. J. S.

## XIV.—TANNING, LEATHER, GLUE, AND SIZE.

### PATENT.

*A New Extract and its Utilisation as a Dye, Ink, Stain, Tanning Liquor, and the like.* E. Braiser, London, and J. H. Knowles, Hull. Eng. Pat. 2360, February 9, 1889. 4d.

See under IV., page 285.

## XV.—AGRICULTURE, MANURES, Etc.

*Fixation of Nitrogen by Leguminous Plants.* E. Béal. Compt. Rend. 109, 670–673.

In his researches on the fixation of atmospheric nitrogen in the soil by the action of micro-organisms, Berthelot has recognised the part played by the leguminosæ (this Journal, 1889, 626). The root of a leguminous plant can be inoculated by pricking it with a needle which has been introduced into a tubercle of a plant of the same family: tubercles develop rapidly upon the root thus inoculated, and these tubercles are found to be full of bacteria. The present communication describes the effects of two such cultures.

I. Two scarlet runners (which had germinated on wet filter-paper) were inoculated with bacteria from a tubercle growing on a root of *Cytisus*. The two plants were bedded in 10 kilos. of river-gravel which did not contain any appreciable amount of nitrogen. The gravel was watered from time to time with a very dilute solution of potassium chloride and calcium phosphate. The experiment lasted 67 days; the plants developed a number of ripe pods and their roots were covered with tubercles. Analyses and weighings are given showing that the weight of the plants (dried) was 24 times that of the seed from which they had germinated, and that the amount of contained nitrogen was 17 times that contained in the seed. The gravel, which originally contained no nitrogen, was now found to contain 0.0581 per 1,000, or 0.581 gram. in the 10 kilos. Deducting the amount contained in the water (which was certainly less than 0.1 gram.) the results are:—

	Grms.
Nitrogen fixed by the plants.....	1.4872
"      "      the soil.....	0.4810
Total.....	1.9682

The surface of the gravel covered about 0.2 sq. metre; this corresponds to 74.36 kilos. of nitrogen fixed by the plants and 24.05 kilos. fixed by the soil per hectare, or a total of 98.41 kilos.

II. A fragment of lucerne root covered with tuberculous growth was bedded in a flower-pot containing 4 kilos. of sandy soil: the weight of the (dry) root and contained nitrogen was estimated from a fragment of equal weight taken from the same root. The amount of nitrogen in the

soil was estimated before and after the culture, during the course of which the lucerne bore three crops, each of which was separately examined. The following table shows the amount of nitrogen contained in the 4 kilos. of soil:—

	In 100 Parts of Soil.	Total Nitrogen.
At the end of the experiment.....	0.1140	Grms. 4.560
At the beginning.....	0.0525	2.100
Nitrogen fixed by the soil.....		2.460

Thus the amount of nitrogen in the soil was more than doubled. The results also show that the lucerne with its crops contained 80 times as much nitrogen as the original root.

The experiments, like those of Hellriegel, Wilfarth and Berthelot, show conclusively that leguminous plants grow readily on soils which are poor in nitrogen, provided that their roots are furnished with tubercles containing bacteria. They produce abundant crops rich in nitrogen, and by means of their roots they fix nitrogen in the soil. They well deserve the name of *plantes améliorantes* given to them by the farmers.—D. E. J.

*On Air contained in the Soil.* T. Schloesing, jun. Compt. Rend. 109, 673–676.

THE author gives the results of a number of analyses of samples of air taken from the soil by a method previously described by him. The samples were generally taken at two depths in each instance, the one (25–30 cm.) from the soil properly so called, and the other (50–60 cm.) from the sub-soil. One-half of the samples were analysed completely, and were found to consist of oxygen, carbon dioxide and nitrogen without any measurable quantity of combustible gas; in the others the amount of carbon dioxide only was determined. With regard to the 23 samples taken from ploughed land, the author simply states that his results were in general similar to those obtained by Bous-singault and Lévy. There was no lack of oxygen, at any rate at the depth of 50–60 cm.; the amount of carbon dioxide increased noticeably with the depth.

Thirty-nine samples were taken from pasture lands which had not been ploughed for years, and which were therefore expected to yield more carbon dioxide and less oxygen. The analyses given show remarkable variations in the amount of carbon dioxide for the same soil at different dates:—Samples taken in June and September, yielding 3.54 per cent. and 0.9 per cent. respectively in one instance, and 8.8 per cent. and 1.4 per cent. in another. The amount of oxygen varied from 10 per cent. to 20 per cent. In general, the amount of carbon dioxide was found to increase with the depth; in the two cases where this rule was reversed the weather was hot and calm, and the author explains the exceptions as follows:—A period of fresh windy weather, accompanied by changes of barometric height (all favourable to renewal of the internal atmosphere), is often followed by hot close weather. The soil is heated sooner than the sub-soil, and is richer in oxidisable organic matter; as long as the calm weather lasts there is little interchange with the internal atmosphere, so that for a time the air in the soil may contain a larger proportion of carbon dioxide than that in the sub-soil. On the slopes of valleys the proportion of carbon dioxide was found to increase in passing down the slope, as might be expected from the density of the gas. This was even noticeable when samples were taken from places only a few metres distant from each other. The author objects to the phrase "confined atmosphere," usually applied to air contained in the soil, and points out the necessity of recognising its mobility. Gases are in any case more mobile than water, and their movements are assisted by every cause which alters their temperature, pressure, or chemical composition.—D. E. J.



*The Changes which Occur in Plants in Presence of Organic Acids.* L. Mangin. *Compt. Rend.* **109**, 716—719.

DE SAUSSURE has shown that the cactus is distinguished from other plants by evolving oxygen without absorbing carbon dioxide. The author has, however, succeeded in inducing these conditions artificially by injecting into the leaves of the laurel-rose, the lilae, &c., various organic acids, such as malic, citric and tartaric acids. Negative results were obtained with acetic, formic, oxalic and succinic acids. A three per cent. solution was injected into the leaf of the plant, and exposed to the sunlight for 4—5 hours. The author found that malic acid increases the exhalation of oxygen more than citric acid, and citric more than tartaric acid. The amount of oxygen also increases with the concentration of the acid up to a given point, the maximum being 3 to 4 per cent. Placed in the dark, the leaves injected with malic acid exhale a volume of carbon dioxide much greater than the quantity of oxygen absorbed, whereas under normal conditions the reverse is the case. Similar results are yielded with citric and tartaric acids. The presence therefore of certain organic acids produce in the leaf a double phenomenon. In the dark the disengagement of a larger volume of carbon dioxide than that of the oxygen absorbed takes place, and in the light the exhalation of oxygen without any corresponding absorption of carbon dioxide.

—J. B. C.

*On the Exhaustion of Land Cultivated without Manure, and the Value of the Organic Matter contained in the Soil.* P. P. Dehérain. *Compt. Rend.* **109**, 781—785.

COMPARISONS have been instituted between two soils, the one having had no manure for 12 years, the other being fertilised in the usual ways. The latter gave in the year of comparison a weight of beetroot equal to about 35,000 kilos., the former gave from the same area but 11,000 kilos. Experiments have been made on the soils to discover the causes producing such effects. There is no appreciable difference in the amounts of total phosphates or nitrogen, but there is in the case of the organic carbon, thus:—

—	1878.	1888.	Difference.
Lot 21, without manure.....	16.30	7.30	9.00
Lot 32, after dunging .....	16.60	16.00	0.60

As to the part played by the organic constituents of the soil, the following hypotheses have been advanced:—

1. That the humus-like matter acts as a sponge, retaining water throughout the summer for the benefit of the plant.

But it is shown that in 100 parts of earth rich in humus the quantity of water was 18.57, whilst in 100 parts of earth poor in humus there was 17.67.

2. That the production of nitrates and carbonic acid gas is aided by the slow combustion of the organic matter.

Here, again, however, analyses of the two soils show but little difference.

3. That the humus acts like a food to the plants.

That this is the correct explanation the author proved by cultivating beetroots in soils, the one of which contained much humus, whilst the other, though poor in this constituent, contained artificial manures. The differences in favour of the former, of the total weight of the plants, and the percentage of sugar, were most marked.—S. G. R.

*Manurial Experiments with Different Phosphates.* Ullbricht. *Der Landwirt*, **49**, 659—662.

BARLEY (*Hordeum vulgare*) was cultivated in a greenhouse, in glass pots, on sterile sand, to which was added a normal manure, and, in various pots, phosphoric acid in different chemical conditions. Moreover some pots contained, in addition, absorption material, indicated by A. in the table,

consisting of 53.7 grms. of ferric hydroxide, and 80.2 grms. of calcium carbonate, and sometimes humus acid (H. in the table) to the extent of 4.9 grms. The so-called oxyapatite was prepared by heating 3 mols. di-calcium phosphate with 4 mols. calcium carbonate, whilst di-calcium phosphate, heated by itself, gave the calcium pyrophosphate. The author takes these as representing two forms of combined phosphoric acid, whilst he recognises a third form in the free phosphoric acid in many superphosphates. Thomas slag probably contains the first two forms. The following table of results is taken from the more detailed table in the original:—

Proportion of Phosphoric Acid.	Phosphatic Manuring.	With or without Auxiliaries.	Yield in Grms. per Pot.		
			Grain.	Total.	
141.7	Mono-calcium phosphate {	..	10.36	22.85	
		A.	4.19	11.51	
		A. H.	6.62	16.72	
141.7	Free phosphoric acid {	..	10.00	22.91	
		H. A.	7.60	17.82	
		..	6.91	17.33	
141.7	Di-calcium phosphate {	H.	9.05	21.94	
		A.	4.99	11.82	
		H. A.	8.63	20.77	
283.4	Tri-calcium phosphate {	H.	0.28	3.27	
		H. A.	2.61	8.42	
283.4	Tetra-calcium phosphate {	H.	..	2.02	
		H. A.	0.45	3.94	
283.4	Oxyapatite {	H.	5.01	10.53	
		H. A.	8.13	19.86	
283.4	Calcium pyrophosphate {	H.	0.02	1.87	
		H. A.	0.22	5.31	
283.4	Ferric phosphate .... {	H.	0.62	3.75	
		H. A.	5.28	22.19	
283.4	Aluminium phosphates {	H.	6.43	13.44	
		H. A.	10.63	23.97	
283.4	Canadian apatite {	Finest powder {	H.	..	1.31
		H. A.	0.26	3.27	
		Coarse powder {	H.	0.61	1.00
		H. A.	0.17	2.38	
283.4	Thomas slag {	Finest powder {	H.	5.49	13.96
		H. A.	0.70	16.57	
		Coarse powder {	H.	2.68	6.74
		H. A.	4.84	12.42	

Noteworthy is the inactivity of the apatite, the superiority of fine slag over the coarse, the action of the absorbent on the soluble phosphoric acid, and of the humus acid on the di-calcium phosphate.—D. A. L.

*Manuring with Ammonium Sulphate and with Chili Saltpetre.* M. Maereker. *Bied. Centr.* **18**, 724—729.

THE experiments were made in 1888, when the meteorological conditions were as unfavourable as possible. The first series of experiments were made to ascertain the effect of using calcium carbonate as well as ammonium sulphate

for manuring, and the results are shown in the following table:—

	Increase in Kilos. per Hectare.		
	With Lime alone.	With Ammonia alone.	With Lime and Ammonia.
Barley grain .....	107·6	228·9	601·6
Oat grain .....	166·0	613·7	1,653·3
Wheat grain .....	67·0	177·5	237·5
Potato tubers .....	957·8	726·8	1,538·4
Mangel roots.....	720·0	5,420·0	4,640·0
Sugar beet roots .....	— 258·0	+ 2,217·0	+ 2,608·0

All crops, except one experiment with summer wheat, yielded a marked increase with ammonium salts alone, and all, except sugar beet, with chalk alone. Heavier dressings of ammonia yielded still larger crops. This was also the case in the next series of experiments made to ascertain the relative manurial value of ammonium sulphate and Chili saltpetre. For barley, the light dressings were 62·5 lb. Chili saltpetre and 50 lb. of ammonium sulphate; the heavy dressings being respectively 100 lb. and 80 lb. For oats, sugar beet, potatoes, and winter cereals the dressings were 100 and 150 lb. of Chili saltpetre and 80 and 120 lb. of ammonium sulphate per plot of  $\frac{1}{4}$  hectare. Some of the results are as follows, in kilos. :—

Crop.	Light Dressings.		Heavy Dressings.	
	Ammonium Salts.	Chili Saltpetre.	Ammonium Salts.	Chili Saltpetre.
Barley .. { Grain	2,160·0	2,252·0	2,433	2,432
{ Straw	2,831·5	2,938·5	3,159	3,249
Oats .... { Grain	2,628·5	2,591·5	2,916	2,820
{ Straw	4,742·0	4,732·5	5,184	5,235
Potatoes .....	17,450·0	17,349·0	18,709	20,176
Sugar beet .....	40,500·0	40,000·0	43,000	45,900

From these numbers and results obtained in another district it is concluded that light nitrogenous manurings with

either Chili saltpetre or ammonium sulphate have given an equal increase in yield with summer cereals, beets, and potatoes, but that heavy nitrogenous manuring applied to beets and potatoes in form Chili saltpetre has proved superior to the same quantity of nitrogen applied in the form of ammonium sulphate.—D. A. L.

#### Manurial Experiments on Light Sandy Soil. E. Güntz. Bied. Centr. 18, 796.

Six plots on a sandy soil received in 1885 dressings of manures in quantities based on the composition of an average rye-crop. They all of them received five times the indicated quantity of ash constituents, and in addition plots I., II., III., IV. had three times, and plots V. and VI. respectively, four times and twice the indicated quantity of nitrogen. Lupinus were grown in 1886 and 1887, and without further manuring a crop of summer wheat was taken in 1888, with the following results in kilos. per  $\frac{1}{4}$  hectare:

Plots.	I.	II.	III.	IV.	V.	VI.
Grain.....	291·88	235·63	193·38	345·63	336·88	365·63
Straw.....	621·25	572·50	491·25	698·75	675·00	701·25
Chaff, &c.....	42·50	36·12	29·87	40·75	44·25	31·87
Total .....	955·63	844·25	714·50	1,085·13	1,056·13	1,098·75

The low yield on plot III. is attributed to previous special and prolonged manuring with ammonium sulphate, and that on plot II. to previous manuring with lime.—D. A. L.

#### Experiments on Heavy Soil. E. Güntz. Bied. Centr. 18, 797—798.

Saxon white Zwiebel potatoes were cultivated on six plots, each a square ruthe (about 17 square yards) in extent, 66 eyes, weighing 2,860 grms., were sown in two rows on each plot with 625 grms. of Chili saltpetre and 818 grms. of potassium salts, besides which plots III. and IV. had 200 grms. phosphoric acid in 829·6 grms. of Thomas-slag powder, and plots V. and VI. the same quantity of phosphoric acid in 1,844 grms. of animal charcoal superphosphate.

The manures were mixed with fine mould and spread in the furrows. A detailed table of the results is given, from which the following numbers are taken:—

#### YIELD IN KILOS. PER $\frac{1}{4}$ HECTARE.

	Without Phosphoric Acid.		With Thomas Slag.		With Soluble Phosphate.	
Total tubers harvested.....	5,453·52	5,505·02	4,569·91	5,362·72	5,881·79	6,136·57
Percentage of bad tubers .....	5·74	11·23	7·77	3·71	5·69	5·08

—D. A. L.

#### “Clover Sickness” and its Remedy. Stand. Agric. Reps. March 1890.

No greater service, in recent times at least, has been rendered to the farmer by the entomologist than that of identifying the cause of the very troublesome plant disease known as “clover sickness.” The frequent growth of clover as a nitrogen-restoring crop is of obvious advantage, and in many parts of the country this is prevented by the disease in question, so that few farmers dare to sow clover more frequently than once in eight years, while on some soils it does not succeed as frequently as that. This subject is referred to in two annual reports on injurious farm insects, just issued, one by Miss E. A. Ormerod, consulting entomo-

logist to the Royal Agricultural Society, and the other by Mr. Charles Whitehead, for the Board of Agriculture. It is dealt with in greatest detail in the former publication, in which the evidence as to the cause of clover sickness being the cecideworm (*Tylenchus devastatrix*) is full and conclusive, confirming that which was obtained in a previous season. Better still, attention is directed to some experiments carried out by Mr. Wilks, a colleague of Sir John Lawes, at Rothamsted, which point to the probable discovery of an effectual remedy for the disease. Last spring, clover sickness was detected in one of the Rothamsted fields, and it appeared to be spreading. On the most diseased portion of the field a dressing of 3 cwt. of sulphate of potash and

1 cwt. of sulphate of ammonia per acre was applied early in April, on another portion 2 cwt. of sulphate of iron, and on a third plot half the quantity of sulphate of iron. By the middle of May it was found that the disease had entirely ceased to exist on each of the first two pieces of land, the clover growing most vigorously, especially on the plot dressed with sulphate of potash and sulphate of ammonia, on which there was not a gap to be seen. At the end of July the good effect of this application was found to have extended to the second crop of clover, while the disease showed no signs of existence on the plot to which 2 cwt. of sulphate of iron had been applied, though it did show itself where only half the quantity was used; but the growth was much more luxuriant where the mixture first named was put on. Mr. John Elder, of Uphall, Linlithgowshire, also cured a crop of clover of the disease in a previous season by the use of sulphate of ammonia and potash. A few more successful trials of the kind would place the effectiveness of this remedy beyond all question.

*Experiments on Grass Land near Reading. Stand. Agric. Reps. March 1890.*

AN account of the financial results of the experiments in the application of various manures to grass land which he has carried out during the last four years, at Dyson's Wood, near Reading, has been issued by Mr. Martin J. Sutton. Unfortunately, this is the final record of these valuable experiments as far as their present location is concerned, as Mr. Sutton has removed to a larger farm in order to find greater scope for his growing herd of Kerry and Dexter cattle. In the pasture, where the results have all along been most trustworthy, the greatest gains have been obtained from the use of certain manures applied twice during the last three years, which have turned out better than dressings applied on another set of plots in the same pasture twice during four years. Two dressings have given practically identical results. The gain per acre during three years from the use of 3 cwt. per acre of dissolved bones amounts to 7*l.* 15*s.* 9*d.*, while that arising out of the application of 1 cwt. of nitrate of soda and  $\frac{3}{4}$  cwt. of muriate of potash is 7*l.* 15*s.* 3*d.* The former of the two dressings is to be preferred, because stimulating nitrogenous manures injure the character of the herbage on permanent pasture. A mixture of 4 cwt. of basic elder, 1 cwt. of nitrate of soda, and 2 cwt. of kainit takes the third place, with a profit of 4*l.* 8*s.* 6*d.*, while for 3 cwt. of raw bone meal a credit balance of 3*l.* 15*s.* 9*d.* is shown, and 10 tons of farmyard manure left a gain of 3*l.* 14*s.* 3*d.* On the plots which have been under treatment for four years, two applications of 3 cwt. of superphosphate and 2 cwt. of kainit proved the most profitable dressing, the net gain for the period being 3*l.* 7*s.* 3*d.* This dressing was one of the most successful in other pastures, and should certainly be included in any future trials. The hay was valued each year at 4*l.* a ton, and the gain is the value of the produce in excess of that from the unmanured plots, less the cost of the manures.

*Experiments on Barley and Grass at Cirencester. Stand. Agric. Reps. March 1890.*

PROFESSOR KINCH has issued a full report of experiments on barley and grass, carried out by him on behalf of the Royal Agricultural College, Cirencester, last season. The mean yield of 10 plots of barley, dressed with 2 cwt. per acre of nitrate of soda, was 40 $\frac{1}{2}$  bushels per acre, as compared with 20 $\frac{1}{2}$  bushels obtained from the unmanured plots, while 14 tons of farmyard manure gave 37 bushels, and half the quantity 27 $\frac{1}{4}$  bushels. A dressing of 3 cwt. of superphosphate and 3 cwt. of kainit gave only 3 $\frac{1}{2}$  bushels per acre over the yield of the unmanured plots, and when these manures were added to nitrate of soda the yield was less than from the latter alone. Less produce was obtained when sulphate of ammonia, containing an equal quantity of nitrogen, was used instead of nitrate of soda. The

results of the grass experiments are as below, the increase being the excess over the produce of the unmanured plots:—

Manures per Acre.	Hay.	Increase.
None .....	Cwt. 23 $\frac{1}{2}$	Cwt. ..
12 tons farmyard manure .....	41 $\frac{1}{2}$	18
2 $\frac{1}{2}$ cwt. nitrate of soda .....	33	9 $\frac{1}{2}$
2 cwt. sulphate of ammonia .....	22 $\frac{1}{2}$	8 $\frac{1}{2}$
5 cwt. kainit .....	22 $\frac{1}{2}$	$\frac{1}{2}$
5 cwt. superphosphate .....	30 $\frac{1}{2}$	7 $\frac{1}{2}$
5 cwt. guano .....	31 $\frac{1}{2}$	8 $\frac{1}{2}$
5 cwt. superphosphate .....	41 $\frac{1}{2}$	18 $\frac{1}{2}$
2 $\frac{1}{2}$ cwt. nitrate of soda .....		
5 cwt. superphosphate .....	36 $\frac{1}{2}$	13 $\frac{1}{2}$
2 cwt. sulphate of ammonia .....		
5 cwt. kainit .....	31 $\frac{1}{2}$	8 $\frac{1}{2}$
5 cwt. superphosphate .....		
5 cwt. kainit .....	37 $\frac{1}{2}$	14 $\frac{1}{2}$
5 cwt. superphosphate .....		
2 $\frac{1}{2}$ cwt. nitrate of soda .....	35 $\frac{1}{2}$	12 $\frac{1}{2}$
5 cwt. kainit .....		
5 cwt. superphosphate .....	39 $\frac{1}{2}$	16 $\frac{1}{2}$
2 cwt. sulphate of ammonia .....		
5 cwt. kainit .....	39 $\frac{1}{2}$	13 $\frac{1}{2}$
2 $\frac{1}{2}$ cwt. nitrate of soda .....		
5 cwt. kainit .....	39 $\frac{1}{2}$	13 $\frac{1}{2}$
2 cwt. sulphate of ammonia .....		

As arranged here, the results of the several dressings of manure may be easily compared. It will be seen that the farmyard manure gave better results than anything except the mixture of superphosphate and nitrate of soda, but not nearly enough extra produce to pay for its cost. The increase from the use of nitrate of soda alone is not very great, and that from sulphate of ammonia is less. Kainit alone gave scarcely any increase, but did well in conjunction with either of the nitrogenous manures, and especially with nitrate of soda. Superphosphate alone did fairly, considering its small cost, and in conjunction with either of the nitrogenous manures it gave a considerable increase. The reduction of yield which took place when kainit was added to the mixture last named is puzzling, and may have arisen from difference of soil. In every comparison nitrate of soda alone, or in combination with other manures, beats sulphate of ammonia. In relation to these experiments, however, it is desirable to point out that the application of nitrate of soda or sulphate of ammonia—especially the former—while increasing the yield of hay for one season, deteriorates the herbage, whereas superphosphate and potash (in kainit) improve it.

PATENT.

*Process of Manufacture of Ammoniacal Calcic Phosphate, G. A. H. Gabet, Paris, France. Eng. Pat. 5340, March 28, 1889. 6d.*

See under VII., page 292.

## XVI.—SUGAR, STARCH, GUM, Etc.

*Invertase and the Inversion of Cane Sugar.* F. W. Tompson. Trans. Laboratory Club, 2, 63—71.

Most plants and animals are able to make use of cane sugar as a food material. Now it has been shown that cane sugar injected into the blood is removed unchanged by the kidneys, and that yeast is quite unable to ferment a solution of cane sugar if a small quantity of potash be present, which destroys its inverting action; whilst, on the other hand, when the cane sugar is first inverted, it rapidly disappears from the blood without elimination by the kidney in the first case, and is quickly and completely fermented in the second. It is thus evident that cane sugar, as such, is not assimilable, but requires to be inverted for use as a food. It has been found that the small intestine of man and the higher animals possesses this property of inversion, as also do all varieties of yeast yet examined.

The sugar cane and beet store up cane sugar as a reserve material; they have no power of inverting it during the first stage of their growth, but during the second (flowering and fruiting) this property is present in a marked degree.

The common method of artificially inverting cane sugar is by the action of dilute acids. Invertase may be used for this purpose; it is prepared from yeast by pounding with sand, washing the broken yeast cells with water and precipitating the filtered washings with alcohol, when a syrupy precipitate is obtained which may be dried and powdered; it is called invertase or invertin, although it probably contains only a small amount of the pure ferment. The published analyses of this body vary greatly. It has no action on any other carbohydrate than cane sugar, which it hydrolyses into a mixture of levulose and dextrose, commonly called invert sugar. Alkalis destroy this action; very dilute acids (1:100,000) increase the action, but quantities only slightly greater destroy it. It is able to invert many times its weight of cane sugar and is probably not destroyed by so acting. The rapidity of the inversion increases with the temperature, being most active at 160°—170° F., but a slightly higher temperature destroys it. As the vitality of the yeast cell is destroyed considerably below the temperature of greatest activity of the invertase, the author has utilised this fact in a patented process for inverting sugar commercially, first heating the cane sugar solution to 160°—170° F. and then introducing the yeast, whereby all fermentative power is destroyed whilst the inverting power is unimpaired. After standing some hours the sugar is completely inverted.—A. L. S.

*Xylose.* H. J. Wheeler and B. Tollens. *Annalen*, 254, 304—320.

This second pentaglucose,  $C_5H_{10}O_5$ , is obtained from wood gum (Holzgummi) by hydrolysis, the yield amounting to about 33 per cent. It crystallises in needles melting at 145°, about 10° lower than the isomeride, arabinose. Its specific rotation is (+) 18°—19°; at the moment of solution much higher numbers are obtained (Biration) e.g.  $(\alpha)_D = 85.86^\circ$ , after being five minutes dissolved.

It yields no levulinic acid on boiling with hydrochloric acid. Heated with sulphuric acid diluted with 3 vols. of water it yields furfuran; converted into furfuranide the yield of the latter is estimated as 25.5 per cent. The products of oxidation by nitric acid contain trihydroxyglutaric and trihydroxybutyric acids, but no mucic or saccharic acid. The osazone (m.p. 155°) has the formula  $C_{17}H_{26}N_4O_8$ . The molecular weight determined by Raoult's method is 151—156.—C. F. C.

*Researches on Wood Gum (Holzgummi).* H. J. Wheeler and B. Tollens. *Annalen*, 254, 320—333.

*Wood Gum from Beech Wood.*—The analyses of the authors gave numbers corresponding with the formula  $C_6H_{10}O_5$ , calculated with exclusion of the ash constituents (2.3 per

cent.). For the determination of rotation it was necessary to dissolve in a solution of sodium hydrate (2 per cent.), in which condition the number —69.62 was obtained, as the mean of 14 readings. By sulphuric acid hydrolysis (Stone and Tollens) and conversion of the furfuran into furfuranide 22.6 per cent. of the latter was obtained.

*Wood Gum from Fir Wood.*—The yield obtained was small, viz., 0.4 per cent. By hydrolysis, xylose was formed and identified after isolation.

*Wood Gum and Xylose from Jute.*—By treatment of the fibre-substance with 5 per cent. sodium hydrate solution, a derivate was obtained and isolated; the yield was 1.7 per cent., and the substance on combustion was found to contain C 42.9, H 6.3. To isolate xylose the original fibre was operated upon, and a yield of 6 per cent. of the pure substance obtained. This observation accords with the researches of Cross and Bevan (*J. Chem. Soc.*, 55, 200) in so far as they have shown that furfuran is obtained in quantity as a final product of hydrolysis of the fibre-substance; the source of which, the authors show, is probably the pentaglucose in question, the molecule of which is represented in the lignone complex.

Substances yielding pentaglucoses on hydrolysis may be generally recognised by means of the well-known reaction with phloroglucol and hydrochloric acid.

The brilliant red colouration is obtained with the following: (a.) which yield arabinose on hydrolysis:—Gum arabic, cherry gum, tragacanth, bran, quince mucilage, and the cellular tissue of the beet; (b.) which yield xylose:—Jute, wood gum (from beech and fir woods), fir woods, husks of cereals, &c. The reagent is best employed in the form of a saturated solution of the phenol in hydrochloric acid of 1.19 sp. gr. The absence of nitric acid must be ensured, as it determines a similar colouration of the reagent.

In conclusion the authors point out that wood gum is distinguished from the lignocelluloses by solubility in alkaline solutions in the cold, whereas they confirm the conclusions of Cross and Bevan (*loc. cit.*) in stating that the former are not resolved by simple hydrolysis.—C. F. C.

*On Compounds of Raffinose with Bases (Raffinosates).*

K. Beythien and B. Tollens. *Annalen*, 255, 195—213.

THE raffinose used was obtained from strontium molasses, by pressing the honey-like syrup, which contained a number of long needle-shaped crystals; recrystallising the solid mass from water, again pressing and dissolving the mass in 60—70 per cent. alcohol, treating with charcoal and crystallising. As the preparation, although in other respects satisfactory, yet reduced Fehling's solution slightly, it was redissolved in half its weight of water, a little slaked lime added, warmed until the reducing power disappeared, then mixed with three parts of 95 per cent. alcohol, treated with charcoal, and the small quantity of lime in solution precipitated by carbon dioxide. The raffinose crystallising from this solution was quite white, did not reduce Fehling's solution in the slightest, and 5 grms. of the dry substance in 50 cc. of water had at 20° C. an optical activity equivalent to  $(\alpha)_D = 104.4$ . The crystals had a composition corresponding to the formula  $C_{18}H_{32}O_{16} \cdot 5 H_2O$ . Before the authors took up the work only the sodium raffinosate was definitely known, although there were reasons for supposing that raffinose formed compounds similar to cane sugar with calcium and strontia, as raffinose is found in quantity in the liquid obtained when the strontium or calcium compound formed in purifying sugar molasses is decomposed by carbon dioxide.

They have prepared the following compounds:—

*Strontia-raffinosate.*—5 grms. of raffinose, 10 grms. of purified crystalline strontia, and 20 cc. of water are placed in a flask furnished with a reflux condenser, and heated 4—6 hours on a salt-water bath. The granular precipitate is then filtered hot by the suction pump and washed first with hot strontia water and then with alcohol, commencing with weak and ending with absolute, and lastly with ether. The white powder is exposed over sulphuric acid for some days, and has then a composition corresponding with the formula  $C_{18}H_{32}O_{16} \cdot 2 SrO, 11 H_2O$ . When the reaction takes

place in the presence of weak alcohol the same compound is formed. If the substance be heated up very slowly to  $80^{\circ}$  in a stream of dry air it loses its molecule of water and remains colourless, but if it be heated up too rapidly it becomes yellow, and it is then almost impossible to remove all the water. Whatever proportions of materials are used no other compound of this nature is formed. It cannot be easily prepared by proceeding exactly as in the preparation of the sucates. The theoretical quantity of raffinose is not obtained on decomposing this compound by carbon dioxide, so that some of it must be inverted or otherwise decomposed in this reaction.

**Baryta-raffinose.**—The attempt to prepare a baryta compound in the same way as the strontium compound was unsuccessful. On dissolving 3 grms. of crystallised baryta in 60 grms. water, and adding as much alcohol as possible without causing a precipitate, and then a solution of 2.5 grms. of raffinose in 5 grms. of water, a precipitate is thrown down which adheres to the side of the vessel, and on drying is found to have a composition corresponding to the formula  $C_{18}H_{32}O_{16}.BaO$ . If half as much again of baryta be employed a compound is obtained whose composition agrees fairly well with the formula  $C_{18}H_{32}O_{16}.2 BaO$ .

**Lime-raffinose.**—3 grms. raffinose, 100 cc. of water and 0.4 gm. of calcium hydrate digested at the ordinary temperature, filtered and heated for some hours on the water bath, gives a precipitate which after having been filtered by the suction pump, washed first with hot water and then with alcohol of gradually increasing strength, and lastly with ether and dried over sulphuric acid, possesses a composition corresponding to  $C_{18}H_{32}O_{16}.3 CaO, 2 H_2O$ . It loses its water when heated to  $100^{\circ}$  in a current of dry air.

**Lead Oxide-raffinose.**—A compound is obtained, having the formula  $C_{18}H_{32}O_{16}.3 PbO$ , either by adding a solution of lead acetate (25 cc. of a sp. gr. 1.232, to which as much ammonia as possible has been added without producing a precipitate) to a solution of raffinose (5 grms. in 20 cc. water); or by precipitating with alcohol a solution containing 3 grms. of raffinose in 10 cc. of water and 10 cc. of the solution of lead acetate, and drying the precipitate obtained in either way at  $100^{\circ}C$ . One of the authors pointed out previously (*Vereinzeitschrift*, 39, 748) that even dilute raffinose solutions are precipitated by ammoniacal lead acetate, but that the precipitation is not complete.

**Soda-raffinose.**—In a former examination of these compounds (*Ann.* 232, 182; this *Journal*, 1886, 243) Rischbieth and Tollens came to the conclusion that the formula of crystallised raffinose was  $C_{36}H_{64}O_{32}, 10 H_2O$ , but in consequence of the experiments of Tollens and Mayer (*Ber.* 21, 1566) and Brown and Morris (*J. Chem. Soc.* 1888, 610), with Raoult's freezing method and of De Vries' (*Compt. Rend.* 106, 751) on the diffusion coefficient of raffinose solutions, which all point to a formula  $C_{18}H_{32}O_{16}, 5 H_2O$ , the authors have considered a further examination to be necessary.

A strong aqueous solution of raffinose is added to sodium alcoholate contained in a mortar. On adding absolute alcohol a tough granular precipitate is formed which adheres to the mortar; the precipitate is purified by resolution in water and reprecipitation with alcohol and rubbed down first with absolute alcohol and then with ether. According to the amount of sodium used, the amount of the element in the resulting precipitates is either one, two, three, or four atoms to one molecule raffinose; the authors consider that two compounds only are formed, having respectively the formula  $C_{18}H_{31}O_{16}Na$  and  $C_{18}H_{31}O_{16}Na.OH$ .—A. L. S.

*On the Behaviour of Inverted Raffinose with Phenylhydrazine.* K. Beythien and B. Tollens. *Annalen*, 255, 214—216.

It has been observed that the inversion of raffinose, by heating with dilute sulphuric acid, occurs in two stages. In the first, the optical activity is almost halved; this occurs after a short heating. In the second, the optical activity falls to about  $20^{\circ}$ , and levulose, galactose, and dextrose are

produced; this is completed only after several hours' heating. One of the authors and others have already investigated the first stage of the inversion, and levulose has been isolated but not crystallised from the products.

The authors have sought to throw further light on this stage. They digested 10 grms. of raffinose, 90 cc. of water, and 9 cc. of hydrochloric acid (sp. gr. 1.19) for 10 minutes at  $68^{\circ}C$ ; after cooling, a slight excess of silver carbonate was added and the filtered solution made up to 200 cc. This was heated on the water-bath with 20 grms. of phenylhydrazine and 40 grms. of acetic acid. The yellow precipitate was filtered out and digested with acetone. The yellow mass remaining was recrystallised from alcohol, and was found to be identical with dextrosazone and levulosazone; but as it has already been observed that the glucose producing this is strongly levo-rotatory, it follows that this glucose is levulose. They agree with Scheibler (*Ber.* 22, 1678; this *Journal*, 1889, 718) in thinking that in partly inverted raffinose, besides levulose, there exists a compound of dextrose and galactose, as galactose will not crystallise out of these solutions. They have not obtained mannose-phenylhydrazine from this solution as Fischer (*Ber.* 22, 368; this *Journal*, 1889, 297) has indicated. The optical activity of the partly inverted solution was found to be  $50.9^{\circ}$  and 1 cc. of Fehling's solution was reduced by 0.0077 gm. of inverted raffinose.—A. L. S.

*Observations on the Melting Points of the Osazones and the Use of Phenylhydrazine.* K. Beythien and B. Tollens. *Annalen*, 255, 217—221.

In determining the melting points of the osazones, the authors find that it is very important that the temperature be raised quickly, and for the identification of an unknown osazone they recommend that the melting point be determined side by side with the osazone which it is supposed to be. They find, for instance, that the melting point of levulosazone varies from  $208^{\circ}$  to  $185^{\circ}$ , when the time of heating varies from 13—101 minutes. A point of importance in the preparation of the osazones is *not* to completely evaporate the mixture of hydrazine and sugar, as Löw has done in preparing the so-called formose derivative. On evaporating to dryness 15 grms. of phenylhydrazine hydrochloride, 22.5 grms. of sodium acetate, and 150 grms. of water on the water-bath, sodium chloride crystallised out and a reddish oil which crystallised on cooling was obtained. The salt was dissolved out with water, and the residue recrystallised from alcohol was found to be monoacetyl-phenylhydrazine, m.p.  $128.5^{\circ}$ . The authors think that this might be mistaken for an osazone. Tollens, in a note, states that phenylhydrazine produces nasty sores if allowed to get on the hand.—A. L. S.

*On the Formation of Lactic Acid by the Action of Bases on Raffinose and Cane Sugar. Raffinose not formed by the Action of Lime or Strontia on Cane Sugar.* K. Beythien, E. Pareus, and B. Tollens. *Annalen*, 255, 222—228.

THE observation (see page 310) that unless strontia-raffinose is dried very gradually it becomes yellow, led the authors to make the following experiments:—

Raffinose (40 grms.) and crystallised strontia (90 grms.) were boiled with water (1,000 grms.) for three days of eight hours each in a flask with reflux condenser. The raffinose at first precipitated gradually redissolved; the solution was filtered, and the strontia removed by the slightest excess of sulphuric acid. The filtered solution was shaken up with ether, the ethereal solution separated and evaporated, when 0.6 gm. of an acid syrup remained; this was diluted with water, boiled with zinc oxide, which it dissolved, and filtered. The filtrate gave crystals, which were found to be zinc lactate. Raffinose was recovered from the aqueous solution from which the ether was separated. Cane sugar treated in the same way yielded the same amount of lactic acid. Cane sugar treated similarly with its own weight of lime (as milk of lime) gave only 0.1 per cent. of lactic acid; when, however, the action was allowed to go on for double the time

(48 hours spread over 5—6 days) 1 per cent. of lactic acid was obtained.

It has been stated that raffinose is formed during sugar refining by the action of lime or strontia on cane sugar. The liquid obtained in the last-mentioned experiment (300 grms. of cane sugar and 300 grms. of lime with 6 litres of water, heated for 48 hours at the boiling point) freed from lactic acid was evaporated. Eight grms. of the syrup (containing 5 grms. of solid matter) were oxidised with 60 cc. of nitric acid (sp. gr. 1.15), the solution evaporated to one-third of its volume, and allowed to stand; not a trace of mucic acid crystallised out. As raffinose, when treated in this way, yields mucic acid, it is evident that no raffinose is formed by the action of lime on cane sugar. The same result was obtained when a similar experiment was made, using strontia instead of lime.—A. L. S.

*On Lactic Acid from Molasses.* K. Beythien, E. Pareus, and B. Tollens. *Annalen*, **255**, 228—229.

ALTHOUGH lactic acid, as shown in the previous abstract, must be formed in sugar refining, besides what may be produced by decomposition or fermentation of the beet-roots, yet no mention has been made of it in the literature of the subject. The authors have estimated the amount of lactic acid in a sample of molasses; 50—200 grms. of molasses from different sources were diluted with half their weight of water, made just acid with sulphuric acid and shaken up with ether. The ethereal solution, smelling of butyric acid, was evaporated on the water-bath with a little water until this smell disappeared, and then converted into the zinc salt; this was purified by precipitation from its solution with absolute alcohol, and, after treatment with charcoal, recrystallised. It was found to be zinc lactate, equivalent to 0.5 per cent. of lactic acid.—A. L. S.

*On Sorbite.* C. Vincent and Delachanal. *Compt. Rend.* **109**, 676—679.

SORBITE occurs in all fruits of the order *Rosaceæ*, such as pears, apples, medlars, cherries, plums, prunes, peaches, and apricots. Some of these are particularly rich in sorbite. From pears the authors have extracted 8 grms. of sorbite per kilo., and from eberries and prunes 7 grms. per kilo.

*Action of Hydriodic Acid upon Sorbite.*—When sorbite is acted upon by a large excess of hydriodic acid and distilled, a mixed product is obtained, consisting of hydriodic acid (and dissolved iodine) together with a dense liquid insoluble in water. This liquid is easily separated by decantation, and consists essentially of hexyl- $\beta$ -iodide,  $C_6H_{13}I$ , coloured brown by excess of iodine. 150 grms. of iodine are gradually added to 90 cc. of water and 35 grms. of red phosphorus contained in a half-litre retort, 60 grms. of crystallised sorbite are then introduced, and the whole gently heated, a current of carbon dioxide being passed through the apparatus. As soon as the reaction begins the heating is stopped; after it has finished the retort should be again heated, so as to distil over the last traces of iodide. The yield of iodide obtained is 63 grms. (theoretical yield = 66.6). The crude iodide, after washing and drying, boils at  $167^\circ$ , under a pressure of 753 mm. Heated with alcoholic potash, it saponifies readily, and on distillation the alcohol is obtained saturated with the hydrocarbon, which separates out on addition of water. The crude hydrocarbon, after fractional distillation, yields  $\beta$ -hexylene,  $C_6H_{12}$ , boiling at  $68.5^\circ$  (735 mm. pressure). The constitution of this is shown by the fact that, when oxidised with sulphuric acid and bichromate, it yields acetic and butyric acids.

The authors find that mannite, when treated with hydriodic acid, yields the same quantity of hexyl iodide; also that this, on treatment with alcoholic potash, yields the same products as in the case of sorbite, and in the same proportion.—D. E. J.

*On a New Sugar of the Aromatic Series.* Maquenne. *Compt. Rend.* **109**, 812—814.

THERE is an article known in commerce as "Pinite," which is a sugar derived from *Pinus lambertiana*. In its ready solubility in water, its slight solubility in alcohol, and its crystalline form it resembles the pinites of Berthelot. It is, however, more dextro-rotatory than pinites. It is proposed to call it  $\beta$ -pinites. Its formula lies between  $C_{12}H_{22}O_{11}$  and  $C_{12}H_{20}O_{10}$ , and it closely resembles sennite, with which it may be identical. It is readily attacked by warm hydriodic acid. The product so produced, when purified by precipitation from alcohol, is very soluble in water, almost insoluble in alcohol, and quite insoluble in ether. Its melting point is  $246^\circ C$ . It exactly corresponds in composition to the glucoses and its molecular weight, as determined by Raoult's method, is  $176^\circ$ — $178^\circ$ . It is an aromatic derivative, and is isomeric with inosite. The name  $\beta$ -inosite has been attached to it.—S. G. R.

*On  $\beta$ -Inosite.* Maquenne. *Compt. Rend.* **109**, 968—970.

THE author has shown (see previous abstract) that by heating pinites with an excess of hydriodic acid at  $120^\circ$ , methyl iodide is formed, together with a new sugar, named  $\beta$ -inosite by the author. Now this sugar is an isomer of ordinary inosite. Its chemical properties are the same as the latter. Thus a benzene additive compound may be prepared from it, which is a derivative of hexamethylene, so far as physical properties are concerned. It is distinguished by far greater solubility, higher melting point, by its crystalline form, and that of its ethers, which have no resemblance whatever to the corresponding compounds of  $\beta$ -inosite. Finally, the new sugar also turns the plane of polarised light to the right ( $\alpha_D = 65^\circ$ ). The latter property may be put down to a dissymmetrical constitution of  $\beta$ -inosite, which find expression in the fact that two hydroxides are joined to the same carbon atom.

Pinites is a mono-methyl derivative of  $\beta$ -inosite. Its composition is  $C_{12}H_{22}O_{11}$ , and it is therefore an isomer of bornesite and quebrachite, but differs both in composition and properties from quebrachite.—A. R.

*On Matézite and Matézo-dambose.* C. Combes. *Compt. Rend.* **110**, 46—47.

MATÉZITE is a sugar isolated by Girard (*Compt. Rend.* **76**, 995) from the juice of the caoutchouc of Madagascar, from which, by treatment with hydriodic acid, he obtained matézo-dambose, a sugar isomeric with the glucoses. Pinites, obtained from *Pinus lambertiana*, and called  $\beta$ -pinites (see two preceding abstracts) to distinguish it from Berthelot's pinites, gives on treatment with hydriodic acid a sugar isomeric with inosite, which is hence called  $\beta$ -inosite. By comparing their melting points and their rotatory power the author shows that matézite and  $\beta$ -pinites are the same body and that matézo-dambose and  $\beta$ -inosite are similarly one and the same thing.—S. G. R.

*Melitriose and Melibiose.* C. Scheibler and H. Mittelmeier. *Ber.* **22**, 3118—3124.

THE authors (this Journal, 1889, 718) have previously shown that the inversion of melitriose by means of dilute acids takes place in two stages, in the first of which levulose and melibiose are produced, the melibiose being subsequently split up into dextrose and galactose. In the present communication the authors prove the identity of melitriose, or raffinose, with Berthelot's melitose. This is contrary to the opinion held by Berthelot himself, that melitose is a compound of raffinose and eucalyn. The authors have prepared melitose from cotton-seed flour, according to Berthelot's directions, and find it to be identical in every respect with melitriose. They further find eucalyn to be identical with melibiose. Invertin acts upon melitriose in a similar manner to dilute acids. Melibiose and levulose are first formed, the melibiose being further converted by prolonged treatment with





than to a very partial refinement, in the mere ridding of these raw products of much of their molasses, without attempting any modification of the structure. It is the European refiner alone who develops pure sugar by eliminating all admixture of uncrystallised syrup, and thoroughly purifies it.

It is partly due to neglect in the selection of seed, and to its use in excess quantity in the North-West Provinces, that, whilst the yield of cane reaches, in Barbadoes and Upper Egypt, from 30 to 36 tons per acre, and in Demerara from 35 to 40, it does not ever in India attain even half such bulk. Apart from these causes, however, there are other radical defects, traceable in the exhaustion of the soil by too rapid a rotation of crops and neglect of open fallows; in the absence of the application of dry manures containing phosphates or mineral matters; in late and irregular planting; in careless surface sowing; in defective irrigation (the crop being submerged); and in the exclusion of air and light from the roots; whilst the crop is too often cut before maturity or too late (when the heat rapidly acidifies the juice). Nor is the expression of the cane comparatively more satisfactorily effected. In the West Indies, up to some 50 to 60 years ago the average of juice extracted represented, roughly, about half the weight of the cane pressed. With improved methods, from 70 to 85 per cent. is now attained, whilst India would appear still content with an average of 50 to 55. By the trituration of the ordinary native *kolhu* substances other than the juice, such as vegetable acids, are moreover pressed out in considerable quantities, owing to the breaking up of the fibres: it is probably due to this cause that European mills are finding such favour. Nor is the slovenly, careless method pursued of heating the juice less open to exception. Where sugar, even in solution, is subjected for too long a period to the action of excessive heat, such is its excessive sensitiveness that it becomes gradually changed into a mixture of dark substances, which are absolutely incapable of crystallisation. When separated finally from the sugar these unreduceable substances take the form of molasses or treacle. How much room for improvement there is yet in these provinces may be gathered from the fact that whilst the quantity thus lost is now reduced to a minimum of 2 per cent. in the West Indies, the loss on glucose in the North-West is still about 12 per cent., or 15 maunds of dry sugar, to 33½ of molasses. Whilst in the returns for the West Indies the weight of the dry sugar on each acre of production is three times as much as that of the molasses, the quantities are reversed in the East, and give twice as much molasses as dry sugar.

The courses in dealing with the juice here are two. It is either boiled to a dry consistency, which if clarified by alkalis is called *shakur*, or unclarified is known as *goor*; or it is boiled to a semi-liquid consistency as *rab*, when it is pressed in bags with the feet, developing uncrystallised syrup. The further treatment of this last is to strain it in crates to a substance which, when kneaded in the sun by trampling, becomes the *kacha chini* of internal commerce. The only further developments of semi-refined sugars in native hands are effected by boiling in milk and straining. The cane juice is allowed to fall unfiltered into earthen vessels accompanied by cane trash and dirt, whilst from this cause, and from the cutting of the cane into short lengths; through carelessness, exposure, and loss of time, it has often commenced actively to ferment before even being removed to the boiling pan. It has been found that the native raw sugars exported to Europe from India are generally of so miserable a quality as to prove valueless for refining purposes, and only to be capable of use by brewers. As to quantity, the amount of *rab* expressed per acre is in the North-West roughly about 4,500 lb., whilst of *goor* the result is less by about 500—a very low average. The average out-turn of dry sugar (Muscovado) in Barbadoes often exceeds 4 tons per acre. In Singapore and the Straits it is about 3 tons 4 cwt. undrained to 2½ dry. The vicinity of Calcutta gives about 1 ton 3 cwt.

## PATENT.

*Improvements in or relating to the Manufacture of Maltose or to the Fermentation of Amylaceous Matter.* W. P. Thompson, Liverpool. From "La Société Générale de Maltose," Brussels, Belgium. Eng. Pat. 5564, April 1, 1889. 6d.

In the saccharification of amylaceous matters by malt a minimum quantity of malt should convert into saccharine a considerable quantity of amylaceous matter. In practice this is not found to be the case, and a relatively large quantity of malt has to be used to arrive at even an imperfect degree of saccharification. This fact is explained by the development in the worts of harmful ferments which weaken the action of the diastase. The difficulty has been overcome by treating the malt with an antiseptic which will destroy the other ferments whilst having no injurious effect upon the diastase. For this purpose hydrofluoric acid is used, and by adding to 1 litre of infusion of malt containing about 200 to 300 grms. of malt, two or three drops of commercial hydrofluoric acid, it can be preserved for 8 or 10 days without losing any of its saccharifying power. For such an infusion the temperature of maximum saccharification is 25–30° C., and not 50–60°, which is usually employed.

In the alcoholic fermentation of yeast hydrofluoric acid has been found to exert a very beneficial action, preventing the development of other ferments and thus improving the yield of alcohol. It is also found that commercial hydrofluoric acid, in the proportion of 5 to 15 grms. per hectolitre of wort, can be employed as a simple preservative, and that the preservative action extends not only to the diastase, but to the yeast.—A. J. K.

## XVII.—BREWING, WINES, SPIRITS, Etc.

*Acidity of Malt Liquors from a Medical Standpoint.*

H. L. Jones. Trans. Laboratory Club, 2, 72–76.

GOUT is produced by the deposition in the tissues of urate of sodium. This is due, not to the excessive production of uric acid by the gouty, but to imperfect elimination of it. The elimination of uric acid by the kidneys may be controlled by certain drugs, a dose of acid decreasing the excretion of urates, whilst alkalis have the contrary effect.

It is stated that certain alcoholic drinks, such as spirits and light wines, do not tend to produce gout, whereas others, such as port, beer, porter, &c., do have these evil effects. This is due, in the case of the heavy wines, to their acidity, coupled with their poorness in the alkaline salts of organic acids, which in the body would be oxidised to alkaline carbonates, and thus neutralise the effect produced by their acidity; whilst in the case of malt liquors, although, besides acidity, there are alkaline organic salts, these latter do not become alkaline carbonates when oxidised by the body, as there is sufficient phosphoric, sulphuric and hydrochloric acids to prevent this.—A. L. S.

*Some further Notes on the Application of the Polarimeter in Brewing.* J. Heron. Trans. Laboratory Club 2, 77–82.

THE determination of the specific rotatory power of the wort is recommended as giving valuable information regarding the relative amounts of maltose and dextrin present (this Journal, 1888, 259–276).

The author finds that the same malts mashed under similar conditions give worts having the same optical activity, and that when the mashing is conducted at a higher temperature the optical activity increases.

The following analyses are given comparing the composition of the beer after fermentation with the specific rotative power of the wort.

—	A <sub>1</sub>	A <sub>2</sub>	C <sub>1</sub>	C <sub>2</sub>	D <sub>1</sub>	D <sub>2</sub>
Matter fermented .....	58.9	53.7	68.9	68.3	63.6	58.9
Maltose unfermented .....	12.5	15.0	8.0	8.3	8.7	12.5
Dextrin .....	9.2	20.7	7.0	8.9	10.2	9.2
Undetermined .....	19.4	10.6	16.1	14.5	19.5	19.4
Polarisation of wort (α) <sub>D</sub> ..	108.3	122.7	103.5	105.0	118.4	119.0

A<sub>1</sub> and A<sub>2</sub> are different malts mashed under the same conditions; C<sub>1</sub> and C<sub>2</sub> are different malts, polarise the same and yield beer having similar compositions; D<sub>1</sub> and D<sub>2</sub> the same malts mashed at different times under similar conditions.

The wort for examination should be taken after it has been well boiled up in the copper, as it is found that samples taken at different periods whilst the wort is running into the copper have different optical activities. The wort should be collected free from hops, made to a gravity of 1.040 at 60° F., cleared with a few drops of finings, and after filtration the optical activity read off in the 200 mm. tube.

—A. L. S.

#### *The Use of Air in Malting.* H. Stopes. Trans. Laboratory Club, 2, 48—62.

In malting, as carried on in this country, little is done to modify or alter the quality of the air having access to the grain during the different stages of the operation, although the amount of moisture, temperature, &c., has a very important effect on the result. As an excess of moisture may be beneficial at one stage of the malting and noxious at another, and in the same way a temperature favourable for one stage may be unfavourable for another, and so on, so the use of air in malting must be considered separately at each stage.

**I. Storage of the Barley before Steeping.**—Before malting is commenced it is necessary that the barley should be sweated, that is the excess of moisture must be driven off the corn, in the course of which certain molecular changes take place in its constituents. It may either take place (1) normally in stack; (2) in bulk; (3) artificially on kiln. It is almost impossible to exercise any control over the sweating in stack, from self-evident reasons; in sweating in bulk the moisture is driven off too slowly and without proper access of air, and so the corn is likely to grow mouldy; sweating on kiln is an easily controlled process, as the amount and temperature of the air employed can be easily regulated and consequently the operation can be carried out with the greatest success.

**II. Steeping.**—Before steeping the malt may be with advantage graded and the dirt and dust removed by an air blast. The corn should not be allowed to absorb water before this stage as this would in some measure neutralise the value of the sweating. During the steeping the water dissolves a certain amount of albuminous matter, salts, sugars, &c. from the corn; it should be frequently changed so that these substances may be thoroughly removed, and between the successive steepings, the air should have free access to the corn.

**III. Couching and Growth on the Floor.**—At this stage the amount of turning, &c., depends on the temperature and amount of moisture in the air, and must be so arranged that while the air has free access to the growing corn, yet the corn is not deprived of too much of its moisture, which would stop its growth.

**IV. Withering.**—Here the air should have just the opposite effect, i.e., it should dry the grain. When this is not done with a proper quantity of pure and dry air there is much danger from mould.

**V. Drying.**—A proper control over the amount and temperature of the air is here of great importance; the air should be dry and of medium heat. If the temperature be not sufficiently high the resultant malt will be likely to produce unsound beer, and if the temperature be too high there may be excessive colouration, and in exceptional cases the diastatic power of the malt might be destroyed.—A. L. S.

#### *The Boiling of Wort.* C. F. Thatcher. Trans. Laboratory Club, 2, 83—92.

The boiling of wort with hops results in the precipitation of a certain amount of nitrogenous matter and in the extraction from the hops of substances having antiseptic properties, and others communicating a pleasant bitter taste to the beer, &c. In order that this may be done properly it is necessary that the boiling should be vigorous; the author gives a theory explaining this. He recommends that the copper be so set that the hot gases from the fire first impinge on the centre of the bottom of the copper and from there travel to its sides. He considers that the want of success which steam boiling has hitherto met with is not due to some inherent fault of the process, but rather to faulty plant; and recommends that the steam entrance to the jacket be below the centre of the bottom of the copper, that steam at not less than 50—60 lb. pressure be employed, and that proper arrangements be made to remove condensed steam so as to prevent its accumulation.

He, however, recommends fire boiling until it be conclusively proved that steam boiling has been used with practical success.—A. L. S.

#### *Some Points in Connexion with Practical Brewing.* E. C. Hansen. Trans. Laboratory Club, 2, 93—108.

**I.—On the Bacteriological Analysis of the Water and the Air for Brewing Purposes.**—The two processes which are in use for this purpose consist either in mixing a definite quantity of the water with liquid meat extract gelatin or with wort and beer, the culture media having been in the first instance sterilised, and forcing the infected solution. The author has made comparative experiments with these two methods, and finds that they yield very different results. For example, 1 cc. of an ordinary water gave in a series of cultures in beer, no vegetations, in a series of cultures of wort, an average of four vegetations, and in a series of Koch's gelatin, 100—1,500 vegetations; also that some organisms which are decidedly harmful to beer will not develop in meat extract gelatin unless infected with very vigorous cultures, such as for instance *Bacterium Pasteurianum* and various species of *Saccharomyces*. On the other hand it has been stated that some varieties of *sarcina* do not readily grow in sterilised wort whilst they do in nutritive gelatin. The author throws doubts on the harmful nature of these, as Petersen has observed a perfectly sound beer containing a number of these organisms.

He recommends that wort should in all cases be used as the culture medium, as organisms harmful to beer will not always grow in it after fermentation, and cannot agree with those chemists who make use of the gelatin culture method for the purpose of determining the worst of the water and air, but considers it, when viewed in the light of his experiments, as likely to yield to erroneous results.

**II.—Pure Yeast Culture and its Application in Top Fermentation Breweries.**—In his researches on bottom fermentation the author found that certain defects of beer, such as yeast turbidity, disagreeable flavours and smells, &c. were due to the presence amongst the yeast of certain varieties of yeast, and he was able to isolate and obtain in quantity a pure yeast which produced a beer having none of these disagreeable characters. This pure yeast has been largely adopted by low fermentation breweries, and is found to possess the following practical advantages: It prevents disease in the beer, and it gives a definite and precise result. It has been found that the high fermentation yeasts used in this country consist of several varieties of *saccharomyces*, and the author

considers that if these were isolated and examined that one of these varieties would be found to produce a beer of exceptional quality, and to be better in every way than the impure yeast now used. This is said to have been carried out with success by Joergensen, at Deventer in Holland, the fermentation being carried out at  $8^{\circ}$ — $12^{\circ}$  C. It was found at first that the pure yeast fermented more slowly, and that the attenuating power was weak, but by a little stronger aëration the difficulty was overcome. In the Australian Brewers' Journal (December 20, 1888, and January 20, 1889) an account is given of the successful application of this system, and it is there stated that no difficulty has been experienced in obtaining the after fermentation; on the other hand, Brown and Morris have informed the author that they have not been successful in isolating a pure yeast with which the impure pitching yeast could with advantage be replaced, and that beers fermented with pure yeast have been very slow and irregular in coming into condition. The author considers that in such a case it might be advantageous to use a second yeast especially for the after fermentation, but thinks that it will be found that such difficulties will not arise when a proper yeast is selected.—A. L. S.

*The Sterilisation of Wort in the Copper.* G. H. Morris.  
Trans. Laboratory Club, 3, 24—42.

THE author states that the present paper owes its existence to the adverse criticism which a statement he made in a former paper (Trans. Lab. Club, 2, 34) has met with. He assumes that the incredulity of those who have questioned the accuracy of this statement is based on the researches of Tyndall on the vitality of putrefactive organisms, and on the experiments on the resistive power of the spores of *Bacillus subtilis* and other organisms which have been made by Buchner, Brefeld, Fitz, Koch, and other investigators. The author considers that the criticisms in question show a total disregard of the conditions prevailing in the copper, and also an incomplete acquaintance with the researches mentioned above. He then shows that on *a priori* grounds we should expect the sterilisation in the copper to be complete.

Numberless germs, which were present on the malt, survive the temperature of the mashing process, and pass with the worts into the copper, where they are subjected to entirely different conditions. In considering these conditions the author examined the influence exerted by—

1. The temperature and duration of boiling.
2. The amount of acid present.
3. The amount of hops added.

In the first place, what are the conditions of temperature and duration of boiling? It is admitted that the temperature in the copper is higher than that of boiling water, but no determinations of the boiling point of wort have hitherto been published. A number of determinations, carefully made with an accurate maximum thermometer, gave temperatures varying from  $214^{\circ}$  to  $216^{\circ}$  F. The temperature naturally depends on the gravity of the wort, on the depth of the copper, and to some extent on the height of the barometer; but  $215^{\circ}$  F. may be considered to be a fair average temperature at the bottom of the copper. The average duration of boiling may be taken at two and half hours from the time at which ebullition commences.

The results of experiments on specific species of micro-organisms as regards temperature are then considered. It is known that the majority of vegetative cells are at once killed at a temperature of  $120^{\circ}$ — $140^{\circ}$  F., and of no species will the cells survive heating to  $212^{\circ}$  F. With endogenous spores the case is, however, different, and it has been abundantly proved that these will, under certain conditions, resist the prolonged action of a temperature of  $212^{\circ}$  F. Tyndall showed that *neutralised* hay-infusion could be boiled for many hours without effecting complete sterilisation, although acid hay infusions yielded much more readily. Brefeld also examined the resistive power of the spores of *Bacillus subtilis* in nutritive solutions; the spores of this organism are perhaps the most resistant of any known, yet it was found that they were still capable of germination

after 15 minutes at  $212^{\circ}$  F., after 60 minutes the number of spores which germinated was much diminished, whilst three hours at  $212^{\circ}$  F. sterilised the liquid. At temperatures higher than the boiling point the action was much more rapid—thus, 15 minutes at  $221^{\circ}$  F. was sufficient to kill all spores. Whilst Fitz found that the spores of *Bacillus butylicus* or *Bacillus amylobacter* could withstand a temperature of  $212^{\circ}$  F. for a time varying from 3 to 20 minutes, depending upon the nutritive solution in which they were heated.

The author considers on the ground of temperature and length of boiling alone there is good reason for assuming that all spores would be killed.

It is known that a wort from good sound malt contains a certain percentage of acid when it leaves the mash-tun, and this is increased when the hops are added to the copper. The following are some determinations of the amount of acid in unhopped and hopped wort:—

	Gravity.	Acid as Lactic Acid.
		Per Cent.
Wort from mash tun .....	1077.5	0.0724
Same wort (hopped) from the copper ...	1090.15	0.1317
Wort from mash tun .....	1078.6	0.0730
Same wort (hopped) from the copper ...	1093.1	0.1338
1st copper wort (hopped) .....	1093.05	0.1450
2nd copper wort (hopped) .....	1051.88	0.0991

With unsound and imperfectly grown malts the percentage would probably be higher. The known action of acid nutritive solutions is then examined in the light of the above determinations of acid in normal wort, and experiments of Pasteur and Tyndall with acid nutritive liquids are quoted to show that the amount of acid present in wort is sufficient to at once kill all spores at a temperature of  $212^{\circ}$  F.

Turning to the examination of the influence of hops, the author states that it is known that hops have a very strong antiseptic power, and the researches of Hayduck have shown that this property resides in one of the hop-resins; but nothing has been done with regard to the comparative value of hops as an aid to sterilisation by boiling. The amount of hops employed in the copper is a very variable amount, and it is almost impossible to give any definite figure on this point. In the experiments about to be described hops at the rate of 2 lb. to the barrel were employed.

The following experiments were carried out in order to prove the question experimentally. They were all conducted with the strictest regard to the requirements of bacteriological research. All vessels and solutions were sterilised in the usual manner, and all transference from vessel to vessel was carried out in such a way as to avoid aerial contamination. Unless otherwise stated, the flasks with the solutions were incubated for at least 21 days at  $80^{\circ}$  F., and a series of at least five flasks were employed for each observation.

*Experiment 1.*—Samples were taken in sterilised flasks, at different stages, of a brew of a light mild ale, with hops in the copper at the rate of 2.22 lb. per barrel, as follows:—

- A. Directly from the mash-tub taps, at a temperature of  $154^{\circ}$  F. (Grav. 1041.1.)
- B. Directly from the copper, 15—30 minutes after the wort broke through. (Grav. 1078.7.)
- C. Directly from the copper, after  $2\frac{1}{2}$ — $2\frac{3}{4}$  hours' boil, and immediately before the coppers were let go. (Grav. 1087.6.)
- D. After the worts had been on the coolers and refrigerators for some hours.

After incubation for 48 hours the samples were examined, with the following results:—

- A. Swarming with long bacilli and rod bacteria.\*
- B. No trace of organisms.
- C. No trace of organisms.
- D. Fermenting briskly, and the sediment showed *Sacch. apiculatus*, *Pastorinus*, and *ellipsoideus* forms, together with rod bacteria, constricted bacteria, and micrococci.

At the end of 10 days:—

- A. As before, the organisms sporulating freely.
- B. No trace of organisms.
- C. No trace of organisms.
- D. Still fermenting briskly, with various forms of yeast and bacteria. The liquid contained 2 per cent. of alcohol by volume.

At the end of 44 days:—

- B. } Still absolutely free from organisms.
- C. }
- D. Still fermenting slightly; 7.04 per cent. of alcohol present.

After 69 days:—

- B. } Still absolutely free from organisms.
- C. }
- D. Still fermenting slightly; 7.79 per cent. of alcohol present.

The experiment was interrupted at this point, and a small quantity of wort, fresh from the mash-tun, was added to each flask of B. and C.; in 48 hours all the flasks contained a large number of bacilli and bacteria.

This experiment shows, firstly, that the wort, as it left the mash-tun, contained a large number of organisms and spores capable of development; secondly, that 15 minutes' boiling in the copper was sufficient to arrest the growth of these organisms; and thirdly, that the wort, before entering the fermenting vessels, contained a vast number of germs capable of development in hopped wort.

That the worts from the coppers were really sterile—i.e., that the germs were actually destroyed, and not simply arrested for a time—was proved by the fact that the addition of a little wort containing germs which have survived the mashing process at once set up a vigorous bacterial growth.

Having regard to the high resistive power of malt-worts, and to the possibility of the germs being simply checked in development and not completely killed by boiling, the author carried out the following series of experiments, in which the resistive power was, on the one hand, reduced to a minimum by the addition of a large volume of sterilised water, and, on the other hand, the wort was largely diluted, and at the same time rendered very favourable to the growth of germs by the addition of sterilised hay-infusion.

*Experiment 2.*—Samples were taken directly from the copper of the first and second coppers of a light mild ale, brewed entirely from barley-malt, after two and a half hours' boil. The former had a gravity of 1086.0, whilst the latter weighed 1031.0, and neither copper was hopped at the rate of more than 2 lb. of hops to the barrel. Small quantities of each wort (at the rate of 1 in 10) were added to series of flasks containing—

- A. Sterilised hay infusion.
- B. Sterilised distilled water.

The latter were again divided into two sets, to each one of which a little of a pure culture of *Sacch. cerevisia* I., Hansen, was added. Seven series of flasks were thus obtained, containing—

- (1.) Hay infusion alone.
- (2.) 1st copper + hay infusion.
- (3.) 1st " + water.
- (4.) 1st " + water + *Sacch. cerevisia* I.
- (5.) 2nd " + hay infusion.
- (6.) 2nd " + water.
- (7.) 2nd " + water + *Sacch. cerevisia* I.

\* In this and the following experiments no definite species of organism are mentioned, but the fields are described in accordance with Pasteur's Plate I. The determination of species by mere microscopical examination is too problematical and uncertain to admit of the use of special names.

These were all forced for three weeks at a temperature of 80° F., and then carefully examined. It was found that in no case had the slightest development of bacteria taken place. The whole of the flasks were absolutely sterile, with, of course, the exception of those forming series 4 and 7, which contained a growth of pure yeast without the slightest admixture of other organisms.

In this experiment the wort was placed under conditions which would favour as much as possible the development of any latent organisms, and the results unmistakably proved that both the 1st and 2nd copper worts were completely sterilised when the samples were taken.

In order to anticipate the possible objection that the conditions prevailing at Burton are particularly favourable to complete sterilisation in the copper, and that different results would be obtained with inferior malts and other waters, samples of average malts from different parts of the country were taken and mashed with distilled water at 158° F. for two hours, in the proportion of 5 grms. of malt to 100 cc. of water. The wort was then filtered through sterilised filters into sterilised flasks, and samples of each mash forced at 80° F.:—

- (1.) Alone and as mashed;
- (2.) After 15 minutes' boil;
- (3.) After 15 minutes' boil with hops at the rate given above (2 lb. per barrel).

The samples were then submitted to microscopical examination, and the results obtained are given in the table at top of next page, which also includes, for comparison, the results obtained when some good and high-dried malts were examined in the same manner. The gravity of the worts varied from 1020 to 1025.

The only malt, the wort from which was not sterilised by 15 minutes' boiling, was that from Wolverhampton, and it was found that this was far the worst when forced before boiling. The wort obtained when the proportions of malt and water mentioned above are employed is of very low gravity, and consequently the acid present is also small, but even in this case the addition of hops brought about complete sterilisation.

The sterilising effect of the amount of acid in normal wort and of hops upon an easily putrescent liquid was then determined. For this purpose hay-infusion was selected, for Tyndall has shown with what difficulty the sterilisation of this liquid is attended. An infusion was prepared by steeping hay in water for some hours; the filtered liquid was divided among three series of flasks, one set of which was boiled for five minutes alone, to the second set lactic acid was added until the infusion contained 0.1 per cent. of acid, and to the third series hops were added at the rate of 2 lb. to the barrel. All the flasks were boiled for five minutes, and then forced at 80° F.

At the end of 48 hours, the liquid in all the flasks containing hay-infusion alone was turbid and swarming with organisms; all the others remained bright, and continued so for 23 days without developing a trace of bacterial or other growth. At the end of that time each flask was inoculated with a few drops of a fresh hay-infusion, with the result that in 48 hours every flask—both with acid and with hops—was swarming with organisms.

This experiment proves that either 0.1 per cent. of lactic acid or the small amount of hops employed is sufficient to sterilise, in five minutes' boiling, a liquid which in the ordinary course it is practically impossible to sterilise by simple boiling; whilst the latter part of the experiment shows that this amount of acid or hops does not prevent or sensibly retard the growth of germs added after boiling—a clear proof that the action in the first place is a true sterilising action, and not merely an arresting action of the growth of the organisms present.

As a further proof of the statement that all malts contain on their surface germs which survive the mashing process, a few examples were given, from among a large number of experiments, of the behaviour of mash-tun worts when

Malt.	Mashed at 158° F.		Mashed at 158° F., and the Filtered Mash afterwards Boiled for 15 Minutes alone.	Mashed at 158° F., and the Filtered Mash afterwards Boiled for 15 Minutes with Hops.	
	After 48 Hours' Forcing.	After 96 Hours' Forcing.			
London.....	Groups of bacilli with long leptothrix forms, and some short active bacteria.	Growth much developed.	No trace of growth after 21 days' forcing.	No trace of growth after 21 days forcing.	
Sheffield.....	Some short bacteria .....	Full of short, constricted bacteria, and some short rod bacteria.	Do. do.	Do. do.	
Wolverhampton .....	Thick film on surface of liquid, sediment showed bacilli, vibrios, medium and short rod bacteria, constricted bacteria and micrococci.	.....	After 7 days a few short bacteria could be detected.	Do. do.	
Hereford .....	Few short bacteria.....	Swarming with bacilli, short rod and constricted bacteria and micrococci.	No trace of growth after 21 days' forcing.	Do. do.	
Lincolnshire, No. I.....	Great many short, constricted bacteria, and some micrococci.	Growth much developed.	Do. do.	Do. do.	
Lincolnshire, No. II.....	Very great many medium and short rod bacteria and some bacilli.	Do. do.	Do. do.	Do. do.	
Burton (mild ale) .....	Trace only bacteria .....	Full of bacilli, short rod and constricted bacteria and micrococci.	Do. do.	Do. do.	
Burton, 1888 (dried at 215°).	Full of bacilli with leptothrix forms, rod and constricted bacteria.	.....	Do. do.	Do. do.	
Burton, 1889 (dried at 215° F.).	Few constricted bacteria.	Great many rod and constricted bacteria and some bacilli.	Do. do.	Do. do.	

forced at 80° F. The samples were all collected directly from the mash-tun in sterilised flasks, and the results well illustrate the universal distribution of germs on malt:—

Malt.	Barrels per Quarter.	Microscopical Examination after 48 Hours.
Pale ale malt .....	2·32	Full of bacilli, short bacteria and micrococci.
	2·26	Swarming with bacilli, leptothrix forms, and rod bacteria.
Mild ale malt.....	2·30	Swarming with bacilli, rod bacteria, and some chaplets.
	2·25	Full of long bacilli and rod bacteria.
High-dried malt (215° F.)	1·85	Swarming with long bacilli, rod bacteria and some micrococci.

Turning to the source of the bacterial and other contamination of beer, if the sterilisation in the copper is so complete, the author mentions several sources of post-copper contamination. In experiment 1, all the samples marked D—that is, those collected as the wort was running from the refrigerator to the fermenting vessels—quickly started into fermentation and developed yeast and bacteria. There are two possible sources of this contamination—namely, from the plant or from the air. The first of these was in this case quite out of the question, and therefore it is necessary to fall back upon the second source.

The author states that he has invariably found that when suitable nutritive media are used, a very large number of organisms is always found in the air of all breweries. This is the case in all localities in the brewery, but naturally the chief source of danger is whilst the wort is on the coolers, and in breweries where the old-fashioned, large, open coolers are used, the contamination at this point is enormous. He then gives the results obtained in two

breweries, designated A and B, in which different systems are adopted, and where the differences are very marked.

In A the cooler is a comparatively deep vessel, with an area of only 70 square yards; in fact, it may be almost more correctly termed a settling-back than a cooler. The last portions of the wort leave this vessel at a temperature of 125°–130° F., running from thence over the refrigerators into the fermenting vessels.

In B the large shallow coolers are employed; these have an area of 426 square yards, and the wort is cooled to the air temperature long before the last portions are off the coolers.

Analyses of the air on both these coolers were made with about the same result as far as regards the number of organisms falling on the square inch; but it is obvious that this number is of less importance in the case of A, with the smaller area and the comparatively high temperature, than it is in the case of B, with the large area and the favourable temperature. Thus, in B, a calculation gave 60,860,000 as the number of organisms falling per hour on the coolers, and if this is multiplied by 4·5, the number of hours the worts are on the coolers, an enormous number is obtained for the total possible contamination at this stage. The author does not for a moment suggest that this number of organisms survive and develop in the wort and pass into the beer, but he has been able to carry this matter a step further, and estimate the number of organisms capable of development in the worts which have passed over the respective coolers. Samples were taken, in sterilised flasks, from the two breweries, when only a few barrels remained up on the coolers, and a measured volume of each wort was mixed with—

(1.) 5 per cent. sterilised gelatin;

(2.) 5 per cent. meat-extract gelatin;

and after three days' culture, the number of colonies developed in each case was determined. From this number the following figures were obtained by calculation:—

A brewery:—

163,548,000 colonies per barrel developed in gelatin alone.

16,354,800 colonies per barrel developed in meat-extract gelatin.

B brewery :—

1,406,512,800 colonies per barrel developed in gelatin alone.

14,065,128,000 to 21,097,692,000 colonies per barrel developed in meat-extract gelatin.

With the gelatin alone, the majority of the colonies consisted of *Saccharomyces* and *Torula*, with many bacteria and moulds. The numbers are simply enormous, but when compared with the number of cells of yeast added at barming, the number is small, and forms but a small percentage of the total cells present, the average number of yeast-cells present per barrel, immediately after pitching, closely approximating to 724,800,000,000 for each pound of barm used.

The author emphasises the great difference there is between the figures for A and B breweries, corresponding to what would be expected from the different systems in vogue in the respective breweries. At both breweries, he says, cleanliness is most rigidly observed in every detail, and the whole contamination must be derived from the air.

Morris considers that in the above there is the germ of a bacteriological test of real value, for the number of colonies obtained will afford some idea of what may be termed the *initial contamination* of the wort, which will depend upon the cleanliness of plant and the purity of the air, which will, again, be dependent upon the state of the brewing premises, the vicinity of "disease-spots" to the coolers, and so on. He states his belief that systematic examination of the worts as they run into the fermenting vessels would be productive of much useful information, and suggests the following simple method for carrying out the determination :—

"Take some wide-mouthed bottles with a capacity of about 12 oz. and with a mouth of about 1½ in. diameter. Plug the mouth with sterilised cotton-wool, and sterilise the whole by heating for 2—3 hours at 150° C. Collect a sample of the wort in a sterilised flask, and add a small measured volume, varying from 0.1 to 0.5 cc., according to the contamination of the wort, to each of a series of the wide-mouth sterilised bottles; a little liquefied and sterile 5 per cent. gelatine is then added, and well mixed with the wort. The mixture is spread over the sides of the bottle, and allowed to solidify evenly. After incubation for a few days the colonies develop, and can then be counted, and, if necessary, removed for examination by means of a bent glass rod."

This method is simple, and has the advantage of telling how many organisms are present in the wort capable of developing in wort. In fact, it gives the *initial contamination* of the wort, and also serves as an index to the state of the plant and atmosphere.

From the foregoing experiments the following three main conclusions are drawn :—

1. That the wort as it leaves the mash-tun always contains germs capable of development; this is the case even with sound, high-dried malt.
2. That these worts are completely sterilised in the copper, and in a much shorter time than it is usual to boil them.
3. That myriads of air-borne organisms are sown in the wort whilst on the coolers, and these can be determined in the wort before it enters the working-squares.—G. H. M.

*Studies on Diastase.* C. J. Lüntner and F. Eckhardt. J. prakt. Chem. 41, 91—96. (See this Journal, 1887, 296, and 1890, 91.)

THE authors treat in the first place of the diastatic ferment of ungerminated barley (and correspondingly of wheat) and question whether it is identical with malt diastase. If this last were so, then they should furnish similar temperature curves in converting a given quantity of starch.

Experiments conducted similarly with soluble starch, however, showed that malt diastase acted best at 50°, but well at 55°, and strongly at 62°; while the most favourable temperature for extract of barley was 45°, although it acted at 50°. This explains why the iodine reaction is longer in

disappearing in the case of barley extract and starch at 50° than in the case of malt diastase, it being the more favourable temperature for the last named.

The authors conclude by commenting on Reyehler's so-called artificial diastase. This name should be reserved for malt diastase with which it is now identified, and not confused by being used for other ferments.—D. A. S.

## PATENTS.

*Improvements in Processes for Rectifying and Distilling Alcohol, and in Apparatus therefor.* E. A. Barbet, Agde, France. Eng. Pat. 4549, March 15, 1889. 8d.

IN an ordinary rectifying plant, consisting of a boiler, a column with trays, a condenser or analyser, and a refrigerator or cooler, the patentee has found that between the liquid which flows back from the analyser on to the trays and the liquid from the refrigerator there is little if any difference, and that the back-flow from the analyser is four or five times greater than the flow from the refrigerator, so that the analyser continually returns a large quantity of the volatile products back into the column. Also that the liquid on the trays is much freer from the volatile impurities than the back-flow, this liquid being at the end of a two hours' flow of as good a quality as the liquid from the refrigerator after eight or ten hours. In order to carry these improvements into effect, he introduces a small supplementary refrigerator and passes through it the liquid from one of the upper trays of the column. Communication between the tray and refrigerator is controlled by a stop-cock. This is closed at the commencement of the distillation until the liquid on the tray is sufficiently pure; to determine this a small testing cock is inserted on the connecting tube. The liquid is tested occasionally during the distillation until the end products begin to appear, when communication with the tray is cut off. During the whole operation the ordinary refrigerator condensing the vapours from the analyser is in operation as usual.

The inventor makes use of a similar device, towards the end of the distillation, to prevent the accumulation of fusel oil in the boiler, having a pipe and stop-cock communicating with one of the lower trays of the column and running liquid off from it as soon as fusel oil accumulates there. Drawings of the apparatus accompany the specification.—A. L. S.

*Improvements in and connected with the Purification of Raw Spirit in order to produce Highly Rectified Spirits of Wine, and Apparatus therefor.* C. Schmitt, Wiesbaden, Germany. Eng. Pat. 5647, April 2, 1889. 8d.

ALTHOUGH petroleum spirit of low boiling point cannot extract fusel oil from alcohol, yet when potassium carbonate solution is first mixed with the alcohol, and the strengths arranged so that the formation of two layers is avoided, then under certain conditions petroleum spirit will extract the whole of the fusel oil. The inventor has made experiments in order to determine the best proportions of the materials, the most successful one being as follows :—300 cc. of a 30 per cent. spirit containing 0.2 per cent. of fusel oil was mixed with 110 cc. of a solution of potassium carbonate (sp. gr. 1.5) and shaken up with 600 cc. of petroleum spirit. On standing the petroleum ether rose to the top and was found to contain the whole of the fusel oil. On a commercial scale the solutions are mixed in the proportions as indicated above in a suitable vessel supplied with stirring gear, the liquid allowed to settle, and the petroleum run off and passed in a finely-divided state first through cold water, (which absorbs all the ethyl alcohol taken up by the petroleum, but very little of the fusel oil), then through water at 50°—60° C., in which all the fusel oil is extracted, and lastly through 50—60 per cent. alcohol, which absorbs all the aromatic bodies, and the purified petroleum is ready to be used over again.

The lower liquid from the mixing vessel consists of the purified alcohol, water and potassium carbonate. The addition of more solid carbonate causes it to separate into two layers; the lower one contains very little alcohol, and

on concentration is ready for use again; the upper layer consists of 94 per cent. ethyl alcohol containing a little carbonate; this latter may be removed by adding either sulphuric acid or gypsum, when the potassium sulphate formed, being insoluble in alcohol, is precipitated. The clear solution consists of almost pure strong ethyl alcohol. A drawing accompanies the specification.—A. L. S.

*Improvements in Yeast-Cakes.* H. H. Lake, London. From J. Barnes, New York, U.S.A. Eng. Pat. 17,523, November 4, 1889. 6d.

A YEAST-CAKE is prepared by subjecting a mixture of yeast and prepared corn meal to great pressure in a mould having a rounded form with no sharp angles. The effect of the pressure is to force the finer granules of corn meal to the surface and thus form a protective coating. A further protective coating is produced by dipping the cake in a solution of cane sugar and drying. A drawing accompanies the specification.—A. L. S.

*Improved Process for Treating Beer and making it keep permanently in Good Condition.* J. F. H. Grouwald and E. H. C. Ochlmann, Berlin, Germany. Eng. Pat. 19,376, December 3, 1889. 4d.

THE inventors propose to avoid the difficulties of the pasteurising process by distilling the beer; the beer residue is then fined and remixed with the distilled liquid. It is afterwards aerated with carbonic acid gas and bottled or filled into barrels.—A. L. S.

*A New Method of Killing Microbe-like Minute Living Organisms, such as Yeast Cells, Bacilli, and the like, contained in Wines, Alcoholic and other similar Liquors.* W. Spilker, Berlin, Germany, H. Bendel, San Francisco, U.S.A., M. Hahn and C. Löwe, Berlin, Germany. Eng. Pat. 19,996, December 12, 1889. 4d.

THE inventors claim that by circulating wine and other similar alcoholic liquids in tubes in an electric or magnetic field, and in the direction of the lines of force from one pole to another, all living organisms are killed, and that the alcoholic liquor is matured in the same way as by lengthened storage. In the case of spirits a maturing effect is produced equal to that resulting from several years' storage.

For treating 1,000 litres of wine, a current energy of about 7,000 volt ampere hours has generally been used; that is to say, in 12 hours about one cubic metre of wine would be treated by 1 horse-power.—A. L. S.

## XVIII.—CHEMISTRY OF FOODS, SANITARY CHEMISTRY, AND DISINFECTANTS.

### (A).—CHEMISTRY OF FOOD.

#### PATENTS.

*A New or Improved Mode and Means of Estimating the Acidity in Milk, Cream, Whey, and Curd in the Process of making Butter or Cheese.* J. Gray, Stranraer, and T. Clement, jun., Pollokshields. Eng. Pat. 4,966, March 21, 1889. 8d.

A CYLINDRICAL glass vessel, provided with graduated scale, and containing the milk with a little phenolphthalein or other chemical indicator, is connected with a bottle containing solution of an alkali. The alkali is gradually run in, during agitation (the connection from the alkali bottle being at the bottom of the milk cylinder), until change of colour denotes that the acid has been neutralised. The quantity of alkali added is ascertained by reading the scale before

and after the experiment, and from this the acidity is calculated. On the graduated scale, at different levels of acidity, markings may be made, naming the stages at which the "rennet" may be added and other operations performed.—T. L. B.

*Improvements in the Making of Butter for the Purpose of Preserving the same.* F. McIntyre, Glasgow. Eng. Pat. 5052, March 23, 1889. 6d.

THE presence of germ life during the whole process of making butter is prevented. In the first place, any germs which may be contained in the milk are destroyed by the application of heat. The churn is so fitted that no germs whatever can enter, and before use it is steamed. An operating box, for washing and packing the butter, is fitted with arm-holes, provided with folds or pucks, so that entrance of germs is prevented during the operation. All the apparatus is steamed before use, and the salt is heated, either in the solid state or in aqueous solution. Water used for washing is likewise previously boiled. Instead of having separate air-filtering tubes on separator, churn, and room in which the butter is worked and packed, one room may be used enclosing the whole, the air entering the room being drawn through a tortuous pipe, part of which is kept at a red heat.—T. L. B.

*Improvements relating to the Preservation of Shell-fish.* A. A. Freeman, Philadelphia, U.S.A. Eng. Pat. 14,670, September 17, 1889. 6d.

UNDER ordinary conditions oysters and other shell-fish, after being kept some time, open their shells and the juices escape; the fish thereby loses its flavour, and in a short time becomes unfit for use. To prevent such opening of the shells the patentee binds them round with wire while fresh.—T. L. B.

*A New or Improved Method for Sweetening Margarine, Butter, and other Oils and Fats.* P. Heinz, Frankfurt-on-the-Main, Germany. Eng. Pat. 19,158, November 28, 1889. 4d.

SACCHARINE is dissolved in the fat; the amount dissolved, and therefore the resulting sweetness, depending on the temperature to which the fat is raised during the process of solution.—W. L. C.

### (B).—SANITARY CHEMISTRY.

#### PATENTS.

*Improvements in Water Purifying Apparatus.* H. Carrol, South Croydon. Eng. Pat. 9569, June 8, 1889. 8d.

THE purification of the water is effected in a lower vessel, having side receptacles for deposit, formed by inclined curved plates, which extend upward to near the underside of a baffle-plate extending across the vessel so as to leave a space at each side above the receptacles, and between the plate and sides of the lower vessel. Above this vessel are two tanks, one for receiving the reagent from a tank above, and the other for the water to be purified. The reagent and water flow down a pipe into the lower vessel, and rising upwards pass between the tops of the receptacles and underside and edges of the baffle-plate and the sides of the lower vessel, and upwards (through a filtering medium, if desired) to the outlet pipe. There are two sheets of drawings and three claims.—E. S.

*New or Improved Means or Apparatus for Softening and Filtering Water or other Liquids under Pressure.* "The Water Softening Company, Limited," E. Easton, Westminster, and H. Carrol, South Croydon. Eng. Pat. 12,973, August 16, 1889. 8d.

A CLOSED vessel, preferably of a conical form, and having its smaller end downwards, contains the lime or other reagent. This vessel stands upon a second, into which the



water or liquid to be treated is admitted under pressure. This stands upon a third vessel, the lower part of which is separated from the upper part by a perforated plate on which is placed filtering material, covered by another plate. The water or liquid under pressure enters the second vessel and the lower end of the conical vessel at the same time, and after mixing with the reagent flows out by a pipe at the top which joins the main supply pipe, the mixed liquid together entering the second vessel from which it overflows through a pipe to the bottom of the third vessel, and after passing through the filtering materials flows out for use or storage. The conical vessel is supplied with the reagent through a funnel regulated by a valve.—E. S.

## XIX.—PAPER, PASTEBOARD, Etc.

*The Process of Engine-sizing.* E. Muth. *Dingl. Polyt. J.* 275, 29–40 and 71–80.

This paper deals with the rosin-sizing process as usually practised, giving a careful survey of the principles underlying it, with deductions therefrom in the form of such practical details as are calculated to lead to the best results. In the beating process the object should be to draw rather than cut the fibres, with the result of securing the maximum of felting with interspaces of minimum size. The alternative method produces a web parting very rapidly with its water on the machine, with proportionate loss of the suspended particles of sizing compounds. These particles are held by a fine well-felted web as in a filter, and the final closing of the interspaces with and by the sizing matters is effected by the pressure of the couch-roll, to the weight of which special attention must be paid. In the preparation of the resin-soap the object should be to secure in the final product the maximum proportion of emulso-resin (Milchharz) i.e., uncombined resin, dissolved, in the soda-resin compound. For this the maximum temperature of the "boil" should be 70°, and the water a minimum. (So much importance is attached by some manufacturers to this constituent of the sizing mixture that the presence of a sufficient quantity is ensured by the solution of an additional proportion in the finished resin-soap.) The completion of the boiling is recognised by dipping a spatula from time to time. On drawing out, the cooling mass should break short off without forming long threads. The time of boiling, with a charge of 6 cwt., should not exceed four hours. The complete separation of the resin-soap requires from 2–3 days' standing. The colouring matters of the rosin are for the most part retained in solution. For their complete removal, with the lye residue, the mass may be treated with successive quantities of salt solution. The quantities of rosin, soda (Solvay) and water are variously formulated by different authorities. The author's table comprises numbers within the following limits: rosin, 100; soda, 18–27; water, 65–2,000. The calculated quantity of carbonate for complete conversion into the soda compound is 17 per cent.; using 18.5 per cent. soda the author found 15 per cent. of emulso-resin in the product. These proportions may be considered normal. In re-dissolving the compound for use, the optimum of dilution is variously stated at 25–70 parts of water to 1 part of total resin.

The effective sizing of the web requires first the absorption of the materials by the individual fibres, and secondly the filling of the interspaces which are, of course, of variable dimensions. The necessary gradations of fineness of the precipitated matters, emulso-resin and resin-alumina compounds—are perhaps sufficiently secured by the usual mode of adding the sizing solution and the aluminium sulphate. Greater efficiency may be attained by adjusting the conditions in view of these requirements. It is also recommended to take into account, in using hard waters, the objection to the precipitation of lime and magnesia compounds of the resin-acids, since by decomposition in the paper the effects of imperfect sizing may be produced.

This difficulty is easily met by treating the water to be used in the beater with a calculated quantity of aluminium sulphate. The utility of using gelatin in the engine-sizing process is very questionable, the amount retained by the paper being necessarily small. The albuminates, on the other hand, are highly recommended, being precipitated by alumina salts in a form especially favourable to the effectual distribution of the albumen, and to the qualities of the paper made with this addition, more particularly in point of elasticity and surface. It is recommended to add the aluminate (ammonium) in concentrated solution (30–40 per cent.) at the beginning of the beating. In regard to the completion of the process on the machine the author directs special attention to the shaking (felting), and the temperature of the drying cylinders, more especially the first, in order that the resin and resin compounds may be brought into the right condition of fusion, ensuring a homogeneous compacting of fibre and sizing materials.

A process involving fine adjustment of so many factors, chemical and mechanical, cannot be laid down on hard and fast lines universally applicable, but requires special study for every set of circumstances, i.e., for each mill. As an illustration of extreme variations between efficient and inefficient consumption of sizing materials, the author cites two cases from his experience, in one of which the percentage of resin used was  $2\frac{1}{2}$ , in the other 8 per cent. on the paper. Adding to these numbers the same weights of alumina salt necessary for precipitation, the difference of consumption of material in the two cases is seen to be very great.

In the summary of points which concludes the article the author emphasises the three more important conditions of efficient sizing, viz.:—

- (1.) The composition of the pulp (fibre) and its condition when beaten.
- (2.) The physical properties of the precipitated resin-alumina mixture.
- (3.) The communication to the web of a satisfactorily high temperature on the drying cylinders.—C. F. C.

*Imitation Japanese Paper.* W. Herzberg. *Mitt. k. techn. Versuchs. Anstalt, Berlin*, 1889, 152–153.

THE author gives the results of an investigation of a paper recently introduced in obvious imitation of the genuine Tokyo paper, offered at one-fourth the price, 1.45 M. per kilo., and stated to be of a quality fitting it for such uses as for official documents, share coupons, &c. The following are the results of mechanical tests: *Breaking lengths* (a) 7.24 k.m., (b) 4.50; mean 5.87. *Elasticity* (extension per cent.), (a) 1.6, (b) 5.1; mean 3.4. Sheets of the paper were exposed freely to the air and light (hung in a window) for three months and again tested. A diminution of 9 per cent. in breaking length and 41.2 per cent. in elasticity was noted. This result is noteworthy, the more so as the paper consisted of a pure wood cellulose, apparently sulphite cellulose. It contained only 3 per cent. of ash constituents, and was engine-sized (rosin and starch). The author promises further tests of the paper after intervals of exposure.—C. F. C.

## PATENTS.

*A New or Improved Wood Felt suitable for Surgical Bandages and other Purposes, and a Process for Producing the same.* G. Walcher, Stuttgart, Germany. Eng. Pat. 16,546, November 14, 1888. 4d.

THE wood fibres are ground up into a paste with spirit or other volatile liquid. The spirit is drawn off, the felt dried by heat, and then compressed between rollers to render it pliant.—E. J. B.

*Improved Method of and Means for Protecting Apparatus used in the Manufacture of Sulphite Cellulose.* J. F. Salomon, Berlin, and H. Brünger, Liegnitz, Germany. Eng. Pat. 18,003, December 10, 1888. 8d.

THIS process relates to the formation on the insides of boilers of a crust or scale which will resist the action of the acid solutions (bisulphites) used in preparing wood pulp. The scale is formed by heating the boiler, preferably by means of hot air passed into an outer casing, while it is filled with a saturated solution of calcium sulphate.

—E. J. B.

*Improvements in Treating Vegetable and Animal Fibre and Textile Fabrics whereby the same are Bleached, Strengthened, Prepared for Dyeing, and Improved in Appearance and otherwise.* M. Ziegler, London. Eng. Pat. 84, January 2, 1889. 6d.

THE fibre is cleansed by soaking it in a "diluted solution of sulphuric acid neutralised by ammonia." Gelatin is dissolved in dilute sulphuric acid, to the solution glycerin and ammonia are added. To this is added oleic acid previously saponified by bicarbonate of potash. The fibre is boiled in this solution for several hours and dried. When dry it is treated in a solution made by boiling oxide of zinc with permanganate of potash. When thoroughly combined, bisulphite of soda is added, then a small quantity of neutral gum or shellac. The fibre is finally treated with dilute sulphuric acid, washed and dried.—E. J. B.

*Improvements in the Manufacture of Parchment Fabric.* L. D. Dobler, Paris, France. Eng. Pat. 1767, January 31, 1889. 4d.

ANY suitable fabric is covered with a layer of paper or other similar material. It may be applied to one or both sides, and is caused to adhere by a size which is insoluble in water such as that described in Eng. Pat. 9949, of 1887 (this Journal, 1886, 516).—E. J. B.

*Improvements in Apparatus for the Manufacture of Paper.* D. M. Watson, Glasgow, and T. T. M. Lumsden, Edinburgh. Eng. Pat. 2439, February 12, 1889. 6d.

THE improvement consists in a new form of "Deckle-shifting" apparatus, whereby the width of the web of paper can be altered at will. Descriptive drawings accompany the specification.—E. J. B.

*An Improved Paper.* G. Newbery, London. Eng. Pat. 4310, March 12, 1889. 4d.

PAPER is coated with a mixture of 9 lb. of moist pipe-clay, 2 oz. of tallow, 14 lb. of size, 1½ lb. of black-lead, or ¾ lb. of oxide of manganese. Such paper, when slightly pressed with a hard point, is marked as with lead pencil.

—E. J. B.

*Improvements in the Preparation of Wood Pulp for the Manufacture of Paper, Cloth, Cordage, or any other Textile Fabric.* F. J. Cheesbrough, Liverpool. From H. and W. Pataky, Berlin, Germany. Eng. Pat. 9694, June 12, 1889. 4d.

GREEN wood is crushed between rollers, and at the same time subjected to the action of a stream of water whereby a number of soluble matters are removed, and the subsequent treatment with alkali simplified, a considerable saving being effected. A small quantity of sulphite of soda may be added.—E. J. B.

*A New or Improved Composition for Treating Paper and similar Material to render Writings or other Markings Indelible thereon.* D. C. Simpson, Edinburgh. Eng. Pat. 16,342, October 17, 1889. 4d.

PAPER is treated with a solution made by dissolving 1 lb. of lead acetate, ¼ lb. of potassium chlorate, and 1 oz. of copper acetate, in 1 gallon of water.—E. J. B.

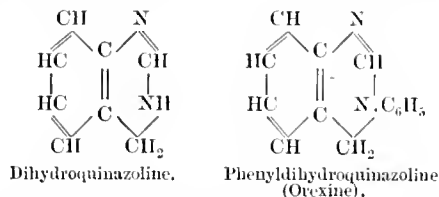
*Improvements in Edge Runners or Mills for Reducing Paper-making Materials or other Substances.* A. E. Ried, Sideup. From A. Abadie, Angoulême, France. Eng. Pat. 18,674, November 21, 1889. 6d.

THE improvement consists in making the bed of small pieces of iron, preferably of a rectangular shape, interspaced with a softer material, such as lead or wood, the surface of the latter being slightly lower than that of the former. In this way the runners are prevented from slipping and the material is more rapidly reduced. There is also an arrangement for raising or lowering the scrapers.—E. J. B.

## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

*Orexine (Phenyldihydroquinazoline).* Pharm. J. 20, 709—710.

THIS is one of a series of compounds recently synthetically prepared by Paul and Busch (Ber. 22, 2683). It is described as a derivative of quinazoline, a body represented by a structural formula differing from that of quinoline in having two CH groups of a naphthalene ring replaced by N instead of one. In dihydroquinazoline there is an imido-group the hydrogen of which is replaceable by an alkyl group, and it is that compound in which the substitution is effected by a phenyl group that is now put forwards under the name "orexine."



THIS compound is manufactured by heating the sodium compound of formanilide with the corresponding quantity of *o*-nitrobenzylchloride, and after the purification of the resulting *o*-nitrobenzyl formanilide, reducing it to phenyl-dihydroquinazoline by means of zinc dust and acetic acid. The hydrochloride of this base is produced in needles containing two equivalents of water of crystallisation, gradually given off in an exsiccator the crystals becoming efflorescent. The melting point of the hydrated crystals is 80°, that of the anhydrous, 221° C. When laid upon the tongue the compound tastes slightly bitter and leaves an intense burning sensation; it also powerfully irritates the mucous membrane of the nose. In ether it is insoluble, but it is readily soluble in hot water and in alcohol, and for this reason the hydrochloride is preferable in dispensing than the free base, which is almost insoluble in water. From an aqueous solution of the hydrochloride the base is separated by alkalis as an oily precipitate which eventually crystallises. Orexine hydrochloride is a substance which induces appetite and stimulates digestion. It is neither an antipyretic nor an analgesic.

In the case of healthy persons the appetite is stated to increase immediately after the first dose, but with most patients the improvement is only manifested after some days.

The formula recommended for administration is 2 grms. of orexine hydrochloride made up with extract of gentian

and althaea powder into 20 pills gelatin-coated, three to five of which are to be taken once or twice daily with a large glass-full of meat broth, a considerable quantity of liquid being required on account of the pungent properties of the compound.

*The Solubility of Quinine and Caffeine.* Pharm. J. 20, 710—711.

ANTIPYRINE increases the solubility of quinine (Pharm. J. 19, 861). It is now stated that it exercises a similar influence on caffeine. Crinon reports that it suffices to add to caffeine a slightly superior weight of antipyrine to render it soluble in the cold, and that if heat be employed 50 centigrams of caffeine may be made to dissolve in 10 cc. of water if 75 centigrams be added, the solution remaining clear on cooling.

*Report on New Drugs and Fine Chemicals.* Merck's Bull. 3, 1890, 1—8.

THE MYDRIATIC ALKALOIDS OF THE SOLANACEÆ—*cont.* (This Journal, 1890, 212.)

*Homatropine* (Hydroxytoluyltropine),  $C_{16}H_{21}NO_3$ —Ladenburg, in the course of his researches on the synthetic production of alkaloids, obtained a series of artificial alkaloids by the action of hydrochloric acid on certain salts of tropine, itself a fractional derivative of atropine. To the group of alkaloids thus obtained he applied the generic term "Tropines." Thus, by the action of hydrochloric acid amongst other products, the following were obtained:—

From tropine tropate—

*Atropine*, in every respect identical with the natural alkaloid.

From tropine salicylate—

*Salicyl-tropine*, a weak poison, devoid of action on the pupil.

From tropine phthalate—

*Phthalo-tropine*.

From tropine mandelate—

*Homatropine*,  $C_{16}H_{21}NO_3$ , *Hydroxytoluyltropine*.

Of all the foregoing, the last named is by far the most interesting, its peculiar action on the eye rendering it of therapeutic value. Experience proves in fact that it will be very serviceable in ophthalmology.

*Homatropine* dilates the pupil just as strongly as atropine does, but it possesses less of the paralysing effect of that mydriatic, and what is of the greatest importance, the recovery from the *mydriasis* is far more rapid than when atropine has been used (Völkers and Goetz, Inaug. Dissert. Kiel, 1880). Atropine dilations, even with very weak solutions, continue for several days up to a week, whereas a homatropine dilation commences to diminish after a few hours' duration, and in the course of 24 hours, as a rule, every trace of it will have disappeared. Moreover, homatropine, unlike atropine, is a very weak poison, and does not, as atropine often does, superinduce cutaneous eruptions. It is likely to be very useful in examinations of the *Fundus oculi*, &c., since it permits of the eye being used again within a very brief period.

*Homatropine* in the pure state forms clear and regular crystals, the form of which has never yet been determined. Its salts with hydrochloric, hydrobromic, and sulphuric acid, are white, and crystallise well.

*Homatropine hydrobromide*.—This salt forms white, crystalline plates, and is soluble in 10 parts of water. The solution is quite permanent and the salt is not hygroscopic. The more recent physiological experiments were principally made with this salt (Schweinitz and Hare, Amer. Med. News, 1887, Dec. 731; Jackson, Amer. Med. News, July 1888). After instillations of large doses of homatropine, its bitter taste becomes perceptible, but not the dryness of the pharynx which follows the use of atropine.

*Aconitine*.—Notoriously the various brands of aconitine in the market exhibit very marked differences in the strength of physiological action, and the observance and expression

of some rigorous distinction between the different grades has become a matter of serious importance. Dr. Van Reuterghem (*Neederl. Tijdschr. voor Geneesk.* 1889, 9) has to this end subjected several aconitines of various origin to a series of precise therapeutic tests, the experiments being made upon his own person.

In order to realise certain definite primary symptoms he had to employ certain amounts of the different brands experimented with. These quantities constituted what he termed the "sufficient dose." The effect indicated was found to be caused by—

From 0.25 to 0.5 mgrm. ( $\frac{1}{250}$ th to  $\frac{1}{125}$ th grain) of the two crystallised aconitines of Merck or Duquesnel.

From 10 to 15 mgrms. ( $\frac{1}{10}$  to  $\frac{1}{6}$  grain) of the amorphous aconitines of Merck and Chantenaud.

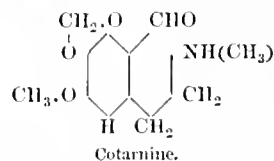
From 200 to 300 mgrms. (3 to  $4\frac{1}{2}$  grains) of Friedländer's aconitine.

This shows a range of comparative degrees of potency from 1,200 down to 1, i.e., a similar effect is secured from the strongest preparation by a dose only  $\frac{1}{1200}$ th to  $\frac{1}{800}$ th the size of those required of the weakest preparation tested under the name of "Aconitine."

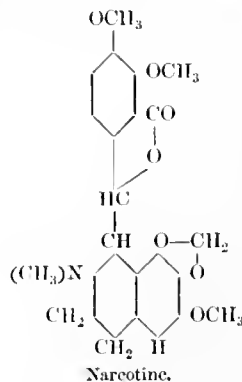
*Jambul* is a powder or fluid extract of the seed or bark of the *Syzygium jambolatum*, a tall tree growing in the American and Asiatic tropics, and belonging to the *Eugeniaceæ*, a branch of the *Myrtaceæ*. It is a specific in cases of *Diabetes mellitus*. That *Jambul* has a retarding action on the diastatic fermentation of starch was first observed by Lascelles Scott. W. Elborne and W. H. Morse succeeded in procuring a reduction of the diuresis and of the sugar in the urine by administering small quantities of the powdered fruit. Gräser, of Bonn (Centralblatt f. klin. Med. 1889, No. 28), has confirmed the preceding by the use of *Jambul* extract, and he has moreover proved that the drug is harmless in the animal economy.

*Researches on Narcotine.* W. Roser. *Annalen*, 254, 334—368.

A RÉSUMÉ of researches bearing on the constitution of cotarnine and narcotine. A closely-reasoned account of the evidence, as regards the former, afforded by these, is summed up in the annexed constitutional formula:—



*Narcotine* containing the residues of hydrocotarnine and opianic acid is formulated therefore as subjoined:—



It is closely related to another opium alkaloid, viz., papaverine, both being derivatives of a benzyloquinoline.

—C. F. C.

*Denaturing Alcohol by Means of Wood Spirit and Pyridine.* A. W. Hofmann, G. Krämer, and L. Loewenherz. Chem. Ind. 12, 521—525.

THIS article treats of the present aspects of the method of denaturing alcohol, after two years' experience of its employment in the German Empire. The complaints at first raised against the use of pyridinised spirit for domestic purposes have now nearly ceased, especially since the quantity to be added has been reduced from 1 per cent. to 0.5 per cent. of pyridine. In a few cases, such as those in which varnish or polishes are manipulated in confined or badly ventilated places, it may still be advisable to allow the spirit to be treated with 5 per cent. of wood spirit only, whilst the method of denaturing at present in force throughout the Empire consists in the addition of 2 per cent. of wood spirit and 0.5 per cent. of pyridine to the alcohol. The greatest difficulty experienced during the introduction of the new method arose in the increased prices of pyridine consequent on the sudden demand for large quantities, but the impetus thus given to its manufacture and import from other countries, especially from England, has eventually brought down the price below that existing before the introduction of the measure, and the values of wood spirit have fluctuated in a similar way, but to a lesser extent. Thus the values adopted by the Commission on Denaturing were 300 M. for pyridine and 120 M. for wood spirit (per 100 kilos.), but these rose to 400 M. and 140 M. respectively, whilst at the present time the prices are under 90 M. in each case, and the mixture itself may now be bought for 80 M. per 100 kilos. In numerous factories, where the use of alcohol with wood spirit was adhered to, the pyridinised alcohol has at length been adopted, its use being found preferable as being free from the control exacted by the excise officials when wood spirit only is the denaturing agent. The following table shows the favour extended by consumers to the pyridine as against the wood spirit method:—

	Quantities of Pure Alcohol denatured.	
	With the Official Mixture.	With 5 per Cent. Wood Spirit.
	HL.	HL.
October to December 1887 .....	37,257	13,785
January to March 1888 .....	28,146	7,847
April to June 1888 .....	31,928	7,681
July to September 1888 .....	37,342	5,241

Pyridinised alcohol, as used in households, gives out no smell while burning in a lamp, but, on blowing out the flame, an unpleasant smell is experienced, which is, however, less disagreeable than the smell of a recently-extinguished petroleum flame, to which consumers generally have easily become accustomed. Alleged harmful results from inhaling the vapour of pyridinised alcohol have been shown to be groundless. In order to cover the objectionable smell of the pyridine, it has been permitted, in some cases, to add a certain quantity of lavender or rosemary oil. Brief reference is made to other substances which have been proposed for the purpose of denaturing alcohol. Amber oil, caoutchouc oil and camphor oil, as well as naphthalene and oil of turpentine, are inadmissible, as they are readily removed by dilution with water and treatment with animal charcoal. Shellac does not render the spirit sufficiently undrinkable, whilst mercaptan or bone oil are much more objectionable than pyridine. The use of wood spirit would not effect the desired purpose in less proportion than 10 per cent., and the quantity then required would be too far in excess of the possible production, and even then the excise would not be fully protected, as experience in England has shown. Experiments made with suitable petroleum fractions showed that it could be removed by dilution with water and treatment with charcoal. Bitter substances are open to the same objection, and they

interfere with the proper burning of the spirit. An additional objection to the use of wood spirit is that acetone and allyl compounds, which are its most important denaturing constituents, prevent its application in many technical industries. On the whole there seems to be no more suitable substances for denaturing alcohol than those which have been selected, the same method having been adopted in Austro-Hungary and Switzerland; at the same time the authors hold the view that a more satisfactory solution of the difficult problem may yet be found. They advocate the establishment of a central institution to which all questions respecting denaturing and other excise matters could be referred, such as exists in England.—G. H. B.

*Synthesis of Aconitic Acid from Acetylenedicarboxylic Acid.* J. M. Lovén. Ber. 22, 3053—3056.

IN preparing acetylenedicarboxylic acid from dibromosuccinic acid and alcoholic potash, the author obtained two acids, viz., oxalic and aconitic acid. This is explained by the presence of an excess of alkali, which, reacting upon the acetylenedicarboxylic acid decomposes it into the two acids above mentioned.

The author supposes that the acetylenedicarboxylic acid takes up the elements of water to form oxalacetic acid, which condenses with a molecule of unchanged acetylenedicarboxylic acid which then decomposes with potash.

—J. B. C.

*Belladonnine.* E. Dürkopff. Ber. 22, 3183—3184.

TO obtain belladonnine free from hydrocarbons, hyoscyne, tropine, &c., the crude material is digested in acid solution with ether and chloroform to dissolve out hydrocarbons. The atropine is converted into tropine and tropic acid, whilst hyoscyne dissolves, and the belladonnine remains unaltered, the dissolved hyoscyne is isolated as gold double salt. The occurrence of hyoscyne in crude belladonnine explains the presence of pseudotropine in its decomposition products. From the latter a base was isolated by Ladenburg and Roth, and also Merling, which, however, they were unable to identify with pseudotropine, but which according to the author is identical.—J. B. C.

*Researches on Crystallised Digitalin.* Arnand. Compt. Rend. 109, 679—681.

THE author finds that tanghinin, one of the active principles of the Madagascar tanghin (this Journal, 1889, 1008) is very similar in its properties to crystallised digitalin. Both substances are non-nitrogenous, both are practically insoluble in water, but are soluble in all proportions in chloroform, and equally soluble in moderately concentrated warm alcohol. They do not belong to the class of glucosides, for they do not yield glucose or any other form of sugar when treated with boiling dilute acids; under such treatment they simply resinify. They are also similar in their physiological behaviour; both are violent cardiac poisons, and both occur in nature in conjunction with true glucosides which have a powerful action upon the organism.

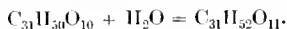
It has been stated by Schmiedeberg that digitalin prepared according to Nativelle's process is a mixture of several crystalline substances. The author contradicts this assertion, and the object of his communication is to establish the fact that digitalin so prepared is a perfectly definite chemical product. The material first used in his experiments was prepared in the author's laboratory by Nativelle's process from 20 kilos. of Vosges digitalis, purified by washing with cold alcohol. The pure digitalin forms thin brilliant white flakes melting at 243°. 100 grms. of absolute alcohol at 14° dissolve 0.650 grm. Contrary to Schmiedeberg's assertion, it is soluble in boiling benzene. The method of examination adopted was that of "fractional solution" by successive washings with absolute alcohol at 14°. The solubility was not found to vary, nor was there any sensible variation in the melting points of the residues,

or of the successive fractions when determined after evaporation and crystallisation. Similar results were obtained with a sample of digitalin prepared and furnished by Adrian.—D. E. J.

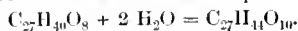
*Digitalin and Tanghinin.* Arnaud. *Compt. Rend.* **109**, 701—703.

By the action of baryta on digitalin in presence of water in a sealed tube heated to 180° for several hours, a crystalline body is formed insoluble in water and containing barium in combination. The compound after purification melts at 305°—310°. The analysis of this compound leads to the formula  $C_{31}H_{50}O_{10}$ . The action of baryta appears to be one of hydrolysis, as the original digitalin cannot be reproduced by eliminating the barium from the compound.

It may be formulated as follows:—



The action of baryta on tanghinin is similar; but an amorphous barium compound is formed and it is probable that two molecules of water are taken up:—



—J. B. C.

*Comparative Activity of Different Digitalins.* G. Bardet. *Compt. Rend.* **109**, 755—756.

CRYSTALLISED digitalin and amorphous digitalin prepared according to the formulae of the French Pharmacopœia are completely soluble in chloroform, they have identical activity and are always comparable in their effects. The German digitoxin is not completely soluble and its effect is two to three times weaker in the different preparations used, than the official digitalin. The French digitalin and the German digitalin are both soluble in water and insoluble in chloroform, though they are not definite products. They are similar in action; but their activity is 20 to 35 times less than that of the digitalin of the Pharmacopœia or that which is soluble in chloroform.—J. B. C.

*Action of Related Chemical Compounds on Animals.* Woleott Gibbs and H. A. Hare. *Amer. Chem. J.* **11**, 435—448.

THE authors desire to ascertain whether general laws exist by which the action of chemical compounds on the system may be predicted. They have set themselves to examine, therefore, and compare the actions of compounds related to each other, such as, for instance, nitrobenzene and the nitrotoluenes, ortho-, meta-, and para-compounds, the nitrophenols, hydrazines. Thus far they have obtained the following results from experiments made on dogs:—

*O-, m-, and p-nitrophenol.*—Of *o*-nitrophenol 0.1 gm. to each kilo. in the weight of the dog produces death; of *m*-nitrophenol slightly less than this suffices; of the para-compound 0.01 gm. per kilo. only is needed to kill. The substances were rapidly injected into the jugular vein. The toxicity is thus in the following order: para, most poisonous, then meta, and least poisonous the ortho compound. They all produce death by paralysing the heart, and not by a respiratory action. On bodily heat they seem to have very little effect, although their true antipyretic value is not certainly determined.

*O-, m-, and p-nitraniline* all slow the pulse. Of the ortho-compound a dose of 0.3 gm. per kilo. of the animal produces death when injected into the jugular vein; methaemoglobin is produced in the blood. When given by the stomach violent sneezing comes on and lasts for about 40 minutes. The meta-compound shows all the symptoms of aniline poisoning. Para-nitraniline is the most poisonous member of the group, 0.04 gm. per kilo. of animal producing death when injected into the jugular vein.

*Amido-benzoic acids* are without effect on the system, whether given by the stomach or intravenously.

*Nitro-benzoic acids* are likewise without effect.—T. L. B.

*Indian Grass Oils.* F. D. Dodge. *Amer. Chem. J.* **11**, 456—469.

THERE are found in commerce at least five volatile oils which appear to be obtained from various tropical grasses of the genus *Andropogon*. They are: oils of citronella, lemon grass, Indian or Turkish geranium, ginger grass, and vetiver. The author has made a detailed examination of citronella oil. Three samples of the oil were obtained; two of these were found to have been adulterated with kerosene, and were therefore discarded; on the remaining sample the work of this communication was performed. The oil was of a light greenish-yellow colour; its density at 16° was 0.8770, at 26° 5', 0.8750; on distillation, all, with the exception of about 10 per cent. of thick oily residue, passed over between 200° and 240°. A saturated solution of sodium bisulphite gives a solid mass when shaken with the oil, phenylhydrazine combines with it with evolution of heat, and it reduces ammoniacal silver solution. In order to isolate the aldehyde, the bisulphite compound was made, filtered, pressed, washed with ether, and air-dried. The dry mass was then mixed with crystallised sodium carbonate, and distilled with steam. The steam-distilled aldehyde, after drying several days over calcium chloride, forms a colourless oil of not unpleasant odour and sharp acid taste. Its specific gravity at 25° was found to be 0.8509; it boils at 202°—207°, leaving a resinous residue. Results of analysis agreed well with the formula  $C_{10}H_{18}O$ , so that the aldehyde is isomeric with borneol and geraniol. The author terms it citronellaldehyde. It is dextro-rotatory; a column two decimetres long produces a deviation of about 7° for sodium light. With bromine it forms a dibromide, which is decomposed on heating to 120°, and on reduction it gives the alcohol boiling at 225°—230°. Ordinary oxidising agents appear to split up the molecule, giving a mixture of acids. By the action of phosphorus pentoxide on the aldehyde, a terpene boiling at 175° is obtained. Discussion of the constitution of the aldehyde is naturally premature.

As regards the other oils, Gladstone noted that oil of lemon grass resembled citronella oil very closely, but no extensive work has been published on the subject. Flückiger has remarked that it behaves with bisulphite solution exactly as citronella oil does. Oil of Turkish geranium has been examined by Jacobsen (*Ann.* **157**, 232), who found it to contain a monatomic alcohol, geraniol,  $C_{10}H_{18}O$ . Oil of ginger grass has not been investigated, and oil of vetiver, which appears to be different from all the other grass oils, is somewhat inconvenient to work on by reason of its costliness.—T. L. B.

## PATENTS.

*A New or Improved Manufacture of Para-Amido-Phenol Ethers.* B. Willecox, London. From "The Farbenfabriken vormals Fr. Bayer and Co.," Elberfeld, Germany. Eng. Pat. 1771, January 31, 1889. 6d.

PHENETIDINE (*p*-amidophenol ether) is diazotised and combined with phenol. This azo compound is converted into the ether which is subsequently reduced with iron and hydrochloric acid, yielding two molecules of phenetidine. The methyl or amyl ethers may also be used. The following are the quantities employed: 50 kilos. of *p*-amidophenol ether hydrochloride are diazotised and combined with 32 kilos. of phenol in an alkaline solution. 30 kilos. of the yellow dyestuff formed—*p*-phenol-ether-azo-phenol—are converted into the ethyl derivative by dissolving them in 300 litres of alcohol, adding 6.5 kilos. of caustic alkali and 15 kilos. of chloride or bromide of ethyl and heating in an autoclave to 100° C. The alcohol is then distilled off and the alkylised derivative, insoluble in soda lye, is separated from any non-alkylised colouring matter. In order to reduce the ether 30 kilos. of it are added to 120 litres of water and 70 kilos. of hydrochloric acid of 20° B., and 20 kilos. of iron filings are slowly added. When the reduction is complete lime is added to the solution, and the phenetidine distilled over with steam. By heating the *p*-amido-phenol ethers with dry sodium acetate and acetic acid they are converted into the mono-acetyl derivatives, which are employed as drugs. The acetyl derivative of *p*-amido-phenol-ethyl ether is phenacetine.—T. A. L.

*Improvements in Fixing Perfumes.* A. Domeier, London. Eng. Pat. 3745, March 2, 1889. 4d.

THE patentee adds about 4 per cent. of glycerol to such common perfumes as consist of odorous substances dissolved in alcohol (e.g. Eau-de-Cologne) and claims that it aids in retaining odour which is apt to be carried off by the evaporation of the alcohol.—B. B.

*An Improved Toilet and Nursery Powder.* H. W. Langbeek, Loughton. Eng. Pat. 4643, March 16, 1889. 4d.

PURIFIED wool fat or lanolin is warmed and mixed with an absorbent such as carbonate of lime, gypsum, fuller's earth, or kaolin; the product, which may contain as much as 20 per cent. of wool fat without losing its powdery appearance, is mixed with one to four parts of starch powder, alone or together with wheat flour, perfumed, and is then claimed as an improved toilet powder. Antiseptics such as carbolic, salicylic, or boric acid, iodoform, or mercuric chloride, may be added if desired. Hard specimens of wool fat may be softened previous to mixing by the addition of 10–30 per cent. of vaselin or lard.—B. B.

*An Improved Process of Recovering Volatile Solvents retained in Substances treated therewith.* H. H. Lake, London. From "La Société Anonyme des Parfums Naturels de Cannes," Paris, France. Eng. Pat. 10,280, June 24, 1889. 6d.

THE process consists in recovering the solvent from the spent material in the liquid state without volatilising it (unless it be a very small part), and so obviating all the expenses necessitated by the successive vaporisation and condensation. A small jet of steam is introduced into the extractor above the material instead of below, as in the ordinary method, and gradually forces the solvent downwards, expelling it in the liquid state through the lower part of the extractor into a reservoir. The steam having slowly passed through the entire material, escapes through the bottom of the extractor, being mixed with the last traces of the solvent, which may be collected by passing it through a refrigerator before entering the reservoir.—E. S.

*An Improved Method or Process of and Apparatus for the Lixivation of Various Materials.* H. H. Lake, London. From "La Société Anonyme des Parfums Naturels de Cannes," Paris, France. Eng. Pat. 10,361, June 25, 1889. 8d.

THIS invention is designed to combine the rapidity of action of that class of apparatus characterised by rapid circulation with the safety of a second class characterised by slow circulation. The improvement is "based upon the employment of two series of conjugated vessels which are all together alternately filled and emptied of the liquid used in treatment." For the mode by which this is effected, the drawing attached to the specification must be consulted. There are three claims.—E. S.

## XXII.—EXPLOSIVES, MATCHES, Etc.

*Smokeless Explosives.* Sir F. Abel. Royal Institution Lecture, January 31, 1890.

THE lecturer said that the introduction of machine and quick-firing guns had shown the great desirability of using a smokeless powder, and caused chemists to experiment with substances which would yield no solid products on combustion. "Amide powder," so called from the volatile potassium amide, which was believed to be formed from the mixture of potassium and ammonium nitrates contained in

it, gives very good results, the smoke rapidly clearing away, but it deteriorates after long storage at high temperatures as in the magazines of ships.

THE next important improvement was the powder produced by the French for their Lebel rifle. It is now known that it differs chiefly from other smokeless powders in containing picric acid. Attention was also called to the attempts made to use gun-cotton, which at first were not uniformly successful. The basis of the modern smokeless powders consists of different varieties of gun-cotton, and the lecturer referred to Schultze and E.C. powders, and then showed how gun-cotton could be dissolved in ether or acetone and dried in homogeneous films, which could be cut into shreds or the jelly could be passed through dies and the solid produced in any desired form. Nobel found that nitroglycerin dissolves or gelatinises gun-cotton, and he has in this way produced a very good powder entirely unaffected by water. Some camphor is incorporated with it to render it less sensitive to shocks, but the volatility of this ingredient is a drawback to the powder. It was pointed out that the very perfection of this powder caused new difficulties, for there being no solids deposited in the bore of the gun, the friction of metal on metal was found to be very great. The lecturer, however, was confident that these difficulties would be overcome, and had no doubt as to the ultimate adoption of smokeless powders for military and naval purposes.—W. M.

*Some Notes on Modern High Explosives.* Eng. and Min. Jour. 48, 567–568.

THE explosives referred to are arranged in five classes—(1) gun-cotton class, (2) the pierates, (3) the chlorate compounds, (4) the nitroglycerin preparations, and (5) the pancastites. In America the use of explosives of the first class is practically confined to military and naval purposes, and the only picric acid preparation known to be on the American market is Emmensite (this Journal, 1888, 868).

THE chlorate class is represented by rackarock, which consists of potassium chlorate put up in cartridge bags of cotton cloth, immersed for a few seconds before use in nitrobenzene or "dead oil."

UNDER the fourth division, the large number of nitroglycerin preparations is very remarkable. The nitroglycerin is used in the form of ordinary dynamite or with an explosive absorbent under the name of giant powder, and consisting in some instances of 40 parts nitroglycerin, 48 parts sodium nitrate, and 12 parts sawdust.

JUDSON powder is another preparation largely used and contains from 5–20 per cent. of nitroglycerin absorbed by an explosive base, which has approximately the following composition: sodium nitrate, 70 parts; sulphur, 15 parts; resin, 3 parts; asphalt, 2 parts; and anthracite coal, 10 parts.

ATLAS powder consists of nitroglycerin, wood fibre, magnesium carbonate, and sodium nitrate in varying proportions.

HERECULES powder in its strongest variety contains magnesium carbonate, 20·85 parts; potassium nitrate, 2·10 parts; potassium chlorate, 1·05 parts; white sugar, 1 part; and nitroglycerin, 75 parts. The "Miners' Friend Dynamite," manufactured by the Hecla Powder Company, is said to be very safe. Foreite is the most typical example of the gelatin-dynamite class, and consists of nitroglycerin partially gelatinised with soluble gun-cotton and then absorbed by an explosive base.

UNDER the fifth head are described, Roburite, Securite, Bellite and Romite, which have been already noticed in this Journal (1888, 589).—W. M.

## PATENTS.

*An Improved Smokeless Safety Gunpowder.* C. F. Hengst, Plumstead. Eng. Pat. 13,656, September 21, 1888; Amended December 21, 1889. 6d.

IN this amended specification (compare this Journal, 1889, 478) the only alteration in the text consists in the deletion of the following sentence: "I do not, however, confine myself to potassium permanganate as an oxidising agent, as



I find in practice I may employ with equal advantage any convenient oxidising substance to effect the desired object." In the fourth claim for the words "The utilisation of the dust or refuse, &c." are substituted "The utilisation in the manner described of the dust or refuse, &c." In the fifth, for "The various processes and materials" are substituted "The combination of the various processes and materials."

—O. H.

*An Improved Mode of Preparing Explosive Compounds for Use.* A. V. Newton, London. From A. Nobel, Paris, France. Eng. Pat. 1888, February 4, 1889. 6d.

THE object of this invention is to prevent nitrate of ammonia and other hygroscopic powders from absorbing moisture by enclosing the charges in explosive water-tight bags or cases. The bags or cases are composed of thin sheets of collodion or soft celluloid, the joints being secured by applying a suitable volatile solvent such as acetone or acetic ether.

The material, composed of nitroglycerin and nitrocellulose, as described in Eng. Pats. 1471 of 1888 and 4479 and 9361 of 1889 (this Journal, 1889, 214), is, however, more suitable for making the explosive bags. This material rolled into sheets can easily be made into bags or cases by heating to 80° C, the parts to be joined and pressing the surfaces together, or volatile solvents may be used, as in the case of collodion.—W. M.

*Improvements in the Manufacture of Explosive Compounds.* H. S. Maxim, Crayford. Eng. Pat. 4477, March 14, 1889. 6d.

By the addition of oil, "preferably castor oil," to compounds of dissolved gun-cotton and nitroglycerin, nitrocelatin, or the like, the inventor is enabled to produce an explosive which burns slowly and acts efficiently in rifles, can be cut or pressed into suitable forms, and does not deteriorate when exposed to atmospheric influences.

Suitable proportions are from 2—5 per cent. of castor oil, 10—16 per cent. of nitroglycerin or the like, and the remainder gun-cotton.—W. M.

*Improvements in Explosives and in the Method of Manufacturing the same.* P. de Montravel, London. From O. Mavaut, Herentals, Belgium. Eng. Pat. 5031, March 22, 1889. 6d.

THE object of this invention is to produce a cheap explosive which shall be more powerful than ordinary gunpowder. For this purpose one of the numerous nitrated hydrocarbon derivatives, such as dinitrobenzene, is added to the three substances composing gunpowder. The explosive thus produced can be exploded without a detonator. After mixing the four substances together, they are heated to 140°, when the dinitrobenzene and sulphur melt and cover the other ingredients with a film, which on cooling prevents the moisture from acting on the explosive.—W. M.

*Improvements in the Manufacture of Explosive Compounds.* E. Liebert, Berlin, Germany. Eng. Pat. 5503, March 30, 1889. 4d.

THIS invention consists in the addition of 3—5 per cent. of iso-amyl nitrate to nitroglycerin; or an emulsion of glycerin and iso-amyl nitrate or iso-amyl alcohol may be nitrated. It is claimed that nitroglycerin thus prepared does not freeze at 35° below zero, is less sensitive to blows and shocks than pure nitroglycerin, and also that the explosive force is greater than that of pure nitroglycerin.—W. M.

*An Improved Process of Producing Ammonium Nitrate.* A. W. and J. A. Wahlenberg, Stockholm, Sweden. Eng. Pat. 12,451, August 6, 1889. 4d.

See under VII., page 293.

## XXIII.—ANALYTICAL CHEMISTRY.

*Pyrometry.* H. Le Châtelier. Soc. tech. de l'Ind. du Gaz.

THE author first enters upon a preliminary discussion of temperature, and the general principles of measurement of temperature as illustrated by the mercury thermometer, the hydrogen or air thermometer, and Wedgwood's pyrometer. He states that the hydrogen thermometer (which is most nearly correct) and the nitrogen and the mercury thermometers are related to one another in the following way:—

Hydrogen Thermometer.	Nitrogen Thermometer.	Mercury Thermometer.
0	0	0
50°	50°01	50°10
100°	100°00	100°00
150°	149°86	149°75
300°	300°15	298°79

The best known temperatures at the present time are:—ice melting point, 0° C.; water boiling point, 100° C.; naphthalene boiling point, 218° C.; then, with possible errors of 2°, we have mercury boiling point, 360° C.; and sulphur boiling point, 448° C.; then, with possible errors of 25° C., we have aluminium melting point, 650° C.; and gold melting point, 1,045° C.; and lastly, with possible errors of 50° C., are palladium melting point, 1,500° C., and platinum melting point, 1,775° C.

*Hydrogen Thermometers.*—Unworkable in furnace operations; glass cannot be used at high temperatures; metals are permeable to hydrogen when hot; porcelain is fragile, its enamel rarely sufficiently impermeable, and the joints are apt to give way by destruction of the mastic. Air is better than hydrogen, but both are only for the laboratory.

*Dilatation of Solids.*—Porcelain expands four millionths of its length per degree Centigrade; the expansion of silver increases from an average of 19·6 millionths between 0° and 100° to an average of 21 millionths per degree for temperatures between 0° and 890° C. Platinum has a more regular expansion than silver, but a smaller one (9 instead of 19 millionths); and in any case these expansions are very difficult to measure. Silver is soft when hot. A rod of platinum 4 in. long would have lengthened at 1,000° C. (1,832° F.) by less than  $\frac{1}{16}$  in.; but the greatest obstacle to this method is that metals, alternately heated and cooled, take up new sets and forms, which vitiate all the observations.

*Shrinkage of Clay.*—Wedgwood's pyrometer now quite abandoned; altogether too variable and irregular.

*Fusible Masses.*—Used with great success at the Sèvres potteries; made of the following mixtures:—

Temperatures of Fusion.	Felspar.	Kaolin.	Quartz.	Marble.	Iron Oxide.
° C.					
1,150	83·5	0·0	65	35	16·0
1,208	83·5	19·5	57	35	4·0
1,266	83·5	25·9	84	35	0·0
1,328	83·5	51·8	132	35	0·0
1,410	83·5	90·6	204	35	0·0
1,468	83·5	112·5	300	35	0·0
1,596	83·5	194·25	396	35	0·0

Intermediate compositions enable a scale of temperature (of about 25° per step) to be obtained. Better suited for watch-



ing a continuous rise in temperature than for preventing fluctuations.

**Fusible Alloys.**—Metals fuse abruptly: hence suitable if otherwise so; but mostly oxidisable. Alloys of silver, gold, palladium, and platinum are used; but they change in composition (liquefaction) and melt only gradually. Between two metals there is always one utilisable alloy which melts like an ordinary metal without liquefaction. For use, these metals and alloys are too expensive.

**Siemens' Electric Pyrometer.**—The electric resistance of metals increases rapidly with their temperature. Siemens' pyrometer, made on this principle, unfortunately does not remain constant in its readings, for the platinum wire employed becomes attacked by sulphur, silica, &c., unless it be enclosed in a platinum tube (iron will not do), which is too expensive.

**Speed of Outflow of Air.**—Mr. Barus has (*American Journal of Science*, 1889, 516) suggested a simple method. Air is driven through a tube; in its course it passes through two small orifices or capillary tubes—one of these is at the furnace temperature, the other at the ordinary. The things to measure are the changes of pressure at the obstructions; from these the temperature may be very simply calculated.

**Water Pyrometer.**—Boullier's. Water is made to circulate with a uniform velocity in a metallic tube exposed partially to the heat to be ascertained. Difficulties:—The cold tube in the hot gases condenses water vapour on it; upon the moisture settle down ashes and smoke. The muddy coating thus formed interferes with the absorption of heat.

**Calorimetric Methods.**—A lump of heated metal thrown into water; measure the rise of temperature in the water; calculate. The only metal the specific heat of which is really known is platinum. The quantity of heat lost by a kilogramme of platinum in cooling from  $t^{\circ}\text{C.}$  to  $t^{\circ}\text{C.}$  is, in great calories, equal to  $0.0317(t-t_0) + 0.000006(t^2-t_0^2)$ . The rise of temperature in the water should never exceed  $2^{\circ}$ , else errors due to radiation come in; and therefore the thermometers should read to  $\frac{1}{1000}^{\circ}\text{C.}$  But platinum and delicate thermometers of this kind are not for rough work; iron and a considerably greater rise in temperature are much resorted to. Consequently, results not reliable within  $100^{\circ}\text{C.}$ , and therefore of no use for calculating heat losses, &c., but still useful for regulating the furnace, if the same observer do the same thing under similar circumstances on each occasion. Iron in itself is an unsuitable metal, because at different temperatures it appears to undergo abrupt changes, and to evolve latent heat; this is apt to be confounded with the true cooling. Nickel is better in this respect.

**Optical Methods.**—(1.) The eye alone: The colour of the glow is watched. Dull red,  $525^{\circ}\text{C.}$ ; cherry red,  $800^{\circ}\text{C.}$ ; deep orange,  $1,100^{\circ}\text{C.}$ ; white,  $1,300^{\circ}\text{C.}$ ; dazzling white,  $1,500^{\circ}\text{C.}$  (Pouillet). (2.) The intermediate yellow part of the spectrum is cut out, leaving only the reds and blues; the change of tint is then far better marked. For this use, (a), a plain cobalt glass is employed, and the colour judged, which passes from red through white to blue; or, (b), Nonel and Masure's instrument—two Nicols with a quartz between. Rotation of one of the Nicol prisms causes a gradual change from red to yellow, green, or blue, according to the temperature of the object. The angle of rotation necessary to extinguish the red is observed. Chiefly useful for detecting variations in temperature.

**Thermo-electric Pyrometer.**—The method preferred by M. Le Châtelier. Becquerel, in 1834, proposed a palladium-platinum couple; first used by Pouillet, who employed iron instead of palladium; condemned by Regnault. Edmond Becquerel found a difficult formula for correcting the readings. The advantages are, small bulk, prompt equilibrium of temperature, reading taken at any convenient place or distance. According to Le Châtelier's results, (1), the couple should be composed of absolutely homogeneous wires which give no current when heated at any one point. Becquerel's, Pouillet's, Regnault's instruments did not fulfil this condition; iron and palladium do not do so. The condition is fulfilled by a couple consisting of platinum and an

alloy of platinum with 10 per cent. of rhodium, the readings given by which are consistent under all circumstances. (2.) The galvanometer ought to be one of high resistance, so as to maintain the intensity practically proportional to the electro-motive force. Galvanometers with movable needles are not adapted for rough work. Deprez and D'Arsonval's galvanometer works best, especially as modified by Le Châtelier with a view of hanging it in two parts on the wall. (3.) The couple may be graduated by plotting on square paper the indications given by boiling water, naphthalene, and sulphur, and by fusing aluminium, gold, and palladium, all which give known temperatures. The result is, with the platinum and rhodium-platinum couple, a curve which is almost straight between  $400^{\circ}\text{C.}$  and  $1,200^{\circ}\text{C.}$  The fusion-temperature readings of the metals are ascertained by putting a lithe wire across from the Pt to the Rh-Pt and gradually heating up. The galvanometer-reading gradually goes up as the temperature rises; it stops going up when fusion begins, and the current suddenly stops when the liquid bead of metal breaks. Once the graduation is accomplished, this thermo-electric couple at once gives, with the aid of the diagram, results correct within  $10^{\circ}\text{C.}$ , a result not hitherto attained. The inconveniences are, (1), that though a workman may use it, an electrician must put it up and graduate it; (2), that reducing (not oxidising) heated gases tend to alter the platinum, but this is much less felt than with the Siemens' instrument so long as the couple is not broken, and the wire is here thicker ( $\frac{3}{16}$  in.) and more durable. Still, it is well to expose no more than 2 in. of the wire, and to cover the rest with insulating porcelain in an iron tube, and in a reducing atmosphere, to cover the tip with an iron cap.

This instrument enables observation to become much more precise, and to give data exactly comparable as between one works, or one industry, and another.

#### *A New Method of Estimating the Oxygen dissolved in Water.* J. C. Thresh. Proc. Chem. Soc. 1890 (76), 1–3.

THE process is based on the observation that whereas, in absence of oxygen, nitrous acid and hydrogen iodide interact to form iodine, water and nitric oxide; in presence of oxygen the nitric oxide becomes re-oxidised, and, serving as a carrier of the oxygen, an amount of iodine equivalent to the oxygen present is liberated, in addition to that resulting from the initial action of the nitrous acid: hence, deducting the amount liberated by the nitrous acid and by the oxygen dissolved in the solutions used from the total amount, the difference will be that corresponding to the oxygen dissolved in the water examined.

The solutions used are: (1) a solution containing 5 grm. sodium nitrite and 20 grms. potassium iodide in 100 cc.; (2) a solution of 7.75 grms. sodium thiosulphate in 1 litre, 1 cc. of which corresponds to 0.25 mgrm. of oxygen; (3) a clear solution of starch; and (4) diluted pure sulphuric acid (1:3).

The apparatus required is as follows: A wide-mouth, white glass bottle of about 500 cc. capacity, provided with a caoutchouc stopper, through which four holes are bored. Through one passes the neck of a cylindrical "separator" funnel of known capacity, and through the second a tube drawn out to a fine point, which is connected by a short length of caoutchouc tubing with the thiosulphate burette; while inlet and exit tubes for coal gas are passed through the third and fourth holes, the exit tube having attached to it a sufficient length of caoutchouc tubing to permit of connexion being established between the bottle and the separator when the stopper of the latter is withdrawn.

The separator is filled with the water to be examined, and 1 cc. of the nitrite-iodide and 1 cc. of the acid solution are added; if the pipette be held vertically, with its end just below the surface of the water, the solutions flow in a sharply defined column to the lower part of the separator, so that an infinitesimally small quantity (if any) is lost in the water which overflows when the stopper is inserted. The admixture of the liquids having been effected by inverting the apparatus several times, a sharp current of coal gas is passed into the bottle to displace the air, the escaping

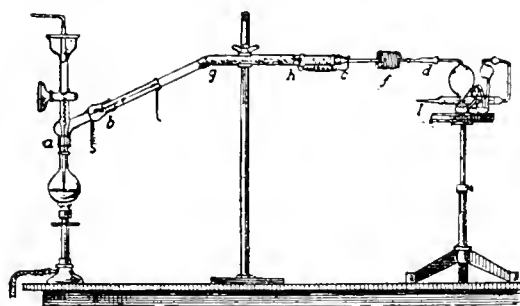
gas being allowed to burn at a jet attached to the exit tube. Fifteen minutes after adding the solutions to the water the flame is extinguished, a cork is attached in place of the jet and is inserted in place of the stopper of the separator, and the water is then allowed to flow into the bottle; the exit tube having been disconnected from the funnel and the gas set fire to, thiosulphate is run in until the colour of the iodine is nearly destroyed; about 1 cc. of starch is then added from the separator and the titration is completed. The effect of the nitrite, dilute acid and starch solutions is readily determined by removing the separator and adding 5 cc. of each in succession and then titrating; the effect of the oxygen in the thiosulphate may be allowed for on the assumption that as much oxygen is dissolved in it as distilled water would contain at the same temperature. It appears that there is no advantage in passing the coal gas through alkaline pyrogallol.

The author stated that concordant results were easily obtained, and that the results in the case of freshly distilled water closely agreed with those recently published by Roscoe and Lunt (this Journal, 1889, 729). Thus:—

Temperature.	Thresh.	Roscoe and Lunt.
°		
10	7.81	7.77
15	7.02	6.96
20	6.17	6.22
25	5.60	5.60
30	5.45	5.43

*Apparatus for Estimating Carbonic Acid in Carbonates.*  
Finkener. Mitt. k. techn. Versuchsanstalt, Berlin, 1889, 156—158.

THE apparatus represented in Figure, at one-eighth actual size, needs further description only in regard to one or two details. The joints are ground, and to give additional firmness are held together by external spirals. The limb *bg* is sufficient to condense for the most part, and return the



acid vapours from *a* kept gently boiling; at *g* is a plug of cotton wool, from *g* to *h* calcium chloride, from *h* to *c* phosphorus pentoxide. The accuracy of the results obtained with the apparatus is sufficiently shown in the numbers given, both for blank experiments and for determinations of carbonic acid.—C. F. C.

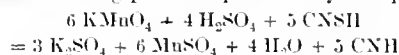
*Gravimetric Determination of Thiocyanates.* H. Alt.  
Ber. 22, 3258—3259.

THIOCYANIC acid when oxidised by nitric acid forms hydrocyanic and sulphuric acids, and this reaction is employed for the determination of thiocyanates, the sulphuric acid being precipitated as barium sulphate.

The thiocyanate is dissolved in water, an excess of crystallised barium chloride added, and then nitric acid, till the whole is strongly acid. The solution is warmed gently at first and then heated to boiling, to drive the

hydrocyanic acid out of solution. The resulting precipitate of barium sulphate is washed, dried, and weighed as usual. The method, compared with Volhard's volumetric process, gives satisfactory results.

The author states that the oxidation of thiocyanates by potassium permanganate in sulphuric acid solution is complete, but the liberation of hydrocyanic acid, and the fact that commercial thiocyanates usually contain some ferrous iron, render the process of little value. The decomposition taking place is represented by the equation—



—C. A. K.

*Separation of Zinc and Nickel.* H. Alt and J. Schulze.  
Ber. 22, 3259—3262.

WHEN sulphuretted hydrogen is passed into a strong succinic acid solution of zinc and nickel, the zinc is completely precipitated as snow-white sulphide, whilst the whole of the nickel remains in solution. The solution may be heated and an excess of the precipitant be employed without effect upon the accuracy of the separation. Test experiments were made with zinc sulphate and nickel nitrate, the salts being dissolved in water strongly acidified with succinic acid, and then treated with sulphuretted hydrogen. The precipitated sulphide of zinc was always quite free from nickel, whilst the analyses show that the separation is a quantitative one. In a mixture containing 22.66 per cent. of zinc and 20.39 per cent. of nickel, 22.66 per cent. of zinc and 20.19 per cent. of nickel were found. The succinic acid solution must be free from salts, otherwise nickel sulphide is thrown down with the sulphide of zinc. The way in which this is overcome, *e.g.*, in the case of an alloy containing tin, iron, copper, zinc, and nickel (German silver) is as follows:—The nitric acid solution of the alloy is concentrated, and the tin separated as oxide, and then the copper precipitated with sulphuretted hydrogen. The filtrate containing iron, zinc, and nickel is freed from sulphuretted hydrogen and the iron precipitated as basic acetate. An excess of precipitant should be avoided, owing to the necessity of removing the whole of the acetic acid before the zinc and nickel can be precipitated as carbonates; for a similar reason ammonium succinate cannot be used to precipitate the iron. After filtering off the iron precipitate, the small excess of acetic acid is removed by the addition of a little mineral acid, and the zinc and nickel precipitated as carbonates. These are filtered off, washed, and dissolved in succinic acid. The separation of the zinc and nickel can then be effected in an aliquot part of the resulting solution, by adding about 5 grms. of succinic acid, diluting with water and passing sulphuretted hydrogen into the boiling solution. The filtrate from the sulphide of zinc is concentrated and the nickel precipitated with caustic potash; the presence of much succinic acid in no way interferes with the complete precipitation of the nickel as hydrate.—C. A. K.

*Volumetric Determination of Combined Sulphuric Acid.*  
L. W. Andrews. Amer. Chem. J. 11, 567—571.

THE method is of general applicability. To the solution of the sulphate is added an excess of a solution of barium chromate in hydrochloric acid, the solution is neutralised with ammonia or calcium carbonate, and filtered; the filtrate is then acidified with hydrochloric acid, potassium iodide is added, and the free iodine is titrated with decinormal sodium thiosulphate solution.

A suitable solution of the barium chromate is prepared by digesting it with hydrochloric acid containing 36 grms. of acid per litre.

The analysis is carried out as follows: the sulphate to be determined is diluted until it does not contain more than 2 per cent. of  $\text{SO}_4$ , it is made approximately neutral, and is heated to boiling. While hot an excess of the barium chromate solution is gradually added, and the boiling continued for a minute or so. Calcium carbonate is next added in small quantities until no further evolution of carbon dioxide is observed. The whole is boiled, filtered

hot and washed. The filtrate after cooling is treated with a sufficient amount of crystals of potassium iodide, and with 5—7 cc. of fuming hydrochloric acid for each 100 cc. of liquid. Titration is then performed with thiosulphate, starch being used as indicator. In presence of ferric, nickel, or zinc salts ammonia must be used in place of calcium carbonate to neutralise the acid liquid.—T. L. B.

*On Crystalline Oxychloride of Mercury and the Estimation of Mercury.* J. Volhard. *Annalen*, **255**, 252—256.

THE oxychloride  $\text{Hg}_2\text{O}_2\text{Cl}_2$  was previously prepared by Millon and Ronches (Michaelis, *Lehrbuch der anorg. Chemie*, **3**, 1082); and Dietl (Tschermak, *Mineral. Mitth.* **1880** [2], **2**, 177). It may be easily prepared by adding sodium acetate to a cold saturated solution of mercuric chloride. In the course of some days crystals appear on the surface of the liquid, and as they increase sink to the bottom. They may easily be separated from a flocculent precipitate which is simultaneously formed. When the crystals cease to increase a solution of potassium bicarbonate is added, a little at a time, when a further crop is obtained. The crystals are granular, glossy, with rhombic faces; they appear to be very pointed rhombohedra. They rub down to a reddish-brown powder.

They have a sp. gr.  $\frac{17^\circ}{17^\circ} = 8.670$ . The substance is insoluble in water, easily soluble in acids, and decomposed by alkalis. On heating it is decomposed into the chloride, which volatilises, and the oxide which remains behind. The crystalline form was examined by Lueddecke, and the results are given in the paper.

It is known that mercury sulphide dissolves in caustic alkalis in the presence of alkaline sulphides. The author has found that it is completely precipitated from this solution by ammonium salts, and bases a new method of analysis on this.

The solution, which must contain the mercury as a mercuric salt, is nearly neutralised with sodium carbonate and then precipitated by ammonium sulphide, avoiding too great an excess of this latter, which should be freshly prepared. A solution of soda is then added with stirring until the dark liquid begins to brighten, when the solution is heated to boiling and more soda added until the liquid is quite clear. The soda must of course be pure, either potash by alcohol or soda from sodium may be used. The alkaline solution is precipitated by ammonium nitrate and boiled until all the ammonia is expelled. The precipitate is allowed to settle, which it does quickly, as it is much denser than the sulphide precipitated by sulphuretted hydrogen, washed with boiling water, and filtered off either on to a weighed filter or into a filtering tube. If it be feared that sulphur has been precipitated with the sulphide, it must be digested with sodium sulphite. In analyses of mercuric chloride by this method, the author obtained 73.86, 73.80, 73.85 per cent. of mercury respectively; 73.80 per cent. being the theoretical number.

—A. L. S.

*The Incineration of Vegetable Matter.* G. Lechartier. *Compt. Rend.* **109**, 727—731.

THE author finds that in determining the inorganic ash in vegetable matter by simple incineration in air there is a considerable loss of sulphur, but practically no loss of phosphorus. The former loss may be avoided by aspirating the volatile products through strong nitric acid.

The following is the method employed: The platinum crucible containing the organic matter is placed upon a clay plate which is heated in a gas furnace. The crucible is covered with an inverted glass funnel, which is fixed slightly above the rim of the crucible. The temperature is gradually raised, commencing at as low a temperature as possible. When the evolution of volatile organic matter ceases the bottom of the crucible is raised to a red heat for a few moments to complete the decomposition of the organic matter. The carbonised residue is washed with hot water and filtered through a washed filter-paper, which leaves no appreciable ash. The filter-paper and contents are introduced into the crucible, dried and heated to a dull

red heat. Incineration is rapid. The filtrate is then added to the incinerated ash, evaporated and the whole weighed, or the residue is treated with nitric acid previous to analysis. If on dissolving in nitric acid small particles of carbon separate out, these must be separated by filtration and again incinerated, for they may contain phosphorus.

For the estimation of phosphorus it is preferable to mix the matter with a quantity of milk of lime sufficient to moisten the mass throughout, which is then desiccated and incinerated.—J. B. C.

*Note on the Purification of Alcohol for Laboratory Uses.* E. Waller. *J. Amer. Chem. Soc.* **11**, 124—125.

THE alcohol is shaken up with powdered potassium permanganate till it is distinctly coloured. It is then allowed to stand for some hours until the permanganate is decomposed and manganese oxide is deposited. A small quantity of calcium carbonate is added and the alcohol distilled slowly. The distillate is tested until 10 cc. of it when boiled with 1 cc. of strong soda or potash gives no perceptible yellow colour on standing for half an hour. The subsequent distillate is collected for use; it is neutral and yields solutions with caustic alkalis and silver nitrate, which remain perfectly colourless even after boiling and standing for a long time.—E. E. B.

*Flashing Point of Lubricating Oils.* Holde. *Mitt. k. techn. Versuchsanstalt, Berlin*, 1889, 153—156.

THE two methods of testing oils for flashing point, viz., in an open crucible and in the closed apparatus (Pensky-Martens) usually employed at this institute, have already been shown to give results differing from 7° to 41° C. (this *Journal*, 1889, 734). Larger variations (to 150°) having recently been observed, the cause has been more closely investigated. Proof was obtained that the variations are due to the presence of small proportions of very volatile constituents. These may be either accidental constituents or may be added to bring a particular oil up to sample. The closed vessel test is of obvious importance for oils to be used in machines working with compressed air, such as torpedo engines.—C. F. C.

*The Distinction of Exalgin, Phenacetin, and Antifebrin.* Hirschsohn. *Pharm. J.* **20**, 710—711.

THE mixture (1 grm.) is treated with 2 cc. of chloroform, which dissolves the exalgin (methylacetanilide  $\text{C}_6\text{H}_5\text{M}(\text{CH}_3)(\text{CO}.\text{CH}_3)$ ) completely, but leaves the other two undissolved (*Pharm. Zeit. Russl.* Jan. 14, 17). Antifebrin in aqueous solution gives with bromine water a bromine compound, thus differing both from exalgin and phenacetin. Further chloroform solution of exalgin with 10 volumes of petroleum ether forms a clear mixture, whilst a solution of phenacetin or antifebrin is turbid under the same condition. Hirschsohn states that this method allows of the detection of an admixture of 20 per cent. of acetanilide or 10 per cent. of phenacetin with exalgin.

*Analysis of the Seed of Calycanthus Glauca.* H. W. Wiley. *Amer. Chem. J.* **11**, 557—567.

THE seeds of *Calycanthus Glauca* are poisonous, and bring death to a large number of American cattle. Eccles has discovered in the seed an alkaloid which he has named *calycanthine*. The present author examines more closely the occurrence of this alkaloid. The oil, which was removed from the seeds by petroleum spirit after crushing, was extracted with dilute sulphuric acid; the acid liquid, after being shaken with petroleum spirit to remove fat, was made strongly alkaline with ammonia and shaken with ether. The alkaloid thus obtained amounted to only 0.027 per cent. The seeds themselves, however, extracted similarly, gave 3—4 per cent.

Calycanthine crystallises from ether in feathery masses, insoluble in alcohol and in water. The colour reactions are interesting: Sulphuric acid, pale yellow; nitric acid, bright green; sulphuric acid + sucrose, purple, changing

finally to blue; sulphuric acid and potassium bichromate, brick red. The presence of sugar in the seed renders it possible to obtain the furfural reaction in the raw seed by the simple addition of sulphuric acid.—T. L. B.

*Adulterations of French Turpentine Oil. Qualitative and Quantitative Determinations.* A. Aignan. *Compt. Rend.* 109, 944—946.

French turpentine is frequently adulterated by the addition of resinous oils, though only to the extent of about 5 per cent., as a large quantity would render the oil viscous and give it a characteristic smell. Now by the addition of these resinous oils the rotatory power of the oil of turpentine decreases. That of the latter is nearly constant. With 16 different samples the numbers only varied from  $-60^{\circ} 26'$  to  $-63^{\circ} 20'$ . The average of  $[\alpha_D] = -61^{\circ} 30'$ . The white oils which are obtained by distilling colophony and further rectification may be divided into three groups. The purer the oils the greater their rotatory power. The oils possessing the following rotatory power are taken as typical:  $[\alpha_D] = -72^{\circ}$  or  $-32^{\circ}$  or  $-21^{\circ}$ .

These different qualities were mixed with oil of turpentine; the results of the determinations of  $[\alpha_D]$  are subjoined:—

Mixture with oil of first quality—

$$[\alpha_D] = -61^{\circ} 30' + \frac{7^{\circ} 30'}{5} h.$$

Mixture with oil of second quality—

$$[\alpha_D] = -61^{\circ} 30' + \frac{8^{\circ} 30'}{5} h.$$

Mixture with oil of third quality—

$$[\alpha_D] = -61^{\circ} 30' + \frac{9^{\circ} 30'}{5} h.$$

$h$  signifies the percentage of resinous oils contained in the mixture.

In certain cases oil of turpentine is mixed with essence of rosin, that portion of the products obtained in the distillation of colophony or pitch, which has the lowest boiling point. It may be detected by its characteristic and strong odour. The results of the above determinations may be summarised in the formula—

Oil of turpentine and ordinary essence of rosin—

$$[\alpha_D] = -61^{\circ} 30' + \frac{6^{\circ}}{5} e.$$

Oil of turpentine and refined resinous oils—

$$[\alpha_D] = -61^{\circ} 30' + \frac{3^{\circ}}{5} e.$$

$e$  denotes the amount of essence of rosin contained in the mixture.—A. R.

*An Application of Thermo-Chemistry.* A. Colson. *Compt. Rend.* 109, 743—745.

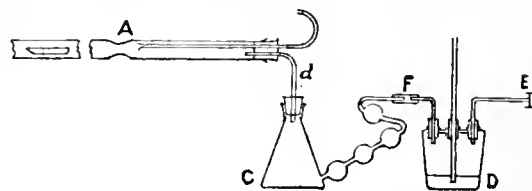
The author points out that thermo-chemistry may yield valuable data in determining methods of analysis of complex organic compounds, as, for example, the polybasic alkaloids. Thus, nicotine forms two salts with one and two molecules of acid. Thermo-chemical determinations show that the heat evolved in the case of the first molecule is double that of the second, and that, according to the results of Berthelot, the alkalinity of the second basic group should be too weak to effect litmus. The author finds this to be the case, and bases upon it a method of titration, using methyl-orange as indicator. He further finds that if the tin be precipitated from a solution of the pure double chloride of tin and nicotine, that titration indicates five-sixths of the total chlorine, four-sixths being derived from the tin, and one-sixth from the nicotine, another proof of the different basicities of the two basic groups in the alkaloid.—J. B. C.

*Estimation of Sulphur in Organic Compounds.* W. M. Burton. *Amer. Chem. J.* 11, 472—474.

The method is a modification of the method proposed by Sauer (*Zeits. Anal. Chem.* 12, 32 and 178), which consists in burning the substance in a current of oxygen, absorbing the sulphur dioxide in bromine water containing hydrochloric acid, and determining the sulphuric acid as barium sulphate.

As however the indicator, tropaeolin OO, is sensitive neither to carbonic acid nor to alkaline sulphites, it is practicable to absorb the sulphur dioxide in an excess of a standard solution of potassium hydrate, and titrate back the excess of alkali by means of a standard acid, using tropaeolin OO as indicator. The titration may be made immediately after the combustion is finished, and the operations of precipitating the barium sulphate, igniting, and weighing are avoided.

The apparatus is represented in the accompanying Figure.



The combustion tube is narrowed, as shown at A, the flask and bulbs C contain the standard alkali solution. D is a three-necked bottle, containing a layer of water, and fitted with a safety tube; at F this bottle is connected with the absorption bulbs, and at E with an aspirator for reducing and regulating the pressure in the combustion tube. After the substance has been completely burned, the tube is swept out by means of a current of air or oxygen, and the apparatus is allowed to cool. The combustion tube, the inlet tube for the oxygen, and the connecting tube  $d$  are rinsed with water to remove traces of  $\text{SO}_2$ , and the rinsings are added to the liquid in the absorption apparatus. A few drops of tropaeolin solution are then added, and the excess of alkali is titrated directly in the absorption apparatus. The standard sulphuric acid solution is made of such a strength that 1 cc. is equivalent to 5 mgrms. of sulphur; the standard alkali is made equivalent. If any of the halogens, phosphorus, or arsenic be contained in the substance under examination, this method, of course, cannot be used.

—T. L. B.

*Ash Determination in Raw Sugars.* G. F. Weichmann. *School of Mines Quarterly*, New York, 11, 15—23.

The old method of determining the ash in sugar consisted in carbonising the sample at a low heat, extracting the soluble salts by boiling water, and igniting the residue, adding this ash to the aqueous extract and evaporating to dryness. In 1864 Scheibler (*Zeits. Rübenzucker, Ind.* 14, 188) published his method of incinerating with sulphuric acid and deducting one-tenth.

Landolt (*Ibid.* 18, 68) holds that the weight of the salts in beet-sugars is about twice the weight of ash found by analysis. Boivin and Loiseau (*J. Fabrics. Sucre*, February 6, 1868) endorse Scheibler's method subject to the condition that the combustion is slowly conducted. Dubrunfaut (*Ibid.* December 18, 1870) considers Scheibler's "Factor 0.9 is nothing but a general average factor, which in many cases may be considerably far from the truth." Violette (*Ann. Chim. Phys.* [4], 29, 514) considers that the sulphuric acid method gives the weight of ash higher than the true weight. Von Lippmann (*Zeits. Rübenzucker Ind.* 31, 399) quotes experiments to prove that Scheibler's sulphate ash, less one-tenth, is exactly equal to the ash estimated as carbonate for beet-root sugars.

The author of this paper has made careful determinations of the ash of various kinds of raw cane and beet sugars both as carbonate (by a special method of analysis which is described in detail) and as sulphate. He finds that the results by the Scheibler method are almost without exception lower than those found by the other process. Direct experiment also proved that this was not due to loss of alkaline sulphates by volatilisation. Finally he finds that if the results by the Scheibler method be taken before the deduction of one-tenth they, in most cases, agree almost exactly with the results of the estimation as carbonate. He therefore endorses the recommendation of Dubrunfaut (*Le Sucre*, 1878, 2) that the sulphate ash as actually obtained be adopted as a working factor.

He says "the possible objection as to the expense of platinum muffles, where several must be used on account of the number of daily determinations, has been overcome in our laboratory by the use of muffles made of Russian sheet iron, which may be had at one-hundredth the cost of the platinum ware, which last on an average between three and four months, and which give absolutely no contamination to the sugar ashes burnt off in them."—A. J. K.

*Analysis of Marine Oils.* R. W. Moore. J. Amer. Chem. Soc. **11**, 155—158.

SOME marine oils examined by the author were found to prove an exception to the general rule, that natural fats and oils contain about 95 per cent. of insoluble acids. These marine oils are known as porpoise jaw and black-fish jaw oils, and are obtained from the soft fat of the head and jaw by cooling the oil which exudes from it and separating the portion which remains fluid. The resulting oil, skimmed and strained, is used for lubricating fine machinery. The samples of oil examined were:—

- 1 and 2. Porpoise jaw oil, skimmed and strained.
3. Porpoise jaw oil, not skimmed and strained.
4. Black-fish jaw oil, skimmed and strained.
5. Black-fish body oil.

The amounts of soluble and insoluble fatty acids were:—

	Soluble Fatty Acids.	Insoluble Fatty Acids.
	Per Cent.	Per Cent.
1 .....	17.18	72.05
2 .....	21.44	68.41
3 .....	.. ..	96.50
4 .....	21.79	66.28
5 .....	2.46	93.67

The acidity of the volatile fatty acids obtained by a modification of Reichert's process should be expressed as valeric acid, but for purposes of comparison with other analyses were calculated as butyric acid.

	Reichert's Figure.	Total Acidity.
	Cc.	Per Cent.
1 .....	47.77	17.18
2 .....	56.00	20.97
3 .....	2.08	1.42
4 .....	65.92	24.31
5 .....	5.60	2.34

In order to obtain satisfactory saponification numbers by Koettstorfer's process a large quantity of alcohol had to be used. Results:—

	Mgrams, KHO per Grm.
1 .....	253.7
2 .....	272.3
3 .....	143.9
4 .....	290.0
5 .....	197.3

The results yielded by Hübl iodine absorption process were:—

	Grms. of Iodine per 100 Grms.
1 .....	49.6
2 .....	30.9
3 .....	76.8
4 .....	32.8
5 .....	99.5

The oils which had received the full purifying treatment, 1, 2, and 4, were thus shown to furnish abnormally high

figures for soluble fatty acids and saponification numbers, and low figures for the insoluble fatty acids and iodine absorption, which indicates that the glycerides of the lower fatty acids have been concentrated in the oil which is finally strained off.

The author intends to examine butter fat by freezing it, straining and analysing the resulting product, and hopes in this way to obtain information regarding the composition of the molecular groupings in butter fat.—E. E. B.

*The Examination of Commercial Glycerin.* J. H. Wainwright. J. Amer. Chem. Soc. **11**, 125—130.

ALL so-called crude glycerin imported into the United States is examined at the United States Laboratory to ascertain whether it is really crude or has been partially or wholly refined, as in the latter case a higher rate of duty has to be paid. Crude glycerin may be freed from impurities by allowing them to subside and then straining or filtering them off, but if glycerin can be proved to have been submitted to further purification than this it is classed as "refined." For practical purposes of classification distillation is regarded as the dividing line between crude and refined glycerin. In order to distinguish between crude and refined glycerin the author attaches great importance to the estimation of the "carbonaceous residue."

Ten grms. of the glycerin are heated in a platinum crucible till it ignites, when the source of heat is removed and the sample allowed to burn away spontaneously. The residue is called "carbonaceous residue." In distilled glycerin this residue is rarely more than 0.5 per cent. and never as much as 1 per cent. (unless some adulterant is present which would be detected by other tests). Crude glycerin, on the other hand, frequently yields as much as 10 per cent. of carbonaceous residue. The ash may be determined by igniting the carbonaceous residue. The author regards as erroneous the statement of Sulman and Berry (this Journal, 1886, 177) that a definite distinction between crude and distilled glycerin is afforded by the amount of ash, and points out that in crude glycerin obtained by saponifying fats with steam the impurities are all organic and would consequently yield no ash. He has frequently met with samples yielding 3 per cent. of carbonaceous residues, but less than 0.05 per cent. of ash.

The silver nitrate and basic lead acetate tests are also considered very important.

*Silver Nitrate Test.*—5 cc. of the sample are diluted with 20 cc. distilled water, mixed with 5 cc. of a 2 per cent. solution of silver nitrate and allowed to stand for one hour. Only a slight precipitate will be formed with distilled glycerin at the end of this time, whereas with crude glycerin the precipitate is large, usually comes down at once and is almost always *flocculent*.

*Lead Test.*—The solution is prepared by boiling 10 grms. of lead acetate and 8 grms. of litharge with 500 cc. of water and filtering. Two volumes of this solution are mixed with one volume of glycerin and one volume of distilled water, and allowed to stand for one hour. Refined glycerin may produce a slight precipitate, but this is never *flocculent*. Crude glycerin produces a more or less abundant *flocculent* precipitate.

It is not safe to rely upon one of these last two tests alone, but if a sample of glycerin will not stand *both* of them it is perfectly safe to call it crude.

The other tests recommended as useful are:—

Addition of an equal volume of distilled water. *Refined* glycerin remains clear. In *crude* glycerin if much oil or fat be present they may be separated from the glycerin in this manner.

Ammonia. — *Refined*, no change; *crude*, precipitate indicates iron and alumina.

Ammonium oxalate. — *Refined*, no change; *crude*, precipitate indicates lime salts.

Barium chloride. — *Refined*, no change; *crude*, indicates sulphates.

Nitrogen peroxide (gas).—*Refined*, no change; *crude*, curdling indicates *fatty impurities*.

Fehling's solution.—*Refined*, no change; *crude*, shows the presence of *glucose*, &c.—E. E. B.

## New Books.

CHEMISCH-TECHNISCHES REFERTORIUM. Uebersichtlich geordnete Mittheilungen der neuesten Erfindungen, Fortschritte und Verbesserungen auf dem Gebiete der Technischen und Industriellen Chemie, mit Hinweis auf Maschinen, Apparate und Literatur. Herausgegeben von Dr. EMIL JACOBSEN. 1889. *Erstes Halbjahr*. Erste Hälfte. Berlin: R. Gaertner's Verlagsbuchhandlung, Hermann Heyfelder. London: H. Grevel and Co., 33, King Street, Covent Garden.

OCTAVO volume in paper cover; the first issue for the first half-year of 1889. It contains 132 pages of Subject-Matter, plentifully illustrated with wood engravings, and in these pages the following Subjects are treated of:—Building Materials, Cements, and Artificial Stone. Colouring Matters, Dyes, and Textile Printing. Fats, Oils, Illuminating and Heating Materials. Fermented Liquors. Tanning, Leather, and the Production of Glue. Textiles. Glass and Earthenware. Wood and Horn. India-rubber. Cements, Gums, &c. Lakes, Varnishes, and Paints. Metals.

ANLEITUNG ZUR CHEMISCH-TECHNISCHE ANALYSE ORGANISCHER STOFFE. Von F. M. HORN. Assistent für Chemische Technologie, derzeit an dem k. k. Technologischen Gewerbe Museum in Wien, früher an der k. k. Technischen Hochschule in Wien. 1890. Vienna: Verlag von Josef Safár. London: H. Grevel and Co., 33, King Street, Covent Garden.

OCTAVO volume, bound in cloth, price 5s. 5d. It contains 244 pages of subject-matter, illustrated by 32 wood engravings. The author has also collected together 35 Tables of the strengths, specific gravities of various solutions, spirits, &c. The following synopsis of contents will give an idea of the class of matter treated of and of the subdivision of the subject:—

*Section I.*—A. Pure Sugar Solution. B. Investigation of refined cane sugar, Cane Sugars, and Syrups. C. Analyses of Sugar Beets. D. Analyses of saturation deposit. E. Starch sugar (Glucose) Analyses. F. Milk sugar Analyses. G. Must Analyses. H. Animal charcoal Analyses.

*Section II.*—A. Investigation of varieties of Grain, of Flour, of amylaceous Cattle Foods, &c. B. Investigation of Potatoes. C. Dextrin. D. Malt and its extracts, worts, &c.

*Section III.*—A. Estimation of Alcohol generally (Spirits of Wine, Brandies, Arack, Rum, Cognac). B. Liqueurs and their Examination. C. Amyl Alcohol. D. Yeasts. E. Wines. F. Analysis of Tartars, &c. G. Beers. H. Glacial Acetic Acid, Vinegars, &c.

*Section IV.*—A. Milk Analysis. B. General Analyses of Fats, Waxes, &c. C. Butter Analysis. D. Analysis of Glycerin. E. Soap Analysis. F. Candles. G. Olein and Oleomargarin. H. Turkey-Red Oils. J. Lubricating Oils. K. Petroleum. L. Analysis of Etheral Oils. M. Balsams, Resins, and Gum Resins.

*Section V.*—A. Paper Analysis. B. Glue and Size Analyses. C. Analysis of Tanning Materials.

The work terminates with an Alphabetical Index.

## Trade Report.

### TARIFF CHANGES AND CUSTOMS REGULATIONS.

(From the Board of Trade Journal.)

#### RUSSIA.

##### The Importation of Patent Medicines.

According to the *Journal de la Chambre de Commerce de Constantinople*, the import into Russia of patent medicines can only be effected conformably to Art. 310 of the Medical Regulations and Art. 151 of the Customs tariff, upon the authorisation of the Medical Council of the Ministry of the Interior, in conjunction with the Ministry of Finance.

The Medical Council only examines patent medicines to which the inventors have attached the detailed description of the various parts of which the medicine is composed.

#### RUSSIA.—FINLAND.

##### Changes in Customs Tariff.

*Note.*—Kilog. = 2·204 lb. avoirdupois.

Mr. J. Michell, Her Majesty's Consul at St. Petersburg, in a report to the Foreign Office, dated the 12th February, says:—

According to information supplied by Mr. Vice-Consul Cooke, of Helsingfors, the Imperial Finnish Senate has ordered the following changes in the Finnish Customs tariff:—

1. The duty on Chilian saltpetre imported into the Grand Duchy during the year 1890, should no instructions to the contrary intervene, to be fixed at 20 penni per 100 kilogrammes.

2. The duty on saccharine to be in future at the rate of 150 marks per kilogramme.

#### SWITZERLAND.

##### Classification of Articles in Customs Tariff.

*Note.*—Quintal = 220·4 lb. avoirdupois. Franc = 9 $\frac{1}{16}$ d.

The following decisions affecting the classification of articles in the Swiss Customs tariff were given by the Swiss Customs authorities in the month of January:—

Water of chloride of zinc.—Category 17. Duty, 1 franc per quintal.

Liquid carbolic acid in receptacles of wrought iron.—Category 18. Duty, 2 francs per quintal. This article was formerly in Category 9a, and paid a duty of 7 francs per quintal.

Fats serving for food not distinguished, cocoa fat (cocoa-nut oil, cocoa butter, cocoa-nut fat, cocoa fat), purified.—Category 188. Duty, 7 francs per quintal.

In Category 259 the words "not purified" are added after "cocoa butter (cocoa-nut fat)."

#### UNITED STATES.

##### Recent Customs Decision.

The following decision affecting the classification of articles in the Customs tariff and the application of the Customs law of the United States has recently been given by the United States Customs authorities:—

So-called "salol" is dutiable as a chemical compound under the provision in the Tariff Act of March 3, 1883 (T. I., 92), for "all chemical compounds . . . not specially enumerated or provided for."

## GOLD COAST.

## Customs Tariff.

The following is a statement of rates of import duty now levied in Gold Coast Colony:—Gunpowder, 6*d.* per lb.; lead in any form,  $\frac{1}{2}$ *d.* per lb.; kerosine and other illuminating oils, soap, and sugar, 10 per cent. *ad valorem*.

The following articles are exempted from duty:—Acids; blacking; blue indigo; candles; charcoal; chemicals; drugs and medicines; filters; glass; india-rubber; scientific instruments; matches; oils (except kerosine as above); ores; paints; pitch and tar; quicksilver; salt; methylated spirits; and tallow.

## VICTORIA.

## New Customs Tariff.

The following is a statement of the rates of import duty now levied under the new Customs tariff of Victoria, dated the 4th November last:—

Classification of Articles.	Rates of Duty.
Drugs, viz.:	£ s. d.
Acid, acetic, containing not more than 30 per cent. acidity .....	Pint or lb. 0 0 3
Do., do., for every extra 10 per cent. or part of 10 per cent. above 30 per cent. ....	" 0 0 1
Do., muriatic .....	Cwt. 0 5 0
Do., nitric .....	" 0 5 0
Do., sulphuric .....	" 0 5 0
Ammonia, carbonate of .....	Pint or lb. 0 0 2
Do., liquid .....	" 0 0 2
Chlorodyne .....	25 ° <i>ad val.</i>
Cocculus Indicus .....	Lb. 0 1 0
Gelatin .....	" 0 0 6
Glycerin, pure .....	" 0 0 3
Do., crude .....	" 0 0 1
Morphia .....	Oz. 0 1 6
Nitrate of silver .....	" 0 0 6
Nux Vomica .....	Lb. 0 0 2
Strychnine .....	Oz. 0 1 0
Earthenware, including packing (except photographic, scientific, and telegraph materials), measuring outside the package as imported .....	Cubic ft. 0 0 8
Explosives (except fine meal powder, not sporting, in bulk and in packages of not less than 25 lb. each), viz.:	
Powder, sporting .....	Lb. 0 0 3
Do., blasting .....	" 0 0 1
Other explosives (except gelatin and gelatin dynamite, the duty on which shall be 1 <i>d.</i> per lb.) .....	" 0 0 4
Glue, liquid, and liquid gum and cements ....	20 ° <i>ad val.</i>
Inks, writing, liquid or powder .....	10 ° <i>ad val.</i>
Oils:	
In bulk (except cocoa-nut, fish of all sorts, mineral refined of which the point of ignition is below 80° F., kerosene, palm lubricating, of which the chief component part is mineral) .....	Gall. 0 0 6
Kerosene .....	" 0 0 6

## Classification of Articles.

## Rates of Duty.

Classification of Articles.	Rates of Duty.
Oils— <i>cont.</i>	£ s. d.
Packed in bottles, jars, or other vessels not exceeding one gallon in size, as under:	
Quarts and over a pint .....	Dozen 0 4 0
Pints and over half a pint .....	" 0 2 0
Half pints and smaller sizes .....	" 0 1 0
Over a quart and not exceeding a gallon .....	" 0 12 0
Paints and colours (except artists' colours):	
Ground in oil, including patent dryers, and putty .....	Ton 2 0 0
Mixed ready for use, from or of any substance .....	" 4 0 0
Paper (except writing and printing in original wrappers and incut edges as it leaves the mill; paperhangings and millboard):	
Cut .....	Lb. 0 0 2
Advertising matter, printed or lithographed, or any printed plates, lithographs, pictures, cards, or matter of a similar description used or capable of being used for advertising purposes ....	" 0 0 4
Cardboards and pasteboards .....	Cwt. 0 4 0
Paper, other .....	" 0 6 0
Do. bags .....	" 0 15 0
Medicines, consisting of two or more ingredients, mixed ready for use, not being in chemical combination; drugs and chemicals packed ready for retail sale or consumption, including medical compounds containing spirits not exceeding the strength of proof by Sykes' hydrometer; and all preparations recommended as beneficial for any portion of the human or animal body, or the cure or the treatment of any disease or affection whatever; and medicine chests or cases, with or without fittings .....	25 ° <i>ad val.</i>

## JAMAICA.

## Customs Tariff.

The following is a statement of some of the rates of import duty now levied under the Customs tariff of Jamaica:—

Classification of Articles.	Rates of Duty now levied.
Gunpowder .....	Lb. 0 1 0
Indigo .....	" 0 0 3
Matches, per gross of 12 doz. boxes, each to contain 100 sticks .....	— 0 5 0
Oil .....	Gall. 0 0 9
Soap .....	100 lb. 0 5 6
Spirits of wine, alcohol, &c. ....	Gall. 0 10 0

The following are admitted free of import duty:—Dye-wood, guano and other manure, resin and rosin, rock salt, soda ash or sub-soda.

An export duty of 1*s.* per ton is levied on logwood and other dyewoods.



## EXTRACTS FROM DIPLOMATIC AND CONSULAR REPORTS.

### CEYLON.

#### Industrial Undertakings.

In the course of the year 1889 a new oil works, with plant erected on European model, has commenced working in Colombo. The manufacture of biscuits from fine dried coprah is extending.

The following figures give some idea of the German share of Ceylon trade in 1889:—

—	Soap.	Spices.	Dyes.	Per- fumery.	Medicines.
	Rs.	Rs.	Rs.	Rs.	Rs.
Total imports..	121,563	211,339	78,979	69,014	175,317
German share..	27,795	6,348	3,534	3,033	1,665

### FRANCE.

#### Colouring Wine.

The Chilean Consul-General in London, in a communication regarding the use of the berries of a plant called the maqui for colouring wines, states that the maqui is a small evergreen shrub, common in Chili along the course of torrents and in shady mountainous woods. It is not cultivated, but grows wild, and the berries, which, in Chili, are eaten either fresh or preserved, are now beginning to be exported largely to Europe for the purpose of colouring wines. In the three years ending 1887 the exports were respectively as follows:—26,592 kilos., 136,026 kilos., and 431,392 kilos. To France alone the exports during the same years were, 500 kilos., 115,000 kilos., and 315,774 kilos. The berries are added to the grapes in order to give the wine a good colour.

### GERMANY.

#### Pharmaceutical Factories in Cassel.

Several firms in Cassel are engaged in the manufacture of surgical and pharmaceutical apparatus. They employ altogether about 500 workpeople, of whom 300 are women, and have an aggregate annual turnover of about 100,000*l*. The production of soaps in Cassel remains stationary at from 1,800 to 2,000 tons yearly, but in order to sell this quantity the makers have to content themselves with very low prices indeed. There are only about 50 workmen in this business, and their pay averages from 12*s*. to 15*s*. per week. There is one important firm of manufacturers of polishing pastes and powders who turn out their goods by means of machinery. They employ 30 hands, and pay the men from 1*s*. 10*d*. to 2*s*. 6*d*., and the women from 10*d*. to 1*s*. 2*d*. per day. They have great trouble to sell their goods in Germany owing to the excessive competition, but they find a good market for their surplus in the United States at remunerative prices.

### ITALY.

#### British Trade in Italy.

Some time ago the Italian Chamber of Commerce in London made inquiries from all the leading firms in Italy concerning their commercial relations with the United Kingdom, and the feasibility of the extension of their purchases from this country. The results of their inquiries were embodied in a long report, published in Italian, from which we take the following notes.

#### Fine Chemicals.

Mr. Melchiorre Petralia, pharmacist of Palermo, writes that in his district only the very smallest percentage of fine chemicals is imported from England, because articles of as good, if not better, quality are obtainable from Germany

at a lower price. Few, if any, of the finer pharmaceutical chemicals are made in Italy; but, on the other hand, all chemicals for current use are manufactured in that country.

#### Heavy Chemicals.

In 1888, writes Mr. Leo Negri from Liverpool, 11,604 tons of soda were imported into Italy from England, the value being 58,800*l*. France, Germany, and Austria also participate in this trade to some extent, but not seriously. Nevertheless, the imports of soda from England are diminishing, although the British article is generally preferred above any others, as its quality is better, and the sale conditions not so stringent. Mr. Negri does not think that there is any reason to believe that Italy will ever become independent from foreign countries for her soda supplies. Potashes are not bought in England at all. There are some factories in Tuscany, but they also do not produce much. The bulk of this article comes from Austria-Hungary, where the largest works exist. Britain, however, holds the field in bleaching-powder, hyposulphite of soda, nitric and carbolic acids, bichromate and chlorate of potash, phosphorus, carbonate of ammonia, nitrate of lead, &c.

#### Medicines.

Mr. Carlo Wiedemann, pharmacist to the German Emperor at San Remo, writes that the bulk of the goods purchased by him in England come under the denomination of proprietary medicines, for which he has a large sale among English residents in his district. He does not think it will be at all possible for Italian industry to compete in any way in this branch of trade.

#### Oils.

The export of linseed oil from Liverpool to Italy has been stopped altogether two or three years ago by the enormous duty put on the article by the Italian Government, and the four or five firms who were engaged in this business have given it up. It is thought that the Italian producers will gradually succeed in supplying the wants of the country independently of the foreigners.

### JAPAN.

#### Japanese Sulphur Mines.

The export of sulphur from Hakodate shows a decrease from 7,096 tons, value 16,917*l*., in 1887, to 3,609 tons, value 8,681*l*., in 1888. This is attributable almost entirely to the fact that no vessels could be induced to call at the port to take the sulphur away, the price, delivered in Hakodate (2*l*. 3*s*. 2*d*. per ton), being sufficiently high to prevent its being shipped. The shipments last year went to San Francisco.

What prevents Japan from rivalling Sicily as a sulphur-producing country are the absence of facilities for increasing the output of sulphur, and for shipping it. At present there is only one place where sulphur is worked on any scale, and that is Atosanobori, near Kushiro, on the south-east coast of the island on which Hakodate is situated.

The Atosanobori mine is part of an extinct volcano. The sulphur covers a considerable portion of the slopes of the hill and of the sides of the old crater. Supposing the ore to be only 4 or 5 feet thick on the surface, there must be at this place over 1,000,000 tons of high class ore. Recent borings (by Japanese) give the thickness of ore as 30 feet in some places, but it is probable that the richness of the ore decreases with the depth below the surface. It would be quite safe to count upon at least 5,000,000 tons of good ore. At present the ore yields over 50 per cent. of pure sulphur, a small portion of it being almost pure. The residue from the refining kettles would well repay a second treatment, as it appears to contain a considerable percentage of sulphur, as much as 15 per cent. probably, or about the average yield of the whole of the Sicilian sulphur ore.

The present output from Atosanobori does not exceed 10,000 tons of sulphur per annum (output about 9,000 tons in 1888). The cost of the sulphur, free on board at Kushiro, would be about 30*s*. 10*d*.

*Aniline Dyes.*

In spite of the fact that 1887 had already shown a strong increase in the import of aniline dyes over the preceding year, the figures in 1888 show a still further enormous increase; the imports have been in 1887, 306,884 cattie; and in 1888, 353,351 cattie, of which 249,443 cattie were imported directly from Germany. The most important aniline dyes for the Japanese market are the following:—Violet is still the most extensively used colour, although its sale is falling off, especially as far as concerns the commonest grade. Of blue, only best qualities are saleable, while the demand for magenta is very small compared with what it used to be, and only the best grades are still demanded. Green, in crystals, maintains its position, but powdered green has become almost obsolete. Brown, for silk-dyeing, is also declining in importance; eosin and erythrosin have become almost obsolete. The demand for good reddish tints of phloxin and scarlet is increasing. Corallin is now bought in lumps in England, this grade having superseded the powdered German article. The demand for picro, orange and yellow, is also on the wane; as regards orange only a few well-known brands which have an old reputation are still saleable, because they are the only ones which, mixed with phloxin, produce a certain colour which is much liked by the natives. Cotton red of the best quality finds a growing market. Alizarine dyes are gradually growing in importance, as the Japanese dyers are gradually surmounting the difficulties which they have hitherto experienced in using these dyes.

## NEW SOUTH WALES.

*Hints to Exporters.*

The import trade into Australia of chemicals, drugs, and medicines from Germany, says the German Consul at Sydney, could be easily increased to a considerable extent, but only by a close familiarity with the exact requirements of the local market. Perfumery also, eau de Cologne especially, is an article of very large consumption, and the same applies to toilet soaps. There should be an opening for goods of a superior grade. Natural mineral waters have not taken much footing yet, though some improvement is discernible. In general, it may be said that it depends, to a very large extent, upon the personal qualifications of the local representative whether a certain new article or unknown brand can be successfully introduced. Advertising is also a very large factor here, and British and American firms frequently spend large sums upon the introduction of their specialties by this means. If once an article has been successfully introduced, it is absolutely necessary to keep a sufficient stock always available here.

*Acetic Acid.*

In 1886 the imports of this article into New South Wales were 5,341 gallons, and in 1887 only 3,022 gallons. The German share of this trade was about 2,000 gallons each year, while the English business fell off considerably in 1887. The German acid, being concentrated, is about 30 per cent. dearer than the English.

## PERSIA.

*Gum Arabic.*

It is well known how the recent wars in the Soudan and the isolation of those tracts have affected the supply of gum. The effects of this are now being felt in Persia, where gum is now being extensively collected from the wild almond, "gavan," and other shrubs and trees, and measures are being taken to prevent the wholesale destruction of such woods. The exports during the last two years have been as follows:—

	Shiraz.		Bnshire.		Bunderabbas.	
	Cwt.	Value £	Cwt.	Value £	Cwt.	Value £
1888	485	940	7,917	7,977	6,010	1,836
1889	485	940	4,149	4,149	6,430	3,113

*Drug Trade Statistics.*

The following are the figures of imports and exports:—

*Imports.*

	Shiraz.	Bushire.	Bunderabbas.	Lingah.
	£	£	£	£
Drugs and medicines	1,458	6,359	3,004	1,293
Glass and glassware.	2,120	8,204	..	..
Indigo .....	40,831	25,985	8,676	..
Oil .....	1,701	5,251	535	4,685
Spices .....	10,719	7,176	5,482	6,481
Tea .....	9,080	14,248	61,311	770
Dyestuffs .....	..	516	350	1,806
Perfumery .....	..	..	..	1,913

*Exports.*

	Shiraz.	Bushire.	Bunderabbas.	Lingah.
	£	£	£	£
Drugs .....	4,857	2,243	26,036	3,207
Dyestuffs .....	1,863	1,351	7,542	979
Gum .....	940	7,977	1,836	..
Opium .....	230,886	148,523	127,393	..
Perfumery .....	5,401	9,466	..	..

*Opening of the Karoon.*

The most important event of the year 1888 was the consent of the Persian Government to throw open the River Karoon as far as Ahwaz to vessels of all nationalities; the ulterior object is, of course, to develop the shorter route for traffic between the sea and Central and Northern Persia. The project in itself is feasible and promising, but its success must largely depend on the measures adopted by the Persian Government to improve roads and secure their safety, to remove undue restrictions and taxation, and allow commerce fair play generally. The route was promptly inaugurated by Messrs. Lynch and Co. running their steamer *Blosse Lynch* between Busrah and Ahwaz experimentally. The success of the Karoon route will doubtless cause some redistribution of trade in Persia, but it may safely be predicted that the general prosperity of the country will be increased.

## SERVIA.

*Piracy of Trade Marks on Druggists' Goods.*

Among Servian imports there are a large quantity of the most shameless falsifications of British trade marks, which are openly resorted to, at the expense of the customers, and to the great danger of our reputation. Amongst such goods are:—Atkinson's scents, lozenges labelled "Savory and Moore," and soap stamped "SOAP" on one side, and manufactured by Messrs. Mousson and Co., Frankfurt-on-the-Main.

*Mineral Waters.*

A monopoly for the manufacture of mineral waters has been granted to Mr. Johann Erlicher, of Belgrade. The concession is exempted from the payment of imported duties for articles and machinery required for the business for 15 years, and the produce of the manufactory may be exported free during 10 years. The concessionaire is bound to begin work within 12 months.—*Chemist and Druggist.*

## MISCELLANEOUS TRADE NOTICES.

INTERNATIONAL EXHIBITION OF MINING AND METALLURGY  
IN LONDON.

During the summer of this year an International Exhibition of Mining and Metallurgy will be held at the Crystal Palace, Sydenham. The offices of the honorary secretary of the exhibition are at 18, Finch Lane, E.C. The following is a summary of the branches which will be represented:—

Machinery in motion and at rest; gold mining; silver mining; diamond mining; ironstone and iron ore mining; the manufacture of iron and steel; lead mining and manufacture; tin mining and smelting; copper mining; zinc mining and manufacture; coal mining; the petroleum industry; natural asphalt mining; mining for precious stones; the salt industry; mining for antimony, quicksilver, asbestos, arsenic, manganese, cobalt, platinum, bismuth, ochres, sulphur, uranium, and other minerals; alloys; the nitrate and phosphate interests; assaying, &c.; chemicals and the chemistry of mining and metallurgy; quarrying; clays; cement; electricity; explosives; the transport and shipment of minerals; scientific instruments; collections of mineral specimens; mining and metallurgical literature; mining tools and appliances; ambulance practice; and the condition of working miners.—*Board of Trade Journal*.

## METALS IN RUSSIA.

The Russian *Nouveau Temps* has published a comparison between the latest report of the Russian Mining Commission, edited by M. Koubeline, and that published in 1878 by M. Skalkovsky. The report does not give any figures later than the end of 1887, but the comparison supplies important data for measuring the progress of the Russian metal industry.

So far as the precious metals are concerned, the product of gold has fallen during these 10 years from 2,572 to 2,128 pounds, while that of silver has increased from 699 to 939 pounds, and that of platinum from 126 to 269 pounds. The figures are different for the precious metals chemically refined, namely, 1,898 pounds of gold and 1,043 pounds of silver in 1887. Those for platinum are unknown, the melting of that metal being mainly carried on abroad. Notwithstanding the affinity which exists between silver and lead, and the increase in the produce of silver, less lead was extracted in 1887 than was the case 10 years before—60,428 pounds instead of 85,281.

It is a misfortune for Russia that the amount of gold extracted is less than it used to be. It appears that less and less auriferous soil is discovered in the region of Yakontsk. On the other side, the fact that more gold is found in the quartz mines than formerly (164 pounds instead of 87) is of favourable augury for the future. It is not believed by Russian economists that there will be much progress in the gold industry until the great Siberian railway is finished, as this scheme takes away the labourers from all other work, and makes the price of machinery abnormally high. The only progress of the gold industry at the present moment is found in the province of the Amour.

Foreign competition has for a long time past been injurious to the production of copper, but the raising of the Customs tariff has made an alteration, by which the Caucasus has benefited. In 10 years the melting of copper has risen from 214,865 to 304,607 pounds. On the other hand, zinc has fallen from 283,398 to 221,250 pounds, in consequence of the decline of the home demand. Very little tin is obtained in Russia (629 pounds in 1887), and that only in Finland. Quicksilver, a rare metal, was not worked in Russia until 1886, but in 1887, 3,911 pounds of it were obtained. During the 10 years the extraction of manganese rose from 11,790 in 1878 to 3,553,535 pounds in 1887. In 1886, however, the figure was a million pounds higher; the sudden fall is due to the rivalry of Chili. There was also in 1887 a temporary diminution in the product of coal, but the 10 years show a great increase, from 153,981,727 to 276,778,774 pounds. It is in Poland and in the region of the Donetz that the quantity

of coal extracted has doubled, while it has diminished in the neighbourhood of Moscow and on the Kirghis Steppe. Fifty-eight per cent. of the coal consumed has been by the railways, of which, however, 16 per cent. was of foreign extraction.

Gigantic progress has been made in the working of naphtha. The manifest exhaustion of the American wells of good quality promises for Russian petroleum the ascendancy in the market of the world. During 10 years the product of Russian raw naphtha has risen from 15½ millions to nearly 167 million pounds. The asphalt industry is concentrated at Syzran. Since the abolition of the excise duty on salt, the extraction of that article has been greatly encouraged. The stimulus given to the iron trade by the protectionist system cannot be estimated by any figures given in the new report, for that system was only first applied in 1887.—*Ibid*.

## UNIFORMITY OF INDIAN WEIGHTS.

According to the *Pioneer Mail* for the 5th February, the Government of India have addressed the local governments regarding the practicability of securing a uniform system of weights in all India. That which appears the best solution, according to the Government, is the tolah of 180 grains, which is the exact weight of the rupee; the seer of 80 tolahs; and the maund of 40 seers. All railways have adopted these weights, and the tendency of trade will naturally be to follow this example. The local governments are asked to take the opinions of the principal trading associations before forwarding their replies.—*Ibid*.

## THE MINES OF CANADA.

The following information respecting the mines of Canada is extracted from the Canadian Official Handbook for 1890, the issue of which was notified on p. 202 of the February number of the *Board of Trade Journal*:—

*Mines*.—The exports under this head are chiefly coal, gold, copper, iron phosphate, salt, antimony, mineral oils, and gypsum. Gold mines are worked to a small extent in Ontario, Quebec, and the territories, but largely in Nova Scotia and British Columbia, the production from these two provinces in 1887 having amounted to 1,108,173 dols., and in 1888 to 1,050,670 dols. In British Columbia there are yet immense fields to open up. Silver mines are being worked in Ontario; and that at Silver Islet, Thunder Bay, has been the richest yet discovered in Canada. There are various deposits of minerals of all kinds in British Columbia. Iron ore is found all over the Dominion, and many mines have been successfully worked. Some of the Canadian iron ores are among the most valuable in the world. Copper is mined to a considerable extent both in Quebec and Ontario, and the deposits of the ore are of great extent.

There are very large coal deposits in Nova Scotia, and many mines are profitably worked. The coal is sent up by the river St. Lawrence and by rail into the interior. The coast of British Columbia is very rich in coal of a quality which commands a preference to the markets of San Francisco, notwithstanding the United States coal duty. Tests made by officers of the United States Government showed the British Columbia coal to excel that of California, Washington Territory, or Oregon, by one-fourth in steam-making power.

As regards the North-West territories, coal is known to exist over a vast region on and to the east of the Rocky Mountains. This region stretches from 150 to 200 miles east of the mountains, and north from the frontier for about a thousand miles. In places where the seams have been examined they are found to be of great thickness and of excellent quality. Beds of true bituminous coal have been found. There are also large quantities of lignite, which, for local use, where wood is scarce, will prove to be a useful fuel. It also occurs in considerable quantity along the valley of the Souris River, near the frontier, 150 miles west of Winnipeg. Good seams of anthracite coal are being worked in the Rocky Mountains at Anthracite and Canmore, and all these coals are now carried east and west by the

Canadian Pacific Railway—a fact of the greatest importance to settlers on the vast fertile belt of treeless prairies east of the mountains.

Specially among the minerals which are used for agricultural purposes may be mentioned apatite or phosphate of lime. The increasing demand for this valuable material has led to a great increase in production, both for home use and exportation. In 1887, 23,152 tons were exported, and the shipments in 1889 are estimated at 25,000 tons. The attention of the Canadian farmers is being directed to the valuable properties of this mineral as a fertiliser. The percentage of purity of native Canadian phosphate reaches from 70 to 95 per cent. It produces valuable results when applied to land in its raw state, reduced to a fine powder. But the conditions for manufacturing it into superphosphate are also favourable in Canada. This raw phosphate of lime commands a price from 25-50 dols. (about 5*l.* 2*s.* sterling) per ton in Liverpool, as appears by a recent market quotation. Phosphate of lime is found in large and easily-workable deposits throughout a large extent of country in the Ottawa Valley. The largest deposits have been found on the Quebec side.

Petroleum is known to exist in several parts of the Dominion, but the wells have only been profitably worked in Ontario. The production from them is very large, and appreciably adds to the wealth of the Dominion. Very extensive sources of petroleum have also been found in the North-West and in the Rocky Mountains.

Salt is found in the North-West; but the principal deposits are at Goderich and other places in Ontario, where there are extensive salt works. Salt is also found in New Brunswick.

Building stone of excellent quality is widely distributed in all the provinces, and there are numerous quarries of dolomite, limestone, sandstone, and granite, from which blocks of every size required can be obtained. There is also a great variety of marbles, but only a few quarries have yet been opened.

Gypsum is found in great abundance in New Brunswick, Nova Scotia, and Ontario. It occurs also in Manitoba and the territories. It may be procured at a cheap rate in any of the older provinces as a fertiliser.

Excellent peat is found in large deposits in all the provinces.—*Ibid.*

#### COPPER IN CANADA.

The copper deposits in Eastern Quebec, operated for many years, are those known as the Capelton mines. They are situated about seven miles south of Sherbrooke, the principal city of Quebec east of the St. Lawrence. At the time of the great copper boom, a number of mines were opened in this section. While at many of these places the veins promised well at the time, the gradual decline in the price of copper, and the generally low grade of the ore, rarely yielding more than 4 to 7 per cent., resulted in a gradual suspension of most of these enterprises. At the present time, while prospecting is again being resumed with some vigour, mining is confined almost entirely to two locations at Capelton, viz., the Nichols and the Eustis mines. The continued success of these workings has been due to several causes. One, probably, is the great size of the vein, which is stated to reach, in places, a thickness of 45 to 60 feet, and has been worked to a depth of over 1,500 feet. A chief cause, however, is that these ores are rich in sulphur, the bulk of the raw ore being used, first of all, for the manufacture of sulphuric acid, the residue being subsequently utilised for the extraction of the copper. A certain percentage of silver in the ore is also, doubtless, a very important factor as regards their present successful working, the amount at times ranging as high as 10 to 12 oz. to the ton, and probably even higher, though probably 3 to 4 oz. represents the average. The output of ore from these mines is large, ranging from 30,000 to 40,000 tons per year for each. While the greater part of this product is shipped in the raw state, smelting works, recently repaired by the Eustis Company, now dispose of over 1,000 tons per month, which is reduced to matte on the spot. At the Nichols mine adjoining, a correspondingly large amount is used for the manufacture of acid. A portion of the latter, with the phosphate of the Ottawa Valley, is converted into

superphosphate of lime. This industry, in the not distant future, bids fair to assume very large proportions.—*Iron-monger.*

#### EXPORTS OF CHEMICALS FROM THE TYNE FOR THE LAST THREE YEARS.

	1889.	1888.	1887.
	Tons.	Tons.	Tons.
Alkali and soda ash .....	17,632	27,054	35,112
Alum and alum cake .....	3,017	3,936	2,830
Ammonia .....	1,551	1,067	553
Arsenic .....	191	156	126
Bicarbonate of soda .....	852	1,294	2,262
Bleaching-powder .....	26,953	29,712	29,724
Caustic soda .....	16,091	12,238	4,525
Hydrate of strontia .....	1,208	431	4,608
Hypsulphite of soda .....	874	662	441
Manganate of soda .....	1,819	1,757	1,285
Manure .....	21,279	22,531	22,907
Magnesia .....	264	229	199
Pearl hardening .....	1,748	883	1,039
Soda crystals .....	81,891	100,115	99,471
Sulphate of soda .....	1,041	927	3,489
Other chemicals .....	1,730	1,278	271
Total .....	178,141	204,270	260,041

—*Chemist and Druggist.*

#### DISCOVERY OF BRINE AT MIDDLEWICH.

It is reported that a large quantity of brine has been discovered at the shaft which has been recently sunk on Newton Farm, Middlewich. The brine, it is said, has risen in the shaft to a height of over 70 yards, the strength being 2 lb. 10 oz. of salt to the gallon. The total depth of the shaft is about 100 yards. The first bed of salt is 60 feet thick, and 12 yards below this a second bed has been discovered, yielding at a depth of 56 feet a very fine quality of amber rock-salt. The discovery has caused some excitement in the salt trade.—*Ibid.*

#### A CHANCE SULPHUR PROCESS COMBINATION.

The alkali makers, who are preparing to recover sulphur by the Chance process, met at Liverpool lately for the purpose of devising a scheme for selling the whole of their produce, so far as it is intended to be placed on the open market, through a single medium and at a uniform price. In view of the already very depressed condition of the sulphur market, such a combination alone can prevent the manufacture under the Chance process from becoming absolutely unremunerative, as, apart from that process, there is already a much greater quantity of sulphur offering on the market than can be assimilated. Mr. Chance, the patentee of the new process, presided over the meeting, and after some discussion it was decided to form a limited company expressly for the sale of the product. It is thought that the quantity of Chance sulphur available for the market during the first year will represent a value of between 40,000*l.* and 50,000*l.*, the bulk of which will be manufactured at Widnes and St. Helens. Four-fifths of the producers were represented at the meeting.—*Ibid.*

#### SOAP IN INDIA.

There is an ample demand for the increased use of soap in India, for at present, after allowing for local manufacture, it may be said of the people of India that soap is to them

an unknown luxury, the consumption being at the rate of less than a shilling's worth for every hundred inhabitants a year.

The imports of soap have, it is true, more than doubled during the last six years, and the trade is steadily increasing from year to year. It is not anything like a large trade even now, for the largest quantity yet imported, that of the year ending April 1889, reached only 71,000 cwt., the value of which was 102,289*l.* The bulk of this was from England, the other European States supplying only a little over 3,000 cwt. The soap factories at Bombay, Jeypore, and Meerut are doing well and increasing their out-turn, and the local demand will most probably now go on increasing from year to year.

The soap manufactured by these companies is much liked by the natives, and particularly that variety called "vegetable" soap is in much request. Hindus of the orthodox type would not touch a soap made of tallow or animal fat, as it is against the principles of their religion to do so. Such men and women in general, therefore, did not use soap at all, and contented themselves by cleaning their hands with simple earth, or the soap-nuts of species of *sapindus* and the legumes of *Acacia concinna*. Since the production of the vegetable soap the objection to the introduction of that article in the native Hindu household is overcome, and soap is beginning to replace the primitive clay and vegetable substances used. About 8,000 cwt. of native-made soap is now exported annually.

The imports of soap of all kinds into British India have been as follows in the last six years:—

	Cwt.
1883—84.....	31,447
1884—85.....	38,975
1885—86.....	49,804
1886—87.....	59,016
1887—88.....	61,139
1888—89.....	71,072

The imports since this have, however, been declining. Of 500,000 cwt. of British soap which we exported in 1888, 75,275 cwt. went to India.—*Journal of the Society of Arts.*

#### A LARGE SALT FIELD.

At the annual meeting of the Stockton-on-Tees Chamber of Commerce, the president stated that the Teeside salt field covered an area of 11 square miles, averaging 70 feet in thickness, although in some places it was as thick as 100 feet. Each acre contained about 200,000 tons of salt, and the district contained enough to supply the whole world for 800 years.—*Industries.*

#### EFFECTS OF THE LATE STRIKES.

The effect of the strikes of the English colliers and of the Liverpool dock labourers has been greatly to reduce the supplies to the United States of alkali products, soda ash, caustic soda, bleaching powder, bicarbonate of soda, and allied chemicals used by the makers of glass and paper. The prices of these articles have much increased, and some have even more than doubled in cost. The result is that trade has been much disturbed, and the establishment of home factories for these materials is projected. The increased cost of camphor is attributed, not to the shortness of the crop, but to the enormous consumption in France and Germany for smokeless powder.—*Standard.*

#### A LARGE MANGANATE OF SODA CONTRACT.

At the weekly meeting of the London County Council, held at the Guildhall on March 18, the seal of the Council was, on the recommendation of the Main Drainage Committee, affixed to a contract with Messrs. C. Tennant, Sons, and Co., for the supply of 1,200 tons of manganate of soda at 12*l.* per ton; and a contract with the Widnes Alkali Company for the supply of 800 tons at 12*l.* per ton.—*Chemist and Druggist.*

#### THE OPIUM CROP IN THE TURKISH EMPIRE.

The following statistics give the production of opium in the whole of the Turkish Empire in Asia and Europe during

the last 26 years, together with the highest and the lowest prices of opium (in Turkish and British currency) on the Smyrna market during the seasons in question. The figures show that since 1864 there have been 128,400 baskets of opium produced in the Turkish Empire, the Persian production not being included in the returns. Deducting from this total the stock of opium now believed to be in existence, viz., 4,800 baskets (1,400 baskets in Smyrna, 700 in Constantinople, 1,700 in London, and 1,000 in America, &c.), we have an average annual consumption, during the last 26 years, of about 4,750 baskets of opium.

The comparatively large crops obtained since 1868 are explained by the fact that until that season the cultivation of the poppy was restricted to four or five provinces in Anatolia (Konieh, Karahissar, Sourahan, Bogaditch, and the neighbourhood of Smyrna), but after 1869, when the failure of the crop had caused an immense advance in the price of the drug, the cultivation was extended to other parts of the Empire, over the whole of Northern Anatolia, and in Roumelia. Since that period the peasants became accustomed to the cultivation of the poppy, and the prices thenceforth became dependent only upon the greater or smaller yield of the crop. It is said that the future of the opium industry in Turkey is becoming more uncertain. The extension of the railway system especially operates against the continuation of opium cultivation, because it encourages the growing of cereals, which require less attention, are less easily destroyed by inclement weather, and for which the demand is not so much influenced by speculation:—

Year.	Yield of Crop.	The Smyrna Prices during the Season.			
		Highest.		Lowest.	
	Baskets.	Piast.	s. d.	Piast.	s. d.
1864	3,400	140	16 6	111	13 0
1865	4,350	130	15 2	94	11 0
1866	1,350	160	18 8	122	14 0
1867	3,000	170	19 10	120	14 0
1868	1,600	390	45 6	146	17 0
1869	3,500	295	34 5	180	21 0
1870	4,300	270	31 6	130	15 0
1871	8,500	200	23 4	130	15 0
1872	4,400	220	25 8	170	19 1
1873	3,150	260	30 4	165	19 0
1874	2,430	274	32 0	130	15 0
1875	6,390	145	16 11	122	14 0
1876	3,250	190	21 6	137	15 0
1877	9,450	138	15 8	122	13 10
1878	6,050	145	16 5	120	13 7
1879	4,300	250	28 4	135	15 3
1880	2,100	250	28 4	135	15 3
1881	11,000	135	15 3	103	11 8
1882	4,500	135	15 3	90	10 2
1883	6,970	115	12 10	85	9 6
1884	5,400	115	12 10	90	10 0
1885	7,400	95	10 7	80	8 11
1886	7,600	164	18 0	63	6 11
1887	1,800	189	19 10	71	7 10
1888	7,300	100	11 0	62	6 10
1889	5,000	..	..	..	..

—*Ibid.*

## OPIUM IN BRITISH INDIA.

Under an agreement with the administration of the opium department of the Bombay Presidency, and with the view of effectually stopping the contraband traffic in opium, the rulers of all native States, except Baroda, have consented to entirely prohibit the cultivation of the poppy in their dominions. Baroda has been permitted to grow and manufacture opium, under a State monopoly, for consumption in its own territory, and for export to China. The State has undertaken to assimilate its opium retail system and selling prices to those for the time being in force in adjoining British districts, and to prevent the introduction of Baroda opium into British territory, and into the territories of adjoining native States.—*Ibid.*

## OPENING OF SULPHURIC ACID WORKS.

The long-closed sulphur works at Tauranga, 100 miles south of Auckland, are about to be opened, and utilised in the manufacture of sulphuric acid, by Messrs. Sharland and Co., of Auckland. The works are now being put in order and are expected to be in operation early next month. On a small islet called White Islet, some miles off the coast, there is an inexhaustible supply of crude sulphur in a high degree of purity, but owing to the lessee and part owner of the island refusing to allow it to be worked, this source is not available; but at Matata, some 50 miles south of Tauranga, there is another enormous deposit of sulphur. Other localities, too, not far removed, are rich in this element, and the proprietors of the works will have no difficulty in obtaining supplies.—*Chem. Trade Journal.*

## FRENCH COAL PRODUCTION IN 1889.

Comparative tables relating to the coal production in France have recently been prepared and issued by the French Government. From these tables it appears that the total production of coal in France in 1889 amounted to 24,588,880 tons, as compared with 22,602,894 tons in 1888, an increase of 1,985,986 tons. The largest quantity came from the Department of the Pas de Calais, namely, 8,624,837 tons, while the Nord furnished 4,809,065 tons, as compared with 4,416,185 tons in the preceding year. The Loire comes next with 3,378,138 tons, the Gard with 1,976,511 tons, and the Saône et Loire with 1,491,279 tons. In the Departments of the Pas de Calais and the Nord, the largest quantity of coal extracted came from the mines of the Anzin Companies, the amount being 2,857,613 tons; the Lens et Douvriin Company comes next with 1,667,398 tons, and the Courrières Company with 1,188,635, the increase in the production in 1889 over that of the preceding year being 262,032, 255,747, and 95,435 tons respectively. The increase in the total production of these two departments amounts to 1,057,413 tons, the actual amounts being 13,433,847 tons in 1889, against 12,376,434 tons in 1888.—*J. Soc. Arts*, 1890, 443.

## PRODUCTION OF METALS IN SWEDEN DURING 1888.

Note.—One Swedish cwt. = 93·5 lb.

		1887.	1888.
Iron ore rock .....	Sw. Cwt.	21,224,753	22,498,500
„ from bays, &c. ....	„	22,888	74,887
Pig iron .....	„	10,624,921	10,645,672
Castware for blast furnaces ..	„	117,284	106,563
„ pig iron.....	„	568,348	664,643
Blooms .....	„	..	5,602,108
Bar iron .....	„	6,007,936	5,954,000
Bessemer steel.....	„	1,604,395	1,614,306
Martin steel .....	„	985,659	1,051,684
Steel, other kinds.....	„	34,542	28,357

PRODUCTION OF METALS IN SWEDEN DURING 1888—*cont.*

		1887.	1888.
Manufactured iron and steel .....	Sw. Cwt.	1,158,632	1,403,993
Gold ore .....	„	33,018	21,878
Silver and lead ore.....	„	367,374	274,778
Gold.....	Sw. Lb.	196	178
Silver.....	„	13,724	10,926
Copper ore.....	Sw. Cwt.	485,869	470,990
Copper .....	„	21,634	24,705
Nickel ore .....	„	2,000	22,805
Brass .....	„	6,381	5,518
Hammered copper, &c.....	„	6,084	6,570
Castware, other than iron ..	„	1,306	1,584
Lead.....	„	6,632	7,711
Zinc ore .....	„	1,087,836	1,175,399
Cobalt ore .....	„	5,440	3,374
Refined cobalt .....	Sw. Lb.	885	760
Oxide of copper .....	Sw. Cwt.	95	77
Manganese ore .....	„	203,706	227,950
Pulverised manganese ....	„	2,843	6,428
Pyrites .....	„	58,964	31,359
Sulphur .....	„	1,276	1,517
Sulphuric acid .....	„	..	40,579
Sulphate of copper.....	„	3,721	4,627
„ iron .....	„	9,111	12,062
Red ochre .....	„	32,474	29,093
Alum .....	„	3,577	15,414
Cerite .....	„	917	654
Lead ore .....	„	621	475
Marble, value in .....	Kr.	9,800	5,000
Coal .....	Cub. Ft.	8,072,182	8,071,654

## THE SUGAR PRODUCTIONS OF THE WORLD.

An extremely able and interesting report on the "Progress of the Sugar Trade," by Robert Giffen, Esq., LL.D., Assistant Secretary, Commercial Department, Board of Trade, was presented to Parliament in May of last year, and issued to the public in the month of June following. For an early copy of it this establishment was indebted to the Board of Trade. The whole document deserves the most careful study, but it is unlikely in its complete form to circulate to any large extent in the colonies. It seems desirable, therefore, to extract some passages which contain information in a very condensed form on the production, consumption, and, to some extent, distribution of sugar.

There is some risk, no doubt, that by detaching passages from a document of this kind a different impression may be produced to that which would be derived from the whole. The passages now reproduced deal, however, with statistical matters of fact, which are not affected whatever view is taken of their significance.

Taking the period 1853—87, the production of cane sugar has more than doubled, and British and foreign cane sugars

have preserved about the same proportion. In 1887 British cane sugar formed about one-fifth of the whole production of cane sugar and about one-ninth of the total sugar production of the world. The development of the beet sugar industry is brought out in the most remarkable way. At the beginning of the period it formed only one-eighth of the total sugar production; at the end it amounted to nearly a half.

Taking the West Indies and British Guiana together, it appears that since 1883—85 the production of cane sugar has pretty well held its own; but if the production of the West Indies be taken separately, it "has not maintained itself so well."

Turning now to consumption, it appears that there has been a falling off, during the period 1877—88, in the importation of raw cane sugar into the United Kingdom from British possessions, "particularly the West Indies," of nearly  $2\frac{1}{2}$  million cwt., the greater part in the last five years. Foreign raw cane sugar over the same period ends with a slight decrease. On the other hand, the West Indies have found a market elsewhere, particularly in the United States. The importation of beetroot sugar during the same period has more than doubled. And it is important to notice that during the last five years, while the import of raw beet sugar showed little increase, that "of refined in the same period has been over three million cwt., or as much as the total imports of refined from beet countries five years ago."

The production of cane sugar in British possessions show "that that production does not find an outlet in the market of the United Kingdom, as it formerly did, its place being taken by beet sugar."

The figures with regard to home consumption are very striking. "The 8 million cwt. consumed 30 to 35 years ago, inclusive of duty, were as costly to the consumers of the United Kingdom as the 22 million cwt. annually consumed in the last two or three years."

In the June Bulletin for 1887 the position of the sugar question in the West Indies was briefly touched upon. There is reason to think that to some extent sugar has slightly gone out of cultivation. When this has happened the particular loss is irreparable. The necessity of keeping the land clean and in tillage, and the machinery in working order, is so paramount that there can be no doubt a sugar planter will strain his pecuniary resources to the uttermost before throwing his estate out of cultivation, because once a sugar planter is compelled to suspend operations, even for a short period, the capital invested is lost beyond hope of recovery. The usual sequence of events, when an estate is abandoned in the West Indies, is that the workpeople are scattered and leave the neighbourhood, the land becomes foul, the buildings ruinous, the working oxen are sold, necessarily at the loss incidental to a forced sale, and the machinery, in spite of every precaution to keep it in order, becomes rusty and useless. Hence it will be no matter of surprise to learn that it is impossible to resume operations

on an abandoned sugar estate in the tropics, except at a cost almost equal to that of starting an entirely new undertaking.

If, therefore, the figures supplied by the Board of Trade do not justify a gloomy view of the present position of the cane sugar industry in British colonies, they scarcely justify a very optimistic one. It is obvious that the capital which should be applied to the improvement of manufacturing processes and machinery is, under present circumstances, practically diverted to the mere maintenance of the cultivation. And this in the long run must be a losing game. At present the fact stands that West Indian sugar has to a large extent been driven from the home market to that of the United States. If in time it should lose that, its fate apparently is sealed.

## I.

## TOTAL PRODUCTION OF DIFFERENT KINDS OF SUGAR, 1853—87.

	1853—55.	1886—87.	Increase.
	Tons.	Tons.	Tons.
" British cane sugar.....	261,000	580,000	319,000
Foreign cane sugar.....	972,000	2,174,000	1,202,000
	1,233,000	2,754,000	1,521,000
Beetroot sugar .....	190,000	2,433,000	2,243,000
Total .....	1,423,000	5,187,000	3,764,000

"Out of a total increase of production amounting to  $3\frac{3}{4}$  million tons, no less than  $2\frac{1}{2}$  million tons, or about 60 per cent., is an increase of beet sugar, which has in fact changed its position as a factor in the production altogether, having now reached the point of being nearly equal to cane sugar, whereas at the beginning of the period it supplied only about one-eighth of the total production. Both British cane sugar and foreign cane sugar have more than doubled in the same period, and the increase in the two together amounts to  $1\frac{1}{2}$  million tons, or 40 per cent. of the total increase of  $3\frac{3}{4}$  million tons, and this increase also is obviously a very large one. It hardly compares, however, with the increase in beet sugar, which is, to a large extent, a new development altogether. Of course this table is subject to the observation that the figures in the last two or three years are specially increased by the inclusion of one or two countries formerly omitted, but a correction on this account can easily be made if thought necessary in a comparison extending over so long a period.

"This is the aspect of the progress when the beginning and end of the whole period, 1853—87, are compared. In the interval of five years, between 1880—82 and 1886—87, the figures are:—

## "INCREASE OF PRODUCTION OF DIFFERENT KINDS OF SUGAR, 1880—87.

	1880—82.	1886—87.	Increase.	1886—87 (deducting Countries included since 1884).	Increase.
	Tons.	Tons.	Tons.	Tons.	Tons.
" British cane sugar .....	419,000	580,000	161,000	531,000	112,000
Foreign cane sugar .....	1,190,000	2,174,000	975,000	1,973,000	474,000
	1,918,000	2,754,000	836,000	2,504,000	586,000
Beetroot sugar .....	1,616,000	2,433,000	787,000	2,433,000	787,000
Total.....	3,564,000	5,187,000	1,623,000	4,937,000	1,373,000

"Thus the increase in the most recent period, keeping strictly to the basis before 1884, amounts to 1,373,000 tons, of which three-sevenths may be credited to cane sugar, and four-sevenths to beet sugar. The progress of beet sugar

is thus in amount as great as ever, though the percentage increase is not quite so great as in the early part of the period since 1853. The most remarkable part of the increase of beet was, no doubt, prior to 1882.



"It will be noticed also that after about 1870 the proportion of British cane sugar in the total production was maintained steadily at about 12 per cent. until about two years ago, so that the great growth in the proportion of beet in the total production was obtained in that period at the expense of foreign cane sugar, which, in fact, declined from 54 to 39 per cent., while beet rose from 34 to 49 per cent.; but in the latest two or three years the proportion of British cane sugar in the total has been barely maintained, having fallen to 11 per cent., while foreign cane sugar has recovered a little, and beet has fallen back a little. The proportion of British cane sugar was also much higher in the earlier periods than it has since been, ranging then between 17 and 20 per cent.

"Still, at no period has there been anything but an increase in the growth of British cane sugar, though it has not kept pace with the growth of beet sugar, and in the last year or two with the growth of foreign cane sugar."

## II.

### PRODUCTION IN THE WEST INDIES.

"The average annual exports of the West Indian colonies (including British Guiana), which have always been specially interesting in this question of production, have been as follows:—

1877—79	.....	5,203,000 cwt. = 26,000 tons.
1880—82	.....	5,548,000 " = 277,000 "
1883—85	.....	6,062,000 " = 303,000 "
1886—87	.....	5,920,000 " = 296,000 "

"Since 1883—85 the West Indies have thus held their own, the difference between 303,000 tons in 1883—85 and 296,000

tons in 1886—87 being nominal only; but they have not more than held their own in these most recent years, although there is still an increase since 1880—82.

"It will be seen, moreover, from the summary in the appendix, that the production in the West Indies, exclusive of British Guiana, has not maintained itself so well on the average of years as the production in British Guiana. The production in both cases in 1887 is about the largest on record; but the average of the West Indies, excluding British Guiana, for the two years 1886—87, is brought down by an extremely low export figure in 1886."

## III.

### DISPLACEMENT OF CANE BY BEET SUGAR.

"The great increase of the production of cane sugar, again, has been an increase for consumption in extra-continental countries, principally the United States and the United Kingdom; but a certain surplus of the beet production has also overflowed into those extra-continental countries, principally the United Kingdom. This has been done, however, without diminishing the consumption of cane sugar in those countries taken altogether, although the United Kingdom in recent years has come more and more to rely largely on beet sugar, and the supply from certain cane sugar districts, particularly the West Indies, has of late rapidly diminished, these districts in turn having found a market elsewhere, particularly in the United States, for their increased production. This disposal of the overflow of beet sugar, and partial displacement of cane sugar in its proportionate importance in the market of the United Kingdom, are points of special interest . . . which may be further illustrated."

### ANNUAL IMPORTS OF SUGAR INTO THE UNITED KINGDOM, DISTINGUISHING CANE FROM BEETROOT SUGAR, &c.

	1877—78.	1882—83.	1886—88.	Increase or Decrease in 1882—83 from 1877—78.	Increase or Decrease in 1886—88 from 1882—83.
	Cwt.	Cwt.	Cwt.	Cwt.	Cwt.
Raw cane sugar from British possessions . . . .	3,450,000	5,193,000	3,292,000	— 282,000	— 1,876,000
From foreign countries . . . . .	7,100,000	8,973,000	6,585,000	+ 973,000	— 1,488,000
Total . . . . .	12,550,000	13,241,000	9,877,000	+ 691,000	— 3,364,000
Raw beetroot sugar . . . . .	3,220,000	6,891,000	7,456,000	+ 3,641,000	+ 565,000
Refined cane sugar . . . . .	215,000	125,000	718,000	— 90,000	+ 623,000
Refined beetroot sugar, i.e., refined sugar from beet-growing countries . . . . .	3,182,000	2,900,000	5,993,000	— 232,000	+ 3,093,000
Total . . . . .	19,117,000	23,127,000	24,080,000	+ 4,010,000	+ 953,000

"Thus, beetroot, though it increased largely in the five years ending 1882—83, did not gain at the expense of cane sugar, but since the date mentioned the gain has been largely at the expense of cane sugar, both in amount and proportion, and in the whole 10 years since 1878 there is a loss of cane both in amount and proportion, and a gain of beet. The imports of raw cane sugar from British possessions alone have diminished in the 10 years from nearly 5½ million cwt. to about 3½ million cwt., or a diminution of 2½ million cwt., the greater part in the last five years. At the same time the imports of raw cane sugar from foreign countries, which increased nearly 1 million cwt. in the first five years, show a falling off of about 1½ million cwt. in the subsequent five years, making a small net balance of decrease in the 10 years. Sugar from beet countries, on the other hand, has more than doubled in amount in the 10 years, the increase being from 6,350,000 cwt. raw and refined together in 1877—78, to 13,455,000 cwt. in 1886—88. There is a notable difference, moreover, in the apportionment of this increase in the two quinquennial periods. Between 1878 and 1882 the increase

of the imports from beet countries was of raw sugar, the imports of refined rather diminishing. Since 1882 the increase of the imports has only been to a small extent in raw sugar, and the increase of refined in the same period has been over 3,000,000 cwt., or as much as the total imports of refined from beet countries five years ago.

## IV.

### CHANGE OF MARKET FOR BRITISH CANE SUGAR.

"The prominent fact . . . . . is the changed amount and proportion of the imports of cane sugar from British possessions. Writing in 1884 . . . . . I pointed out that while the amount of the imports from British possessions was maintained at nearly the high figures of 1853 and subsequently, yet the *proportion* of these imports to the total had steadily declined, owing to the large growth of imports from other countries, especially beet countries. In the last five years a still greater change has taken place. The imports from British possessions have greatly diminished,

from nearly 5,000,000 cwt. in 1882, to just over 3,000,000 cwt., and the proportion, which had steadily fallen from 65 per cent. in 1853—55, to 23 per cent. in 1880—82, has since farther fallen to 13·7 per cent. only. These are not the figures of production in British possessions, which have already been dealt with, and which have not diminished, but they show that that production does not find an outlet in the market of the United Kingdom, as it formerly did, its place being there taken by beet sugar."

## V.

## INCREASE OF HOME CONSUMPTION AT DIMINISHED COST.

"The net effect, however, is that for a good many years the great increase in the quantities of sugar consumed in the United Kingdom has been accompanied by an actual decrease in the aggregate sum spent upon it by consumers in the United Kingdom. The 1,100,000 tons and upwards now consumed annually cost only in the last two years about 16,500,000*l.* annually, whereas the 900,000 to 1,000,000 tons consumed seven or eight years ago cost from 20,000,000*l.* to 24,000,000*l.* The comparison is even more striking if we go farther back. The 8,000,000 cwt. consumed 30 to 35 years ago, inclusive of duty, were as costly to the consumers of the United Kingdom as the 22,000,000 cwt. annually consumed in the last two or three years. Of course this is again subject to the above qualification as to final consumption by the people of the United Kingdom, but the reduction of price is also an important matter for the manufacturing industries in which the sugar is used."—*New Bulletin.*

## BOARD OF TRADE RETURNS.

## SUMMARY OF IMPORTS.

	Month ended 28th February	
	1889.	1890.
	£	£
Metals.....	1,988,850	1,918,364
Chemicals and dyestuffs.....	955,619	873,097
Oils.....	536,295	442,051
Raw materials for non-textile industries.....	2,353,142	2,119,543
Total value of all imports ....	32,340,096	31,018,842

## SUMMARY OF EXPORTS.

	Month ended 28th February	
	1889.	1890.
	£	£
Metals (other than machinery) ....	2,674,084	3,580,286
Chemicals and medicines .....	603,427	725,887
Miscellaneous articles.....	2,516,060	2,672,701
Total value of all exports.....	18,670,352	21,084,228

## IMPORTS OF METALS FOR MONTH ENDED 28TH FEBRUARY.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Copper:—			£	£
Ore..... Tons	10,031	7,901	78,640	51,638
Regulus .....	11,156	8,834	415,721	243,358
Unwrought ....	4,000	2,175	323,342	111,833
Iron and steel:—				
Iron ore .....	365,711	483,907	274,391	394,230
Iron bolt, bar, &c. ..	4,677	5,048	44,436	46,440
Steel, unwrought..	557	450	5,487	5,349
Lead, pig and sheet ..	9,423	12,952	122,377	168,671
Pyrites .....	67,327	48,248	130,671	89,156
Quicksilver .....	640,962	888,975	69,741	114,749
Tin .....	42,322	65,015	202,308	302,126
Zinc .....	3,661	5,121	68,146	116,339
Other articles ... Value £	..	..	253,590	274,476
Total value of metals	..	..	1,988,850	1,918,364

## IMPORTS OF CHEMICALS AND DYESTUFFS FOR MONTH ENDED 28TH FEBRUARY.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
			£	£
Alkali..... Cwt.	347	2,447	1,205	1,672
Bark (for tanners, &c.) .....	21,454	19,471	7,007	5,863
Brimstone .....	116,992	86,422	24,182	18,057
Chemicals..... Value £	..	..	103,168	88,866
Cochineal .....	859	1,222	5,076	8,432
Cutch and gambier Tons	1,580	2,615	41,724	71,451
Dyes:—				
Anilino .....	..	..	19,132	24,703
Alizarine .....	..	..	24,697	26,795
Other .....	..	..	1,600	2,026
Indigo .....	19,395	19,628	425,658	371,603
Madder .....	990	846	1,222	1,046
Nitrate of soda....	155,300	104,219	85,015	38,436
Nitrate of potash .	25,846	26,850	23,279	23,953
Valonia .....	3,110	1,932	43,849	38,306
Other articles... Value £	..	..	148,825	151,888
Total value of chemicals	..	..	955,619	873,097

## IMPORTS OF OILS FOR MONTH ENDED 28TH FEBRUARY.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
			£	£
Cocoa-nut..... Cwt.	21,335	10,204	27,899	12,056
Olive ..... Tuns	1,814	2,187	63,889	83,638
Palm ..... Cwt.	60,304	55,190	65,531	57,049
Petroleum ..... Gall.	8,616,780	5,552,607	225,761	136,748
Seed ..... Tons	912	1,440	25,455	40,503
Train, &c..... Tuns	1,101	438	23,668	9,122
Turpentine ..... Cwt.	27,313	13,887	47,968	21,986
Other articles .. Value £	..	..	55,968	80,859
Total value of oils ...	..	..	336,295	442,051

## IMPORTS OF RAW MATERIALS FOR NON-TEXTILE INDUSTRIES FOR MONTH ENDED 28TH FEBRUARY.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
			£	£
Bark, Peruvian .. Cwt.	12,040	8,570	41,303	31,815
Bristles..... Lb.	193,934	134,428	21,898	23,067
Caoutchouc..... Cwt.	37,731	22,978	489,400	304,525
Gum :—				
Arabic..... "	5,822	3,772	20,246	14,827
Lac, &c..... "	5,058	6,248	17,619	22,929
Gutta-percha .... "	1,813	7,216	20,918	88,841
Hides, raw :—				
Dry..... "	48,780	31,142	130,584	90,156
Wet..... "	42,206	35,134	101,899	80,005
Ivory ..... "	437	888	21,823	47,304
Manures :—				
Guano..... Tons	4,516	202	27,768	1,461
Bones..... "	4,826	7,281	23,129	37,257
Paraffin..... Cwt.	25,332	25,254	30,791	32,245
Linen rags..... Tons	3,009	3,071	30,816	31,655
Esparto, &c. .... "	15,304	18,819	81,467	94,102
Pulp of wood .... "	9,410	8,813	52,819	48,077
Rosin..... Cwt.	160,600	57,174	30,334	9,847
Tallow and stearin .. "	80,489	58,871	118,181	73,490
Tar ..... Barrels	2,633	3,693	1,733	2,059
Wood :—				
Hewn ..... Loads	78,896	118,667	147,575	200,133
Sawn ..... "	57,946	58,340	150,007	155,505
Staves ..... "	4,468	2,953	23,526	13,489
Mahogany ..... Tons	3,979	296	34,289	2,988
Other articles.... Value £	..	..	744,017	716,766
Total value .....	..	..	2,353,142	2,119,513

Besides the above, drugs to the value of 75,117, were imported, as against 89,882, in 1889.

## EXPORTS OF METALS OTHER THAN MACHINERY FOR MONTH ENDED 28TH FEBRUARY.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
			£	£
Brass..... Cwt.	5,405	8,670	28,505	37,279
Copper :—				
Unwrought ..... "	10,104	85,139	30,423	229,406
Wrought..... "	8,321	30,033	38,269	92,269
Mixed metal .... "	13,509	31,773	46,609	87,435
Hardware ..... Value £	..	..	237,328	212,430
Implements..... "	..	..	74,496	105,737
Iron and steel..... Tons	262,278	277,062	1,962,958	2,392,042
Lead ..... "	4,860	4,878	71,934	72,288
Plated wares... Value £	..	..	28,261	27,278
Telegraph wires... "	..	..	32,086	201,594
Tin ..... Cwt.	7,880	6,499	39,191	32,011
Zinc ..... "	14,387	11,857	10,007	11,097
Other articles .. Value £	..	..	64,567	78,420
Total value .....	..	..	2,674,084	3,580,286

## EXPORTS OF DRUGS AND CHEMICALS FOR MONTH ENDED 28TH FEBRUARY.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
			£	£
Alkali..... Cwt.	440,985	472,784	115,934	144,640
Bleaching materials .. "	120,060	147,046	49,619	43,173
Chemical manures. Tons	25,220	26,168	188,048	212,398
Medicines..... Value £	..	..	67,934	93,309
Other articles ...	..	..	184,892	232,367
Total value .....	..	..	603,427	725,887

## EXPORTS OF MISCELLANEOUS ARTICLES FOR MONTH ENDED 28TH FEBRUARY.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
			£	£
Gunpowder..... Lb.	1,179,800	768,290	188,048	212,398
Military stores.. Value £	..	..	76,665	113,851
Candles..... Lb.	822,400	1,165,000	16,386	21,455
Caoutchouc ..... Value £	..	..	82,355	85,224
Cement..... Tons	48,359	50,880	92,279	101,332

EXPORTS OF MISCELLANEOUS ARTICLES FOR MONTH  
ENDED 28TH FEBRUARY—*cont.*

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
			£	£
Products of coal Value £	..	..	72,630	109,674
Earthenware ... ..	..	..	146,675	161,611
Stoneware ..... ..	..	..	12,845	12,610
Glass:—				
Plate..... Sq. Ft.	287,773	271,265	16,743	15,166
Flint..... Cwt.	9,312	8,181	20,374	19,198
Bottles..... "	61,778	74,356	28,802	34,924
Other kinds.... "	15,394	13,390	11,754	11,858
Leather:—				
Unwrought .... "	9,729	12,797	93,683	118,514
Wrought ..... Value £	..	..	40,380	28,681
Seed oil..... Tons	5,798	5,855	123,203	129,067
Floor cloth ..... Sq. Yds.	1,502,400	1,570,000	68,818	73,201
Painters' mate- rials ..... Value £	..	..	116,057	127,431
Paper ..... Cwt.	78,214	80,017	138,176	139,474
Rags..... Tons	5,071	4,548	40,692	32,955
Soap ..... Cwt.	31,903	33,501	34,430	35,881
Total value .....	..	..	2,516,060	2,672,701

QUICKSILVER.

	IMPORTS.			
	1890.	1889.	1888.	1887.
	Bottles.	Bottles.	Bottles.	Bottles.
February .....	11,853	8,546	10,758	9,641
Jan.—Feb. ....	23,069	15,798	22,198	19,045
	EXPORTS.			
	1890.	1889.	1888.	1887.
February .....	4,478	4,180	1,576	3,222
Jan.—Feb. ....	10,390	8,973	3,757	9,206
Average Price:	£ s. d.	£ s. d.	£ s. d.	£ s. d.
February .....	9 2 6	8 8 6	8 10 0	7 5 6

—*Alor. S. Pickering.*

## Monthly Patent List.

\* The dates given are the dates of the Official Journals in which acceptances of the Complete Specifications are advertised. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the said dates.

### I.—GENERAL PLANT, APPARATUS, AND MACHINERY.

#### APPLICATIONS.

2618. H. J. Haddan.—From S. T. Wellman, United States. An improved method of, or mechanism for, charging furnaces. Complete Specification. February 18.

2632. W. P. Thompson.—From A. Stehlik, Austria. Improvements in centrifugal apparatus for filtering water and other liquids. February 18.

2935. L. Vojacek. Improvements in apparatus for propelling and measuring fluids. February 24.

2961. F. E. Beeton. An improved apparatus for producing a liquid spray. February 25.

3017. H. C. Sergeant. Improvements in compressors. Complete Specification. February 25.

3186. J. Procter. Improvements in mechanical stokers for steam boiler furnaces. February 28.

3192. R. S. Baxter and G. D. Macdougland. Improvements in centrifugal machines. February 28.

3261. H. H. Lake.—From O. H. Jewell, United States. Improvements in apparatus for filtering water. Complete Specification. March 1.

3279. W. H. Skinner. Heating water or other liquids by a novel construction. March 1.

3804. Sir W. Thomson. Improvements in rheostats. March 11.

3834. R. A. Haggard. Improvements in apparatus for heating water and other liquids. March 11.

3907. R. Harvey. Improvements in and relating to evaporating apparatus. March 12.

3915. J. Schwager. Improvements in condensing apparatus for steam and other vapours. March 12.

4024. J. Foster. Improvements in and relating to evaporating and distilling apparatus. March 14.

#### COMPLETE SPECIFICATIONS ACCEPTED.\*

1889.

5156. J. W. Wailes. Liquid valves for producer and other gases and air. February 26.

5496. M. W. Household. Apparatus for raising or forcing hot or vaporous liquids. March 5.

5497. M. W. Household. Apparatus for raising or forcing hot liquids. March 5.

6102. P. Cornish, jun. Means for drying or desiccating substances or materials. March 19.

6770. H. Finch and J. Mackenzie. Metallic hampers or skips for earboys, &c. March 19.

6989. E. Edwards.—From G. Lunge and L. Rohrmann. Apparatus for treating gases by liquids. March 19.

7087. J. Hill. Presses for compressing semi-fluids into solids. March 12.

8342. G. Olberg. Rotating furnaces. March 5.

10,385. R. Fölsche. An improved centrifugal machine. March 12.

18,529. W. T. Walker. Stills or apparatus for submitting liquids to the action of vapours or gases. March 19.

1890.

1476. A. J. Boulton.—From G. Schweickhardt. Improvements in tuyeres. March 5.

1877. A. Riedler. Apparatus for compressing air or gases. March 12.

## II.—FUEL, GAS, AND LIGHT.

### APPLICATIONS.

2559. H. S. Maxim and G. S. Sedgewick. Improvements relating to the carburetting or enrichment of coal and other gas and to apparatus therefor. February 17.

2611. W. W. Popplewell.—From H. Conried, United States. New or improved artificial fuel. Complete Specification. February 18.

2745. A. Moseley. Preparing chalk or limestone or gypsum to be used as a flux for improving the combustion of coal and coke in domestic and other fires, and for reducing the amount of smoke emanating therefrom. February 20.

2746. A. Moseley. Improvements in the materials and preparation of compressed coal and coke block fuel. February 20.

2927. J. Petrie. Improvements in supplying air to the flues of steam boilers for the purposes of preventing smoke and for consuming the inflammable gases therein. February 24.

2978. C. H. Mowell. Improvements in the composition of artificial or block fuel. February 25.

3081. C. Kingsford. Improvements in apparatus for inserting and withdrawing the charges from gas retorts. February 26.

3160. B. von Steenbergh. Improvements relating to the manufacture of water-gas, and to apparatus therefor. February 27.

3306. H. Ekelund. A furnace for continuous carbonising or coking. Complete Specification. March 1.

3332. B. C. Sykes and G. Blamires. Improvements in separating hydrogen gas and the light carburets of hydrogen, such as methane, from water-gas or producer-gas, or from both of them when mixed together. March 3.

3363. C. Hencken. Improvements in coke ovens. March 3.

3415. J. Armour and H. Armour. Improvements in or connected with retorts for distilling shale or other oil-yielding minerals. March 4.

3684. T. Holgate. Improvements in the method of and apparatus employed in the manufacture of coal gas and of compounds of ammonia. March 8.

### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

3984. J. O. O'Brien.—From W. Underwood. Manufacture of blocks of fuel from granulated or pulverised coal and other materials. March 5.

4040. E. L. Stacey. An improved firelighter, and machinery for manufacturing same. March 5.

6747. W. Bliss and others. Burning gas tar or other liquid fuel in furnaces. March 5.

6917. J. N. Flesch. Rendering petroleum less explosive, and improving its illuminating power. March 5.

6937. C. J. Langley. Artificial fuel. February 26.

7424. A. M. Chambers and T. Smith. Improvements in coke ovens, and in methods and apparatus for collecting and utilising the products of combustion therefrom. March 12.

7741. G. W. Hart. Process and apparatus for manufacturing hydrocalcium gas. March 19.

7792. J. Broad and G. C. Fowler. Improvements in nightlights, &c. March 19.

7812. G. Rose and others. Method and apparatus for the manufacture of water-gas. March 19.

8263. S. B. Darwin. Improvements in compounds for manufacturing illuminating gas, the prevention of stoppages in ascension pipes, and the deposition of naphtholine. March 19.

10,829. B. von Steenbergh. Manufacture of gas and apparatus therefor. February 26.

19,449. G. Hargreaves and others. Manufacture of gas and apparatus therefor. March 5.

1890.

1698. C. Heyer. Carburetting gases, and purifying hydrocarbons for that purpose. March 5.

## III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

### APPLICATION.

2955. H. Prople. An improved process of and apparatus for the continuous distillation of tars or mineral oils, and their products of distillation. February 24.

### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

6726. N. McFarlane Henderson. Retorts for the destructive distillation of shale or other oil-yielding minerals. February 26.

7634. J. Armour and H. Armour. Retorts for distilling shale or other oil-yielding minerals. March 12.

## IV.—COLOURING MATTERS AND DYES.

### APPLICATIONS.

3095. H. H. Leigh.—From R. G. Williams, United States. Improvements in the manufacture of colouring matters. February 26.

3098. H. H. Lake.—From Messrs. Wirth and Co., Agents for A. Leonhardt and Co., Germany. Improvements in the manufacture of colouring matters. February 26.

3303. B. Willecox.—From The Farbenfabriken vormals F. Bayer and Co., Germany. Improvements in the manufacture of fast dyestuffs for printing and dyeing. March 1.

3397. B. Willecox.—From The Farbenfabriken vormals F. Bayer and Co., Germany. Improvements in the manufacture of alpha-naphthol sulpho acids and dioxynaphtholine sulpho acids, and of dyestuffs therefrom. March 4.

3398. B. Willecox.—From The Farbenfabriken vormals F. Bayer and Co., Germany. Improvements in the manufacture of mordant dyeing azo colouring matters of the triphenylmethane group. March 4.

3788. M. G. Lindemann. Improvements relating to the treatment of dyewoods and their extracts. March 10.

### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

4539. E. Eisenmann and A. Arche. Manufacture of picric acid. March 5.

5079. B. Willecox.—From The Farbenfabriken vormals F. Bayer and Co. Manufacture of periodides of phenols and salicylic acid. February 26.

6434. O. Imray.—From The Farbwerke vorm. Meister, Lucius, and Brünig. Production of an imide compound of fluoresceine chloride, and of colouring matters therefrom. March 5.

6777. J. E. Bedford. Manufacture of colouring matters. March 19.

6778. J. E. Bedford. Apparatus for the production of colouring matters. March 5.

7067. S. Pitt.—From L. Casella and Co. Manufacture of dyestuffs from oxyethers of alpha-naphthylamine and their sulpho acids. March 5.

7550. C. D. Abel.—From The Actiengesellschaft für Anilinfabrikation. Manufacture of green and violet colouring matters. February 26.

8264. H. H. Lake. — From Leonhardt and Co. Production of blue-black colouring matters. March 19.

12,560. S. Pitt. — From L. Casella and Co. Production of new red colouring matters. March 19.

1890.

2011. W. S. Conrad and H. T. Lilley. An improved copying ink. March 19.

## V.—TEXTILES, COTTON, WOOL, SILK, ETC.

### APPLICATIONS.

2827. A. J. Boulton. Improvements in or relating to the manufacture or treatment of textile or similar fabrics. February 21.

3261. A. A. Haigh. See Class XIX.

3749. H. Blakeley and C. Beving. A method of ornamenting textile fabrics. March 10.

3971. H. A. A. Dombraun. Improvements in cleansing wool, and apparatus therefor. March 13.

### COMPLETE SPECIFICATION ACCEPTED.

1889.

10,010. C. Pusch. Separating the wool from pieces of rabbit skins and other similar skins. March 19.

## VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

### APPLICATIONS.

3009. S. D. Keene. Improved method or process of, and apparatus for, bleaching, dyeing, or otherwise treating fibrous materials. February 25.

3626. W. Norton and F. Hinchliffe. An improved mode of, and apparatus for, dealing with warps and hanks whilst being put through dyeing. March 7.

3651. G. T. Teasdale-Buckell. Improvements in printing in colours on paper or other material, and in the means or apparatus employed therein. March 7.

3703. W. Norton and F. Hinchliffe. An improved mode of, and apparatus for, dealing with hanks whilst being put through dyeing processes. March 8.

### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

5590. E. Bentz, C. Edmeston, and others. Scouring and bleaching textile fabrics, warps, and yarns formed of cotton or other vegetable fibres, and apparatus therefor. March 5.

8237. T. Parkinson. Methods of drying cotton yarns, &c. in the process of mordanting, and apparatus therefor. February 26.

9432. G. A. Greeven. Impregnating, washing, and dyeing yarn in hanks or skeins, and apparatus therefor. March 12.

## VII.—ACIDS, ALKALIS, AND SALTS.

### APPLICATIONS.

2526. The Borax Co., Lim. A new method of treating borates of lime and boracite with a view to the manufacture of borax and of other borates. Filed 17 February. Date applied for October 2, 1889, being date of application in France.

2633. J. Leith. Improvements in or appertaining to the distillation or treatment of ammonium chloride with Leblanc alkali waste or kindred substances. February 18.

3087. E. G. Lawrence. — From S. Pick, Galicia. Improvements in apparatus for evaporating liquors containing salts, and for separating such salts when rendered insoluble by evaporation of the liquors. February 26.

3220. J. Greenwood. See Class X.

3391. O. E. Pohl. Improvements in the manufacture of salt, and in apparatus and appliances employed therein. March 4.

3460. T. Michel. Improvements in the manufacture or production of carbonate of soda and hydrochloric acid from chloride of sodium. March 4.

3992. G. Horsley and A. C. Wilson. Improvements in the means or apparatus to be employed in the production of solutions containing sulphurous acid or its salts. March 14.

4095. E. Bergmann. Improvements in the manufacture or production of cyanide compounds from ferrocyanide compounds. March 15.

### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

7716. C. F. Hine and J. H. Noad. Process for producing cuprammonia. February 26.

16,711. E. Augé. Manufacture of soda alum. March 12.

1890.

1300. H. H. Leigh. — From T. de Brochoeki. Manufacture of anhydrous barium oxides. March 19.

## VIII.—GLASS, POTTERY, AND EARTHENWARE.

### APPLICATIONS.

2583. J. H. Bailey. Washable, glazed, or enamelled railway signal arms. Complete Specification. February 18.

2626. C. Huelser. — From J. Kemper. Process for producing opalescent glass or enamel from a glass mixture containing silico-fluorides of alkalis or compounds thereof. February 18.

2906. E. V. Caspar. Improvements in glass decoration with metallic linings. February 24.

3121. D. Rylands. Improvements in machinery for the manufacture of glass bottles or other hollow glass ware, and in the process of such manufacture. February 27.

3137. W. Ambler. An improved method and means to be used in the manufacture of glass bottles. February 27.

3138. W. Ambler. An improved method and means to be used in the manufacture of glass bottles. February 27.

3203. C. Leigh. Tinting opal glass. February 28.

3229. E. Leak and H. Aynsley. Improvements in and relating to means used in supporting biscuit and glost ware in kilns and ovens. Complete Specification. February 28.

3286. J. G. Sowerby. An improvement in the manufacture of glass. March 1.

3343. T. Fletcher, R. Crag-Brown, and W. Ure. Improvements in brick and pottery kilns. March 3.

3496. T. G. Turnbull and M. Turnbull. Improvements in or relating to glass manufacture. March 5.

4021. A. J. Boulton. — From P. Sievert, Germany. Improvements in the production of solution of alkaline silicate or water-glass, and in apparatus therefor. Complete Specification. March 14.

COMPLETE SPECIFICATIONS ACCEPTED.

1889.

5138. A. W. Turner and G. Tomkinson. *See* Class X.  
 7659. J. Quaglio. Process and furnace for preparation of crude glass according to Leuffgen's method, by the combined use of water-gas, coal-gas, wood-gas, oil-gas, or natural gases and generator gas. March 5.  
 8049. T. Davidson. Improvements in pressed glass dishes and their manufacture. March 19.

IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

APPLICATIONS.

2896. W. Joy. Improvements in the manufacture of cement, and in apparatus for use in the said manufacture. February 22.  
 2940. H. Benjamin. A new and useful composition of matter, which he calls "ferasphalte," to be used for roadways, sidewalks, roofing, grouting, and other purposes, including submarine works and other constructions of masonry. February 24.  
 3025. G. West. Improvements relating to plastering material or compositions. Complete Specification. February 25.  
 3070. W. Weaver. India-rubber-wood road pavements. February 26.  
 3162. J. C. Merryweather. Improvements in paving materials. February 27.  
 3184. A. Rovedino. An improved wood paving block. A similar wood paving block combined with concrete or other substance for paving. An improved paving brick of similar form and treatment as wood paving block. February 28.  
 3413. W. T. Sier. Preparing stone for enamelling and other purposes. March 4.  
 3433. W. G. Williams and S. Taylor. An irradiaut surface decoration. March 4.  
 3462. Baron A. von Solemacher. New or improved manufacture of bricks and plates or tiles. March 4.  
 4069. W. Conway. An improved brick, applicable for building purposes. March 15.

COMPLETE SPECIFICATIONS ACCEPTED.

1889.

5363. B. Rhodes. Non-corrosive material, suitable as a cement packing or covering to resist acids. March 5.  
 5412. T. K. Ferguson. Combination of ingredients for use as mortar, plaster, &c. February 26.  
 5919. W. S. Lea and H. M. Lynam. Manufacture and composition of refractory or fire-resisting cement or cements. March 5.  
 6874. J. N. Tata and J. Lyon. Manufacture of mosaics for floors, walls, ceilings, &c. March 5.  
 7913. S. Hindley and G. R. McKenzie. Manufacture of cement for joining wood, stone, &c. March 12.  
 20,914. A. Klapperstuck and A. H. Meyer. Fireproof ceilings and walls. March 12.

1890.

1525. I. Button, E. Peters, and J. W. Goodsell. Kilns for burning bricks, earthenware, limestone, cement, &c. March 12.

X.—METALLURGY, MINING, Etc.

APPLICATIONS.

2533. C. Thompson. The recovery of metal from sheet iron, scrap, &c. February 17.  
 2544. J. W. Chenhall. Improvements in or relating to blast or smelting furnaces having water jackets. February 17.  
 2772. E. L. Mayer and J. G. Lorrain. Improvements in or connected with the separation and recovery of gold and the treatment of ores or other matters containing gold, or with which gold is combined or associated. February 20.  
 2901. F. J. Jones. An improved method of making tubes, cylinders, and other hollow ware, of copper, its alloys, and aluminium. February 22.  
 2981. M. F. Coomes and A. W. Hyde. Improvements in the manufacture of steel. Complete Specification. February 25.  
 3154. W. A. Thoms. Improvements in the manufacture of alloys. February 27.  
 3220. J. Greenwood. Improvements in the manufacture or production of metallic sodium and of chlorine gas. February 28.  
 3245. W. D. Bohm. Improvements in the separation of gold and silver from ores or materials containing them. February 28.  
 3246. W. D. Bohm. Improvements in and apparatus for the separation of gold and silver from ores or materials containing them. February 28.  
 3275. G. Simonin. Improvements in the reduction of metallic ores. March 1.  
 3369. G. H. Cammell. Improvements in the welding of metals. March 3.  
 3409. F. W. Harbord. Improvements in the utilisation of tin-plate scrap. March 4.  
 3410. A. C. Hyde. Improvements in the process of removing tin from tin plate, and in part applicable to the preparation of pure tin. March 4.  
 3412. A. Hodgkinson, jun. Improvements in the treatment of mines and minerals containing iron, whether calcined, partly calcined, or raw, and whether mixed or unmixed, and the manufacture of the same into blocks or lumps for the better extraction of iron therefrom. March 4.  
 3440. J. L. Sehenius. Process for removing gases and foreign impurities from metals or alloys. Complete Specification. March 4.  
 3483. L. L. Burdon. Seamless compound ingot or wire. Complete Specification. March 4.  
 3532. W. Crookes. Improvements in amalgamating and extracting gold from refractory and other ores. March 5.  
 3782. L. Pszczolka. A method for obtaining uniform products from the Thomas converter, Siemens' open hearth, or other basic furnaces. Complete Specification. March 10.  
 3829. G. A. Jarvis. Improvements in plant or furnaces for the reduction of sodium or other metals. March 11.  
 4030. J. H. Darby. Improvements in the manufacture of steel or iron. March 14.

COMPLETE SPECIFICATIONS ACCEPTED.

1889.

4335. W. E. Everitt. Coating sheets of iron with copper or brass, and apparatus therefor. March 5.  
 5138. A. W. Turner and G. Tomkinson. Annealing furnaces. March 19.  
 7047. W. F. Jackson and W. Galbraith. Manufacture of steel. February 26.  
 8789. M. Crawford. Means for separating particles of different specific gravities, especially the separation of gold, &c. from crushed ores. February 26.  
 9905. H. A. Becker. Conversion of crude iron into malleable iron and steel, and apparatus therefor. March 12.



10,472. J. V. Johnson.—From C. Pieper. Improvements in compound steel armour plates, and in the manufacture thereof. March 12.

19,622. D. Dennes and T. K. Rose. Apparatus for extracting precious metals from their ores. March 5.

1890.

1634. G. D. Burton. Manufacture of forgings. March 5.

1641. F. W. Dähne. Calcining and refining copper ore, regulus, and precipitate, and construction of furnaces therefor. March 5.

1682. R. Heathfield. Apparatus for coating metal sheets with metals or alloys. March 5.

1888. R. C. Totten. Improvements in dies, ingots, and other castings. March 12.

## XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

### APPLICATIONS.

2687. A. W. Armstrong. The utilisation of a waste product for the means of producing electricity. February 19.

2819. C. Lütke, jun. An improved electrolytic solution for galvanic batteries. Complete Specification. February 21.

2845. E. T. Mackrill. Improvements in secondary batteries. February 21.

2945. A. M. F. Laurent-Côly and I. A. Timmis. Improvements in and relating to, and in the manufacture of, electric secondary cells. February 24.

3008. W. B. Hollingshead. Improved storage or secondary batteries. Complete Specification. February 25.

3013. C. J. Hartmann. Improvements in storage or secondary batteries. Complete Specification. February 25.

3028. A. C. Reigner. Improvements in dynamo-electric and electro-dynamic machines. February 25.

3152. W. A. Thoms. Improvements in the manufacture of electrical conductors. February 27.

3153. W. A. Thoms. An improved mode of manufacturing electrical conductors. February 27.

3271. C. N. Russell and R. A. Scott. Improvements in dynamo-electric machines. March 1.

3322. F. Keep.—From T. and S. Morrin, Limited, New Zealand. An improved voltaic battery. March 3.

3373. M. Sappey. Improvements connected with the charging of electrical accumulators or secondary batteries. March 3.

3374. G. E. B. Pritchett. Improvements in electric accumulators or secondary batteries. March 3.

3439. S. C. C. Currie. Improvements in secondary or storage batteries. Complete Specification. March 4.

3455. H. H. Lake.—From T. Ewing, United States. Improvements in and relating to electric secondary batteries or accumulators. Complete Specification. March 4.

3572. Sir C. S. Forbes, Bart. Improvements in primary batteries. March 6.

3697. J. Anderson. Improvements in galvanic cells and batteries. March 8.

3717. L. Weigert. An improved electrolytic solution for galvanic batteries. Complete Specification. March 8.

3731. The Mining and General Electric Lamp Co., Lim., and D. G. FitzGerald. Improvements in or connected with the positive elements (negative electrodes in charging) of secondary batteries, which elements are applicable also as cathodes in electrolysis. March 8.

3924. H. H. Lake.—From The Crosby Electric Co., United States. Improvements in electric batteries. Complete Specification. March 12.

3967. J. S. Stevenson. Improvements in or connected with the manufacture of secondary battery elements. March 13.

4087. H. T. Cheswright. Improvements in secondary voltaic batteries. March 15.

### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

4871. H. A. Seegall. Electrolytic process of obtaining copper, zinc, and tin. February 26.

6745. M. Kiliani. Method and means for electrolysis of substances in a state of fusion. February 26.

7428. C. Pollak. Electric accumulators and plates or electrodes therefor. March 12.

14,148. J. Kümme. Manufacturing pipes, tubes, and other articles of metal by galvanic deposits. March 12.

20,217. F. Marx. Manufacturing metallic electrodes for galvanic batteries. March 12.

1890.

1094. H. H. Lake.—From T. Ewing. Secondary or storage batteries. February 26.

1110. C. A. Hitchcock.—From W. Burnley. Galvanic batteries. February 26.

1495. H. H. Lake.—From G. A. Johnson. Storage batteries or accumulators. March 5.

## XII.—FATS, OILS, AND SOAP MANUFACTURE.

### APPLICATIONS.

2864. W. Young. Improvements in purifying or treating paraffin wax, and in apparatus therefor. February 22.

3303. M. Williams. Improvements in the manufacture of soft soap. March 5.

3601. R. C. Scott. Improvements in or appertaining to the manufacture or shaping of soap. March 6.

### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

11,383. C. Lahnson and C. Feuerlein. Extracting pure neutral grease from wool or the like. March 12.

1890.

1659. P. Plisson. Manufacture of lubricating grease, and apparatus therefor. March 5.

## XIII.—PAINTS, PIGMENTS, VARNISHES, AND RESINS.

### APPLICATIONS.

2539. N. B. Dennys. An improvement in preservative and anti-fouling compositions for ships' bottoms, &c. February 17.

2682. J. Sloan and E. W. Bell. Improvements in anti-fouling or preservative paints or compositions. February 19.

2727. J. Horne. Improved manufacture of hand-clean white and coloured crayons and materials for marking and colouring surfaces. February 20.

2808. W. Noad. Improvements in the manufacture of white lead. February 21.

3786. G. Larrony. Improvements in or relating to the manufacture of litharge and red lead. March 10.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

6815. R. W. E. MacIvor and others. Production of white lead or carbonate of lead. February 26.

7156. J. Macfarlane. Obtaining yellow pigments. March 5.

9412. M. Williams and J. Aseough. Manufacture of blue colouring matter for laundry, bleaching, and other purposes. March 5.

9565. H. Noerdlinger. Manufacture of oil varnish. February 26.

16,706. P. Bronner. Manufacture of white lead. March 19.

## XIV.—TANNING, LEATHER, GLUE, AND SIZE.

## APPLICATION.

3295. P. Houston and C. Beakbaue. Improvements in the manufacture or production of tannage or tanning liquors. March 1.

## COMPLETE SPECIFICATIONS ACCEPTED.

1890.

556. G. Rothel. An improved glue or cement. March 19.

1482. W. P. Thompson.—From F. G. Hermann. Apparatus for removing the hair from skins. March 5.

## XVI.—SUGARS, STARCHES, GUMS, Etc.

## APPLICATIONS.

3030. A. Weisz. Improvements in the manufacture of starch. February 25.

3240. A. A. Brehier and B. G. Talbot. Manufacture of artificial tartaric acids by means of cellulose, saccharine, or amylaceous substances. February 28.

3589. C. Steffen. Improved process for obtaining the entire crystallisable sugar contained in more or less pure saccharine solutions, juices of plants and the like, by employing the same for washing crystalline sugar mass in special manner before the said saccharine solutions are subjected to a novel method of boiling same. Complete Specification. March 6.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

7517. G. Adant. Apparatus for the manufacture of sugar. March 12.

1890.

1282. C. D. Abel.—From Messrs. Pfeifer and Langen. Treatment of sugar crystals or grains for facilitating the purifying thereof. March 5.

2197. W. P. Thompson.—From K. Korenar and F. Zwicker. A new adhesive cement, and process of manufacturing same. March 19.

## XVII.—BREWING, WINES, SPIRITS, Etc.

## APPLICATIONS.

2958. J. Stevenson.—From E. Riglet, France. The production of alcoholic beverages and alcohol without the employment of cereals. February 24.

2990. H. S. G. Stephenson. Improvements in apparatus for generating and supplying carbonic acid gas to vessels containing fermented liquor. February 25.

3579. C. Hof.—From L. Rüksam, Bavaria. Improvements relating to the production of malt. March 6.

3606. E. Carez and La Société Generale de Maltosé. An improved method of saccharification of amylaceous materials. March 6.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

3707. C. R. C. Tielborne, A. Darley, and M. F. Pureell. Method and appliances for the collection and utilisation of the carbonic acid and other products given off during the process of all fermentations. March 12.

3981. G. Reininghaus. Preparation of malt. March 5.

6918. C. R. Bonne. Manufacture of German yeast. February 26.

8313. L. A. Enzinger. Apparatus for sterilising fermented alcoholic liquors. February 26.

8839. C. Huelser.—From J. Kuntze. Apparatus for pneumatic malting. March 19.

## XVIII.—CHEMISTRY OF FOODS, SANITARY CHEMISTRY, AND DISINFECTANTS.

## APPLICATIONS.

*A.—Chemistry of Foods.*

2620. F. Grognet. A new or improved method for preserving meat, fish, and the like. February 18.

2742. J. Y. Johnson.—From C. Risk, West Indies. Improvements in curing cocoa and the like, and in the means and apparatus employed therefor. February 20.

3856. E. Leconte. Improvements in the treatment or regeneration of yeast to be used in the process of bread-making, and in the preparation of paste and dough for pastrycooks and biscuit-bakers. March 11.

3995. G. Dosmond and F. Rozes. An improved process and apparatus for preserving meat and other alimentary substances. March 14.

4102. A. A. Brehier. Improvements in the preservation of meat and other articles of food, such as poultry, game, fish, eggs, cheese, fruits, vegetables, &c. March 15.

*B.—Sanitary Chemistry.*

2563. S. F. Milligan and R. Barklie. Apparatus for destroying deleterious emanations from sewers. February 18.

3062. F. L. H. Daneshell. Improvements in the treatment of sewage or other liquids having matters in suspension, and in means or apparatus employed therein. February 26.

3312. W. E. Adeney and W. K. Parry. Improvements in the purification of sewage and other waste liquors containing organic matters. March 1.

*C.—Disinfectants.*

3134. I. S. McDougall and J. T. McDougall. Improvements in compounds for washing or dipping sheep, cattle and horses for the extermination of insects or parasites. February 27.

3628. F. Bale and C. Sims. Improvements in the manufacture and production of hydroxyl and peroxide of hydrogen or oxide of water, and in the apparatus employed therein. March 7.

## COMPLETE SPECIFICATIONS ACCEPTED.

*A.—Chemistry of Foods.*

1889.

3172. E. Sonstadt. Processes and apparatus for preparation of an extract of coffee, and utilisation of the by-products. March 5.

1890.

1326. A. Schmidt-Mulheim. Method and apparatus for production and preservation of sterilised milk. March 5.

1328. J. T. Appleberg. Apparatus for and method of preserving and purifying milk. March 12.

*B.—Sanitary Chemistry.*

1889.

5753. B. Jagger, H. B. Jagger, and A. Turley. Means for purifying sewage and other waters, and for deodorising and utilising the solids and excreta. March 12.

8088. C. H. Beloe and F. Candy. Purification and filtration of sewage and other foul liquids. March 12.

*C.—Disinfectants.*

1889.

19,309. G. T. Moody and F. W. Streatfield. Producing and liberating aerial or gaseous disinfectants. March 19.

**XIX.—PAPER, PASTEBOARD, Etc.**

*APPLICATIONS.*

2698. W. B. Kemshead. Improvements in the manufacture of paper and like textile fabrics. February 19.

2832. E. Applegarth. Improvements relating to the treatment of straw, esparto grass, and other fibrous materials, and to compounds therefor. February 21.

3264. A. H. Haigh. Improved waterproof paper and textile fabrics. March 1.

3593. S. W. Marchant, jun. Improvements in the manufacture of paper, and in apparatus therefor. March 6.

*COMPLETE SPECIFICATIONS ACCEPTED.*

1889.

17,496. E. A. Blumel. Covering various articles with xylonite or celluloid. March 19.

19,638. R. Hannan. Toilet or sanitary paper. March 12.

1890.

682. O. Imray.—From the Celluloid Manufacturing Co. Collar, cuff, and front, with waterproof body, and coverings having a coating of pyroxyline varnish. March 12.

683. O. Imray.—From the Celluloid Manufacturing Co. Waterproof collar, cuff, and front, composed partly of vegetable parchment. March 12.

1514. F. C. Alkier. Extracting substances dissolved in the waste-water of paper manufacture. March 5.

**XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES, AND EXTRACTS.**

*COMPLETE SPECIFICATION ACCEPTED.*

1889.

7391. J. F. von Mering. Production of a substance to be called "chloralformamide" or "chloralamide." February 26.

**XXI.—PHOTOGRAPHIC PROCESSES AND MATERIALS.**

*APPLICATIONS.*

3393. E. W. Foxlee. An improved flexible film as a support for photographic negatives and positives. March 4.

3958. J. W. Clarke. A new or improved process for the reproduction of photographs or other devices or purposes, and in readily reproducing same. March 13.

*COMPLETE SPECIFICATION ACCEPTED.*

1889.

8085. H. W. Jones. Production of developers for photographic use. March 12.

**XXII.—EXPLOSIVES, MATCHES, Etc.**

*APPLICATIONS.*

2547. J. Y. Johnson.—From C. Pieper, Germany. Improvements in the manufacture of gunpowder from nitro-cellulose. February 17.

2769. H. A. Schlund. Improvements in percussion fuses. February 20.

2883. D. D. Eason. Improvements in fuses. February 22.

2960. W. D. Borland. Improvements in nitro-compound powders for ballistic purposes. February 25.

2993. J. Beswick. Improvements in or appertaining to blasting and other operations where gunpowder is not used. February 25.

3176. C. F. Martin. An improvement in matches. February 28.

3475. J. C. Bull. Improvements in percussion fuses. March 4.

3604. F. E. D. Acland. Improvements relating to time and percussion fuses. March 6.

3819. E. C. Mance. Improvements in signal rockets. March 11.

3852. H. H. Lake.—From S. H. Emmens, United States. Improvements in explosives. March 11.

3865. M. E. Steedman. An improved lucifer match. March 12.

*COMPLETE SPECIFICATIONS ACCEPTED.*

1889.

3761. H. C. L. Holden. Electric tubes and fuses for firing ordnance and exploding torpedoes, mines, &c. March 12.

4539. E. Eisenmann and A. Arche. Manufacture of picric acid. March 5.

5208. C. R. E. Bell. Manufacture of matches, and apparatus therefor. March 5.

6185. H. T. Arbuthnot and G. Hookham. Percussion fuses for explosive shells. March 12.

6595. L. Cobbaert. A machine for the continuous manufacture and boxing of matches. March 12.

# THE JOURNAL OF THE Society of Chemical Industry:

A MONTHLY RECORD

FOR ALL INTERESTED IN CHEMICAL MANUFACTURES.

No. 4.—VOL. IX.]

APRIL 30, 1890.

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## NOTICES.

In accordance with the provisions of Rule 18 of the Bye-laws, notice is hereby given that those Members of Council whose names are placed in italics in the annexed list will retire from their respective offices at the forthcoming Annual General Meeting.

Mr. E. Rider Cook has been nominated to the offices of President and Treasurer; and Sir Lowthian Bell, Bart., F.R.S., has been nominated Vice-President under Rule II.

Mr. David Howard, Mr. B. E. R. Newlands, and Mr. J. C. Stevenson, M.P., have been nominated Vice-Presidents under Rule 8; and Mr. R. R. Tatlock and Mr. T. W. Stuart have been nominated Ordinary Members of Council under Rule 17, in the place of Mr. B. E. R. Newlands and Mr. J. C. Stevenson, M.P., nominated Vice-Presidents.

The Foreign Secretary has been nominated for re-election.

Members are hereby invited to nominate fit and proper persons to fill four vacancies among the Ordinary Members of Council under Rule 18. Special nomination forms for this purpose can be obtained from the General Secretary upon application.

*Extract from Rule 18:*—"No such nomination shall be valid unless it be signed by at least ten members of the Society who are not in arrear with their subscriptions, nor unless it be received by the General Secretary, at the Society's office, at least one month before the date of the commencement of the Annual General Meeting, to the election to take place at which it refers. Nor shall any such nomination be valid if the person nominated be ineligible for election under Rules 12 or 15. No member shall sign more than one nomination form."

#### ANNUAL GENERAL MEETING.

Notice is hereby given that the next Annual General Meeting will be held in Nottingham on Wednesday, Thursday, and Friday, the 9th, 10th, and 11th of July next. Detailed arrangements and the programme of proceedings will appear as soon as complete. Tickets of membership will be issued in time for the meeting, and will form, as heretofore, vouchers for visits to works and excursions.

Post Office Orders should be made payable at the General Post Office, London, to the Honorary Treasurer, E. Rider Cook, and should be forwarded to him at Bow, unless it be desired to notify a change of address.

Members who require extra sets or back numbers of the Journal are requested to make application to the General Secretary only, to whom also changes of address should be communicated.

Authors of communications read before the Society, or any of its Local Sections, are requested to take notice that under Rule 41 of the bye-laws, the Society has the right of priority of publication for three months of all such papers. Infringement of this bye-law renders papers liable to be rejected by the Publication Committee, or ordered to be abstracted for the Journal, in which case no reprints can be furnished to the author.

Notice is hereby given, for the information of members and advertisers, that the advertisement columns of this Journal have been contracted for by Messrs. EYRE and SPOTTISWOODE, the Society's printers and publishers, to whom all communications respecting them should be addressed.

The Secretary is instructed to negotiate for the purchase of copies of the Society's Journal for January 1883, and January, February, and April 1886. Members possessing odd copies of these numbers are particularly requested to communicate at once, stating price required, with Mr. Cresswell. The stock of all other numbers is at present sufficient for the Council's requirements.

#### LIST OF MEMBERS ELECTED, 23rd APRIL, 1890.

- Allen, Wm. N., 2, Chesnut Street, Philadelphia, Pa., U.S.A., chemical manufacturer.
- Bentz, Ernest, 147, Bishop Street, Alexandra Park, Manchester, lecturer on dyeing.
- Blakey, A. J., Dudridge Mills, Stroud, Gloucestershire, foreman dyer.
- Craig, Saml. E., 110, Cannon Street, London, E.C., colliery representative.
- Fleck, Hermann, 108, Rittenhouse Street, Germantown, Philadelphia, Pa., U.S.A., chemical student.
- Frost, Howard V. (Ph.D.), Polytechnic Inst., Brooklyn, N.Y., U.S.A., professor of chemistry.
- Hoskins, A. Percy, 19, Ashness Road, New Wandsworth, S.W., analytical chemist.
- Kerr, Saml. T., c/o Alex. Kerr Bros. and Co., Philadelphia, Pa., salt manufacturer.
- Lang, Jas. G., 5, Viewfield Terrace, Hillhead, Glasgow, analytical chemist.
- McCulloch, Norman, 7, Melrose Street, Glasgow, analytical chemist.
- McDougall, Jas. T., Dunolly, Morden Road, Blackheath, S.E., manufacturing chemist.
- McFarlane, W. W., 110, Oxford Street, Philadelphia, Pa., U.S.A., dye works superintendent.
- Marvin, Chas., Grosvenor House, Plumstead Common, Kent, author.
- Mayer, Nelson B., 945, North 8th Street, Philadelphia, Pa., U.S.A., chemical student.
- Morris, Herbert N., Littondale, Manley Road, Manchester, chemical student.
- Porter, Herbert, Pleasley Meadows, near Mansfield, Notts, wool spinning manager.
- Pullman, Arthur, "Stalheim," Godalming, Surrey, leather manufacturer.
- Pullman, Edw. E., Westbrook Mills, Godalming, Surrey, leather manufacturer.
- Pullman, Wm. R., Hermitage, Godalming, Surrey, leather manufacturer.
- Reid, J. Keith, Buckingham, Prov. of Quebec, Canada, mining agent.
- Rogers, Harry, 5, Stoke Newington Common, London, N., secretary to Patent Stone Co.
- Smith, Henry H., Royal Laboratory, Woolwich Arsenal, S.E., analytical chemist.
- Strawson, Geo. Fred., Newbury, Berks, chemical manufacturer.
- Studer, Simon J., Phoenix Chemical Works, Bradford, Manchester, chemist.
- Welsh, Thos. L., 51, St. Vincent Crescent, Glasgow, chemical student.
- Wetter, Jasper, 433, Strand, London, W.C., patent agent.
- Wood, Ebenezer, Stephenson Street, Canning Town, E., manufacturing chemist.
- Wyatt, Francis (Ph.D.), 24, Park Place, New York, U.S.A., consulting chemist.

#### CHANGES OF ADDRESS.

- Bahington, P., l/o Rotherham; Astyra Mining Co., Dardanelles, via Constantinople.
- Bayley, Thos., l/o Clonskeagh; 3, Herbert Avenue, Merrion, co. Dublin.

Bell, J. Ralston, 1/o Doneaster; 27, Lansdowne Creseent, Glasgow.

Branson, F. W., Journals to 24, Mount Preston, Leeds.

Burrows, Edw., Journals to Belle Vue Road, Low Fell, Gateshead-on-Tyne.

Collins, C. Spenceer, 1/o Manchester; 16, Albert Place, Leith Walk, Edinburgh.

Collins, H. S., 1/o Thames Street; e/o Langton and Co., 38, Wilson Street, Finsbury, E.C.

Donald, W. J. A., Journals to Castle Park, Irvine, N.B.

Elborne, Wm., 1/o Manchester; 4, Willis Road, Petersfield, Cambridge.

Fraser, W. J., Journals to 98, Commereial Road East, London, E.

Grundtvig, C. H. W., Journals to e/o Lake Copais Co., Thebes, Greecee.

Jenkins, Thos., 1/o Wales; Laboratorio, Minas de Rio Tinto, Spain.

Kent, W. H., 1/o Wokingham Road; The Lodge, Bulmershe Road, Reading.

Lake, G., jun., 1/o Manchester; 6, Turn Lee Road, Glossop, Derbyshire.

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Robinson, H. H., 1/o Canton; Imperial College, Hankow, China.

Smith, H. H., Journals to Wilton House, Vicarage Park, Plumstead, S.E.

Tervet, R., 1/o Glasgow; 41, Morpeth Road, South Hackney, E.

Tothill, M. A. L., 1/o Riebeck Square; e/o Castle Chemical Co., Cape Town, South Africa.

Wills, G. S. V., 1/o Tulse Hill; Southwood, Croham Road, South Croydon.

Wilson, C. J., 1/o Gordon Square; 19, Little Queen Street, Westminster, S.W.

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<i>C. C. Hutchinson.</i>	

*Hon. Local Sec. and Treasurer: Thos. Tyrer, Battersea, S.W.*

The names in Italics are those of members of Committee who retire at the end of the current Session.

The following have been elected to fill the vacancies and will take office in July next:—*Chairman: T. Tyrer; Secretary: T. W. B. Mumford; Committee: J. Dewar, A. G. Green, C. W. Heaton, D. Howard, W. S. Squire, T. E. Thorpe.*

### SESSION 1889-90.

1890.

May 5th:—Mr. M. L. Grillin. "Moisture in Paper Pulp" and other communications.

June 2nd:—Dr. W. S. Squire. "The Artificial Production of Cold theoretically and practically considered."

July:—Annual General Meeting in Nottingham.

Mr. Wm. Webster's paper on "The Electrical Treatment of Sewage" is postponed.

*Meeting held Monday, March 31st, 1890.*

MR. DAVID HOWARD IN THE CHAIR.

### NOTE ON A MACHINE FOR A METHODICAL SEPARATION OF SOLIDS AND LIQUIDS.

BY W. S. SQUIRE, PH.D.

SOME time ago I had occasion to design a machine for the separation of solid from liquid matters in such a way that the solid matter should be completely washed out and exhausted of its soluble constituents, while, on the other hand, this should be accomplished with the least possible dilution of the liquid. As this machine worked very well in practice, I thought a description of it might interest the members of the London Section of this Society. It is, however, a machine which is not of universal applicability. It will not, for example, deal with freshly-precipitated sulphate of barium, or anything of that kind, but is intended rather for dealing with vegetable matters, such as beetroot, brewers' grains, and substances of that description.

This machine consists of a series of cylinders, usually four in number, constructed of perforated metal, or of wire gauze, or of cloth stretched on suitable frames. These cylinders revolve horizontally, but slightly inclined over troughs placed underneath them to receive the liquid which passes through, while the solid matter is discharged at the lower end of the cylinders into vessels placed so as to receive it. Each of these vessels is provided with an agitator to mix up the solid matter with any liquid that may be added to it, and is also provided with a pump in order to pump away the mixture so made.

The mixture of solid and liquid matter to be treated is admitted in a stream into the upper end of cylinder No. 1. The liquid passes through into the trough below, while the solid matter is expelled at the lower end of the cylinder into the vessel placed to receive it. It is here mixed with the liquor flowing from the trough placed under cylinder No. 3, and the mixture is pumped into cylinder No. 2. The liquid which passes through into the trough below is either added to that coming from cylinder No. 1 or it is

## Death.

J. B. Hutcheson, of Glasgow University, at Dunoon, N.B. April 15th.

used to dilute the original mixture before it is admitted to cylinder No. 1, or it may be used for treating fresh solid matter, the soluble constituents of which it is desired to extract. The solid matter expelled from the lower end of cylinder No. 2 is in like manner mixed with the liquid coming from the trough under cylinder No. 4, and is then pumped into cylinder No. 3. The solid matter expelled from this cylinder is mixed with water or other menstruum and pumped into cylinder No. 4, from which it is finally expelled, washed out and exhausted. In this way the solid

matter travels successively from cylinder to cylinder, encountering on its course liquid always more and more dilute whilst the liquid travels in the opposite direction, encountering on its course matter more and more charged with soluble matter. If the exhaustion is not complete a larger number of cylinders may be used.

Fig. 1 is a side elevation of the apparatus. Fig. 2 is a plan in which two of the four cylinders are shown in view and two in section. Fig. 3 is an elevation of the upper ends of the four cylinders, and Fig. 4 is an elevation of the lower ends

Fig. 1.

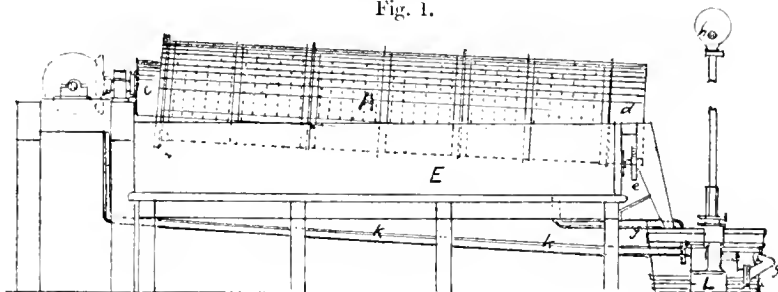


Fig. 2.

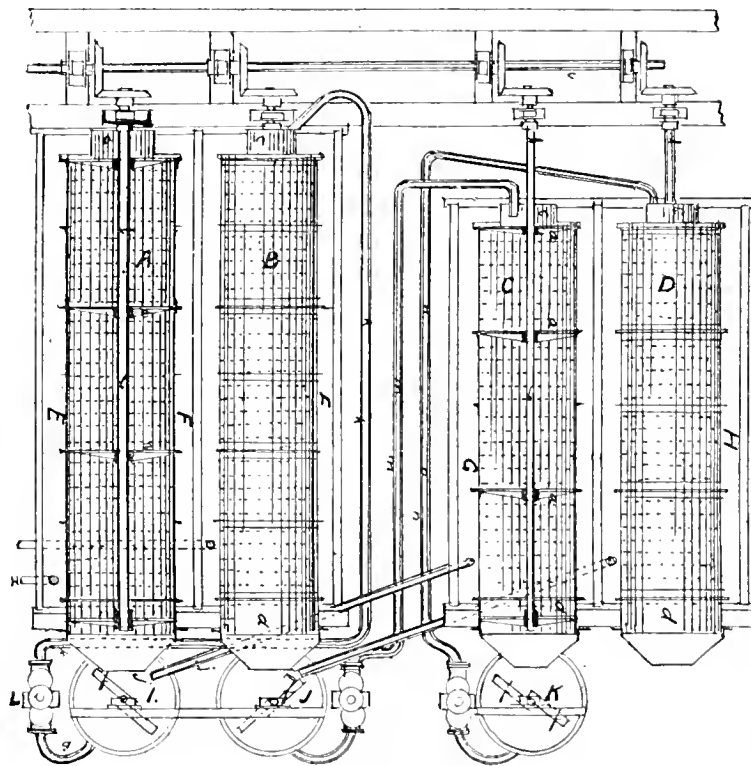
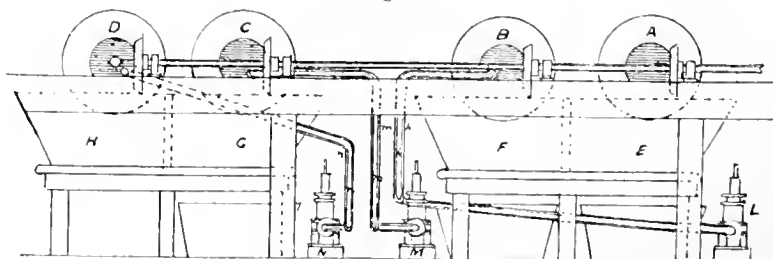


Fig. 3.

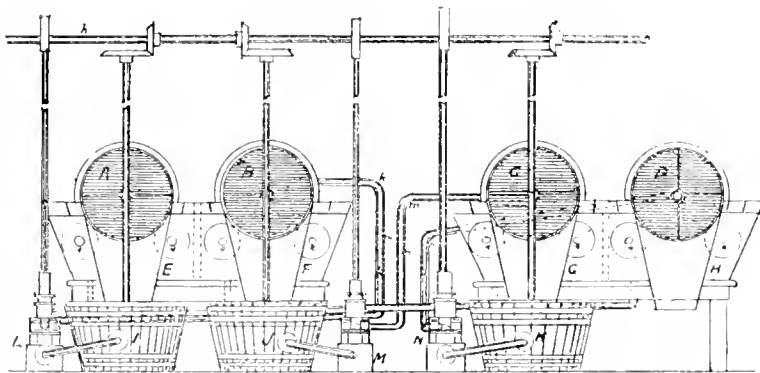




of the four cylinders where the solid matter is discharged. A B C D are the four perforated cylinders, E F G H are the four troughs into which the liquid falls through the perforations of the cylinders. (In this case there are only two troughs, but each is divided into two by a division in the

centre so as practically to form four troughs.) I J K are the vessels into which the solid matter is discharged from the mouths of the revolving cylinders, and L M N are the pumps by means of which the mixture of solid and liquid matters are pumped into the successive cylinders.

Fig. 4.



The perforated cylinders may be made in one piece, but I prefer to build them up of short perforated drums bolted to flat spoked wheels *aa* (Fig. 2) keyed on to the driving shafts *bb*. The upper ends of the cylinders are closed by means of a plate with an orifice in the centre furnished with a short drum (*c*) not perforated, which serves for the introduction of the mixture of solid and liquid matters by means of pipes entering these drums by the side of the driving shafts. The other end of the cylinders consists of a drum of metal *d* of the same diameter as the cylinder, but not perforated, furnished with spokes, and keyed on to the driving shaft. Through these drums the solid matters are discharged. These drums run on small wheels, *ee* (Figs 1 and 4), which support the lower end of the cylinder.

The vessels I J K into which the solid matter is discharged are furnished with agitators *f f* which stir up and mix the discharged solid matter with any liquid which may run into these vessels. The pumps L M N are lift and force pumps connected by the pipes *g g* with the receiving vessels, and are intended to pump the mixture of solid and liquid matters into the cylinders. Both pumps and agitators are driven by means of an overhead shaft *h* furnished with eccentrics and bevil wheels for that purpose. The cylinders are driven by means of a main shaft *i* by means of mitre wheels keyed on to the main shaft and the driving shafts of the cylinders.

The course of the operation is as follows :—The mixture of solid and liquid matters is introduced into the cylinder A through the small drum *c*. As the cylinder revolves the liquid runs through the perforations of the cylinder into the trough E placed below it, and is drawn off by the pipe *r*. The solid matter in consequence of the inclined position of the cylinder gradually makes its way to the lower end, where it is discharged. As the solid matter has been thoroughly rolled over and puddled in its course from the upper to the lower end of the cylinder, it is deprived of most of the liquid matter. It is discharged into the vessel I, where it meets the liquid matter flowing from the trough G placed below the cylinder C through the pipe *j*, and the mixture so formed is pumped by the pump L through the pipe *k* into the cylinder B. The liquid which runs through into the trough F may be mixed with the liquid which falls into the first trough E, or it may be used for treating a fresh quantity of solid matter before it is introduced into the cylinder A. The solid matter which is discharged into the vessel J is mixed with the liquid from the trough H under the cylinder D through the pipe *l*, and the resulting mixture is then pumped by means of the pump M through the pipe *m* into the cylinder C. The liquid which passes through this cylinder falls into the trough G and flows through the pipe *j* to the vessel I. The solid matter is discharged into the vessel K, where it is mixed with water or other menstruum

used to exhaust and wash out the solid matter. This mixture is then pumped by the pump N through the pipe *n* into the cylinder D. The liquid which runs through the cylinder D falls into the trough H and flows through the pipe *l* to the vessel J. The solid matter is discharged from this last cylinder D, washed out, and exhausted of its soluble matter.

It will be noticed that the solid matter travels progressively from A to B, from B to C, and from C to D, while the liquid travels in the opposite direction, from D to C, from C to B, and if used as the solvent of the soluble matters of any fresh solid matters from B to A. The process is, therefore, thoroughly methodical and continuous, and while on the one hand a very concentrated solution of the soluble matters is obtained, on the other hand the solid matter is obtained thoroughly exhausted of its soluble constituents.

Although I prefer to use cylinders for the purpose indicated, the inclined revolving perforated chambers may be of hexagonal, octagonal, or polygonal section.

When the apparatus is at work the separated liquor is discharged in a constant stream, while the exhausted solid matter falls continuously from the mouth of the last cylinder.

#### DISCUSSION.

The CHAIRMAN said that chemical engineering was a subject of primary interest to this Society, and, recognising the importance of the subject, the Society was always glad to welcome the suggestions of its engineering members. In the subject of the paper before them, they had one of those valuable suggestions which would be found applicable to a large number of bodies, and whose importance was enhanced by its suggestiveness of even further applications than those referred to by the inventor. There was one point of general practical interest which occurred to him :—How to get the maximum of exhaustive effect with a minimum of wash-water or solvent. The problem presented itself differently in different industries, whether in exhausting black ash or in making the tincture of a valuable drug. The apparatus described by Dr. Squire seemed to him to be a happy solution of at least one form of the problem.

## WOOL-FAT AND PROCESSES OF OBTAINING IT, WITH SPECIAL RELATION TO WOOL-WASHING.

BY H. W. LANGBECK.

WOOL-FAT mixed with water, in relation to medicine, has first been mentioned by Dioscorides, a Greek physician and author, living in the first century A.D. Such mixture of wool-fat and water was then called "Oesypus" (Οἶσυπος), and it retained this name for many centuries. Plinius also gives a description of Oesypus and how it is to be prepared. The Greek text and its translation into English is as follows:—

Κεφ. πδ'.—Περὶ Οἶσυπου.

Οἶσυπος δὲ λέγεται τὸ ἐκ τῶν οἰσυπρῶν ἐρίων λίπος· ὃ κατασκευάζεται οὕτως· λαβὼν ἕρια μαλακὰ οἰσυπρὰ ἐκπλυνον μὴ ἐστρουθισμένα θερμῷ ὕδατι, ἅμα ἐκθλίβων πᾶσαν τὴν ῥυπαρίαν, ἣν βαλὼν εἰς κρατῆρα πλατύστομον καὶ ἐπιχέας ὕδωρ ἀνάχει ἀρυτήρι ταράσσω βαγδαίως, ἕως ἂν ἀφρίσῃ· ἡ ξύλλω ἀνατάρασσε εὐτόνως, μέχρις ἂν ὁ πολὺς ἀφρὸς καὶ ῥυπαρὸς συλλεγῇ. ἔτα κατάρβαινε θαλάσῃ, καὶ ὅτε καταστῇ, τὸ ἐπινυχόμενον λίπος, ἀναλάμβανε εἰς ἕτερον κεραμεῦον ἀγγεῖον, ἐπιχέας τε ὕδωρ εἰς τὸν κρατῆρα, πάλιν τάρασσε καὶ κατάρβαινε τῇ θαλάσῃ τὸν ἀφρὸν, καὶ ἀναλάμβανε· καὶ τὸ αὐτὸ ποιεῖ, ἄχρις ἂν μηκέτι ἀφρὸς ἐφίστηται, δαπανηθέντος τοῦ λίπους. Τὸν μόντον ἀνηρημένον οἶσυπον τῇ χειρὶ μαλάσας, παραχρῆμα ἀναφοῦ, εἴ τινα ἔχει ἀκαθαρσίαν, στραγγίζον τὸ πρῶτον ὕδωρ καὶ ἄλλο ἐπιχέων καὶ κινῶν τῇ χειρὶ, ἄχρις ἂν προσαγόμενος τῇ γλώττῃ μὴ δάκνῃ, ποσὼς δὲ στύφῃ, λιπαρὸς καὶ καθαρὸς καὶ λευκὸς φανῇ· οὕτω δὲ ἀποτίθεο εἰς ἀγγεῖον κεραμεῖον· ἐν ἡλίῳ δὲ θερμῷ γινέσθω πάντα· ἔνιοι δὲ διηθήσαντες τὸ λίπος πλύνουσιν ἐν ὕδατι ψυχρῷ, ταῖς χερσὶν ἀνατρίβοντες, ὡς τὴν κηρωτὴν αἱ γυναῖκες· γίνεται δὲ ὁ τοιοῦτος λευκότερος· οἱ δὲ πλύναντες τὰ ἕρια καὶ ἐκθλίψαντες τὸν ῥύπον, ἔψουσιν μεθ' ὕδατος ἐν λέβητι πυρὶ κούφῳ, καὶ τὸ ἐπιστάμενον λίπος ἀφαιρούντες πλύνουσιν ὕδατι, ὥσπερ εἴρηται· καὶ διυλίσαντες εἰς λοπάδιον κεραμεῖον, ἔχον ὕδωρ θερμὸν, λυφὲ τέ ῥάκει περιτωμάσαντες, τίθεασιν ἐν ἡλίῳ, μέχρις ἂν αὐτάρκως παχὺς καὶ λευκὸς γίνηται· ἔνιοι δὲ διὰ δύο ἡμερῶν ἀποχέοντες τὸ πρῶτον ὕδωρ, ἄλλο ἐπιχέουσιν· ἔστι δὲ βελτίον ὁ ἀστρούθιστος καὶ λεῖος, ὅζων ἐρίων οἰσυπρῶν, ἀνατριβόμενός τε μεθ' ὕδατος ψυχροῦ ἐν μνάκι, καὶ λευκαίνόμενος, καὶ οὐδὲν ἔχων ἐν ἑαυτῷ σκληρὸν ἢ συνεστηκὸς, ὥσπερ ὁ δολιζόμενος κηρωτῇ ἢ στέατι. Δύναμιν δ' ἔχει θερμαντικὴν, πληρωτικὴν ἐλλκῶν, μαλακτικὴν, μάλιστα τῶν περὶ δακτύλιον καὶ ὑστέραν σὺν μελιωτῇ καὶ βουτύρῳ.

“CHAP. 84.—CONCERNING WOOL-FAT.

“Wool-fat is called the grease out of the filth of new shorn wool. You will prepare it in this manner: Having taken sweet newly shorn wool, not treated with soap wort, wash it in hot water, at the same time squeezing out all the filth; having put this into a wide-mouthed vessel pour water over it, and agitating it violently until froth is formed, pour it out again of the vessel; or agitate it vigorously with a stick until much scum and filth collects; thereupon sprinkle sea water over it, and when the fat swimming on the top has set put it into another earthenware vessel; pour more water into the tub, stir it up again, sprinkle the froth with sea water and take it out again, and do so until after the fat has been exhausted, no more froth gathers. Smooth the collected wool-fat with the hand, and if there is any filth remove it forthwith, squeezing out the first water and pouring on fresh and agitating it with the hand until applied to the tongue, it does not bite but has an astringent taste and looks brilliant and pure and white. In this condition store it away in an earthenware vessel. But all must be made in hot sunshine. Some treat the fat by washing it in cold water, rubbing it with the hands as women (do with) wax, thus it is rendered whiter. Others, after washing the wool and squeezing out the grease, boil it together with water in a cauldron over a slow fire, and having gathered the fat which swims on the top they wash it with water, as has been said, and straining it into an earthenware

dish containing hot water, and covering it with a linen cloth they place it into the sun until it becomes sufficiently thick and white. Some pour off the first water after a lapse of two days, and pour on fresh. Better is that which has not been treated with soap wort and is smooth, smelling of the oily wool, washed with cold water in a shallow dish, and having been bleached and containing in itself no hard or solid (particles), as for instance that which is adulterated with wax or tallow. It possesses a power to close up wounds, to soften especially with melilot and butter.”

Nicolas Culpeper, gentleman student in physick and astrology, in his *Pharmacopœia Londinensis*, published 1653, gives also a recipe for making Oesypus:—

“*The Manner of Preparing Oesypus.*—Take of wool uncleaned which was taken off from the neck, ribs, and shoulder pits of the sheep, put it into warm water oftentimes, and wash it diligently till all the fatness be come off from it into the water; afterwards press it out and lay it by, then pour that fat and filthy water out of one vessel into another, holding the vessel on high, pouring and repouring till it be frothy, then again take away the fat that swims at the top, do so so often till no more froth appear nor fat swims at top; then take the fat with the froth and wash it up and down with your hand in clear water so often and so long till the filth be washed from it, which may be known by the water remaining clear, and the fat being tasted do not bite your tongue, then keep it in a thick and clean pot in a cold place.”

There is another recipe for making the above preparation, published 1680, where it is described as free from dirt, not biting the tongue, and of white appearance.

In later *Pharmacopœias* or *Dispensatories* “Oesypus” is not mentioned; it seems it has been abandoned together with many other antique remedies, such as burnt swallows, fox lungs, man's cranium, &c.

In the year 1868, Hartmann analysed the fat obtained from wool by means of solvents and found it to consist chiefly of the ethers of fatty acids and the ethers of cholesterine with fatty acids.

Two years later, A. Schulze proved also the presence of isocholesterine ethers with fatty acids (*vide Berichte der deutschen Chemischen Gesellschaft*).

Later on Drs. Braun and Liebreich, working on raw wool-fat, so-called recovered fat, consisting, besides of the above-named substances, also of the fatty acids of the soap which is used in washing wool, took out a patent (18·2) for the purification of wool-fat, which, mixed with water, they termed “lanoline.” They described their lanoline as a combination of pure wool-fat with water.

The appearance of this preparation showed that it was far from pure, the colour of their anhydrous wool-fat was greenish-brown, and when mixed with water, dirty yellow, containing still free fatty acids, and if kept for some time, even in a closed vessel, assumed a brown colour on the surface, besides retaining the characteristic disagreeable smell of sheep. It is prepared thus:—

“Fresh lye, from wool-washing works, passes through a centrifugal apparatus in which the dirt and water are separated from each other, the raw lanoline is kneaded with cold flowing water until the water which flows away is as clear as the inflowing water.”

This process seems to be copied from the recipes of Dioscorides, Plinius and Culpeper.

“The raw lanoline is then heated with water whereby it is separated into water and fat, and the latter skimmed off. It can then be again operated on in a centrifugal apparatus, or dissolved in ether, ethylic or methylic spirits or other solvents.”

This does not seem correct, as the cholesterine ethers of the fatty acids which constitute the pure anhydrous wool-fat are insoluble in ethylic or methylic spirits.

“The solvents can afterwards be recovered. The fat so cleansed is kneaded with water, with which it combines to make a perfectly white neutral inodorous unguent. Instead of the lye, raw wool-fat emulsified with alkalis, soap, or a mixture of both is subjected to the above process.”

In December 1885 the *Fabrie Chemischer Producte* in Berlin (Messrs. Jaffe and Darmstädter) patented another

process: "In the emulsified wool-fat fat and water are separated by allowing the liquid to stand for some time, as then separation will take place spontaneously, and the upper layer consisting of the fat can then be treated as stated in the former patent, 1882. The separation also takes place and more quickly if a strong and copious jet of water is allowed to flow into the emulsion liquid. The separation may be also easily effected if, instead of soluble alkaline soaps, those of insoluble alkaline earth or heavy metals are used in emulsifying the wool-fat, but in this case the separation of wool-fat and insoluble soap is more difficult."

Other vegetable ingredients are mentioned, which tend to emulsify the wool-fat, such as pancreatine, and drugs containing saponine.

On the 8th of January 1886 the same firm applied for another patent, in which they propose to either dissolve the emulsified wool-fat in suitable solvents, such as benzene, benzol, xylol, toluol, isobutylalcohol, amylalcohol, sulphide of carbon, &c., which dissolve the fat and the existing soaps, and leave the impurities undissolved. Or to treat the raw wool-fat with such solvents as will dissolve the fat only, and leave the soaps undissolved, for instance ether and acetone. Working on raw wool-fat the fatty acids contained in the latter are saponified by alkalis, alkaline earth, or the oxides of the heavy metals, and then treated with either of the above-named solvents.

Both methods are impracticable, as the emulsified wool-fat forms, when stirred or shaken with any of these solvents, a jelly, which it is almost impossible to separate.

A patent was taken out by myself and my assistant, Mr. Ritsert, on May 7th, 1886, by which we treat the raw wool-fat with boiling alcohol, containing about 10 per cent. of ether, by which means we dissolve the free fatty acids, the free cholesterine and isocholesterine, and the volatile ethers of the free fatty acids present in the raw wool-fat. The remaining cholesterine ethers are then treated with steam in order to drive off remnants of the solvent. Or, we dissolve the raw wool-fat in ether and separate the cholesterine ethers by the addition of alcohol. Thus purified, they are mixed with about 20 per cent. of water and called by us cholesterolol.

Instead of working on raw wool-fat we may use the lyes of wool-washing works.

Wool-fat thus purified leaves much to be desired, as the presence of cholesterine ethers of higher melting point, wax-like, render it tough, and make it unfit for pharmaceutical or cosmetrical purposes.

I obtained a second patent in September of the same year, by which I use either the lyes of the wool-washing works or raw wool-fat, commercially known as recovered fat. In working with the latter, I first separate the free fatty acids by means of solvents, such as alcohol, mixed either with ether, chloroform, carbon bisulphide, acetone, benzoline, or naphtha, or by distilling the fatty acids off, preferably by superheated steam, at a temperature of 400°–450° F.

The fatty residue, after separation of the acids, is then filtered through animal charcoal, especially that known as prussiate waste, and this may be done in either of two ways: either by maintaining the fatty matter in a fluid condition by heat applied to it and to the filter, or by first dissolving the fatty matter, preferably in benzoline, carbon bisulphide, or acetone, and filtering the solution. The solvent is then recovered by distillation, and steam or warm air is blown through the fatty matter until all traces of the solvent have disappeared, when such has been used. The so purified wool-fat is anhydrous, of a yellow colour, and free from smell; if required, as basis for ointments, pomatums, &c., 20–30 per cent. of either water or a solution of peroxide of hydrogen are incorporated with it, thus producing a pure white material.

Dr. A. von Rad, of Bavaria, applied for a German patent in March 1887. In his process he precipitates the lyes of wool-washing works with slaked lime and deodorises the precipitate by means of chlorinated lime or manganate or permanganate of potash or soda. The precipitate is then dried and the wool-fat dissolved out successively by alcohol, benzene, carbon bisulphide, benzol, and a mixture of ether and chloroform, and in order to give to this fat a proper

consistency, he mixes it with neutral ethyl or methyl ethers of oleic acid and calls it "Lanesine."

Later on Dr. von Rad obtained a second patent for a process by which he heats raw wool-fat with alcohol and sulphuric acid, thereby changing the free fatty acids into the corresponding ethers.

Messrs. Singer and Judell extract the fat from wool by means of carbon bisulphide, a method tried years ago, but abandoned owing to the difficulty of rendering the wool odourless, or when driving off the solvent by heat spoiling the wool. Messrs. Singer and Judell seem to have avoided this difficulty by using a very elaborate apparatus (this Journal, 1889, 24).

In order to prepare wool for the spindles, the dirt, fat, salts, soap, &c., naturally contained in wool, must be separated from it. This is generally done by washing and scouring the wool with soap lyes, which are then called liquor, and are of a dirty, greyish, milky appearance of the smell of ordinary soapsuds when fresh, of a putrid, ammoniacal smell after standing some time, and consist chiefly of potash salts, naturally found in the fleece of sheep, of wool-fat, dirt, and soap.

Formerly this liquor was wasted: in later years it was saturated by means of acids, especially sulphuric acid, thereby recovering part of the fat. By this treatment the soap that has been used in scouring is decomposed, and its fatty acids, together with the wool-fat and dirt, form a so-called magma, from which after dehydrating the fats are obtained by hot pressure; these are then remelted, allowed to settle, and filled into casks. Till about two years ago the value of such recovered fat was 7*l.* per ton; now 11*l.*, 12*l.*, and even 13*l.* are obtained, because large quantities are used in the United States for softening leather. I am told that in the course of 12–18 months the thus greased leather deteriorates on account of the large amount of the free fatty acids present in the fat. Coarse wool contains about 7–10 per cent. of fat, whilst fine wool, such as imported from the Cape, Australia, and New Zealand, contains 30–40 and even 50 per cent.; but the liquor yields by the acid treatment only about half the quantity it contains.

By my present invention I recover all the fat as well as the potash salts, dirt, and soap used in washing wool.

The wool is twice soaked in warm water of a temperature not exceeding 110° F., and pressed or dried by centrifugal action. The water thus obtained contains the potash salts naturally present in wool, together with a small quantity of potash soap, formed by the action of potash on the cholesterine ethers and volatile acids, and also part of the dirt adhering to the wool; it is allowed to settle and then concentrated in a suitable vacuum apparatus, preferably a quadruplex Yaryan, then finally evaporated and dried, thereby obtaining the potash salts in an alkaline but impure condition; or the water is saturated with hydrochloric, sulphuric, or phosphoric acid, in order to more speedily form a sediment, and concentrated as before, thereby obtaining the potash salts as chlorides, sulphates, or phosphates. The sediment is either dried by heat, filter press, or centrifugal apparatus, or the potash salts are dried together with the dirt and used as manure. If it be desired to obtain the potash salts in a purer state, they have to be calcined, lixiviated, and recrystallised.

After concentrating the water and acidifying it the emulsion is broken up and the small quantities of fat, together with the dirt, are removed and worked up together with the concentrated liquor of the subsequent washing with soap lye.

After the first treatment with water, the wool is washed and scoured with soap lye, consisting generally of an emulsion of olive oil and potash lye, diluted with water, by which means all the fat, together with the remaining dirt, is removed and forms with the lye an emulsion; the wool is then rinsed in water, pressed, and dried. The emulsion, together with the rinsings, are as far as possible concentrated in a Yaryan apparatus and treated repeatedly with a mixture of alcohol and water, for economy's sake methylated or otherwise denaturated spirit. I take, for instance, equal parts of spirit and water, but it may be varied to three parts of spirit and two parts of water, or two parts of spirit and three parts of water.

By this treatment the emulsion is broken up and the soap dissolved in the diluted spirit, leaving behind the wool-fat and dirt; the soap and spirit are separated and recovered by distillation.

The wool-fat is then extracted from the dirt either by hot pressure or by means of solvents, such as carbon bisulphide or benzine, for instance, and the solvent recovered by distillation.

The wool-fat is then practically free from soap and dirt, and, being also free from fatty acids, forms a valuable lubricant or leather grease, or it can be further purified by my patented process described before. The recovered soap is by itself unsuitable for washing purposes, because it contains a large amount of a soap which does not give a good lather with water, besides also a small quantity of cholesterine ethers of a higher melting point, probably stearate of cholesterine, which can be separated by treatment with strong spirit.

It is advisable to filter the soap solution when cold through flannel bags; the filtered liquid consists chiefly of oleine soap and is recovered by distilling off the solvent, whilst the remnant on the filters can be made use of, after decomposition with acids, for the manufacture of candles, for instance. This remnant soap is very sparingly soluble in water, but dissolves easily in hot spirit, from which solvent it separates whilst cooling, similar to stearine soap.

I continue studying the nature of the fatty acids, of which this soap is composed, and shall later on report about it.

Sometimes it may be desired not to recover the potash salts and dirt separately; in that case the water in which the wool was soaked is to be concentrated and dried without allowing it to settle; or it may not be required to recover the soap separately. In that case the preliminary soaking of the wool can be dispensed with, and the wool-washing water is, after concentration, over-saturated with an acid, such as sulphuric acid for instance, and heated, by which means the soap is decomposed and the so liberated fatty acids of the soap, together with the wool-fat and dirt separated from the water, and recovered either by hot pressure or solvents as above stated.

Instead of concentrating the wool-washing water or liquor, common salt in sufficient quantity may be added; in this way the wool-fat, soap, and dirt, are also separated from the water, which process is quickened by heating, preferably by steam. The amount of salt required is about 12—15 per cent. of the liquor and can be used again, when recovered by concentration.

The liquor, having a specific gravity of 1.019, contains per gallon about 800 grains of fatty matter; a tank of 10 cubic feet dimension, in which the liquor is generally stored, would contain about 6,232 gallons and yield 712 lb. of fatty matter, whilst by the old way of recovering the fat, only about 300—320 lb. are obtained. For concentrating 6,232 gallons of liquor in a quadruplex Yaryan about half a ton of coals is required.

#### DISCUSSION.

The CHAIRMAN said that the thanks of the meeting were due to Mr. Langbeek for a paper so exceedingly interesting from various points of view. It was very interesting to have recalled to one's mind the ancient processes of the Greek pharmacists—who were by no means so backward as some people imagined. It was interesting, too, to be reminded that in that quaint book "*Culpeper's Pharmacopœia*," there were descriptions of processes which were still not entirely obsolete or absurd. He must confess that he was unaware of this before hearing the paper, although he thought he knew the book well, with its recipes for the manufacture of oil of puppy dogs, oil of worms, &c.; for the preservation of mummies for human consumption, and other curious things. But here, among the Greeks, and in the Middle Ages, was the knowledge of a process which had been entirely lost in modern times, until the last few years. If, 10 years ago, one had consulted a text-book on the subject of wool, the utmost information one would have got would have been that it was a pity to waste the "suint" by washing it away in a stream; that it was a valuable manure on account of the potash it contained,

&c. It was therefore very interesting to hear that this most valuable medicament had been not invented, but recovered from the oblivion into which it had fallen. It was curious to find that this lanolin, as it was now called, had, by reason of its origin, a remarkable power for penetrating the skin. If, therefore, a medicament was mixed with it and rubbed on the skin, it would be absorbed much faster than if mixed with soft paraffin or vaselin, the lanolin itself being absorbed as well as the medicament. This "dirt of wool," which a few years ago was regarded as a necessary evil, due to the scarcity of water or the conveniences for washing wool in the countries from which it was generally exported, had now come to be regarded as the source of a most useful product. Mr. Langbeek's paper, therefore, was of practical as well as scientific and historical interest. It dealt with a subject on which the information obtainable hitherto was very imperfect, and was in fact the first complete résumé of the subject he had ever seen. On this account therefore it was specially welcome.

Dr. C. R. ALDER WRIGHT had listened with great interest to the paper, the more especially because some 15 or 16 years ago he had been engaged in an endeavour to utilise the fat extracted from wool. The process, however, died in its infancy, and so brought his experiments to an end. The method he referred to was that invented by Toepler, and consisted in treating the wool with fusel oil, methodically lixiviating it, so that a concentrated solution of wool-fat in fusel oil was obtained. There were various reasons why the process was not successful. The works in which it was started were situated near Rochdale, and the perfume of the fusel oil dominated the locality and raised the ire of the residents, who, having got the idea that it was a powerful intoxicant, took measures to suppress the use of the process. But the arguments they employed on the question of injury to health were somewhat startling. It was stated that typhoid fever was caused by the exhalations of the fusel oil, and letters were written to the local papers stating that the foul odours caused all the people for several miles round to feel "drunken sick, as if on the fuddle." The results of this opposition, and the practical difficulty of obtaining fusel oil in quantity, and workmen with the requisite skill for dealing with a new manufacture, led to the suppression of the industry at an early stage. But one of the practical results was that the fusel oil solution of the fatty matters, soaps, cholesterin, isocholesterin, ethers, &c., was obtained in considerable quantities, several tons at a time. There was no great difficulty in working out a variety of products from this raw material, but whether it could be done on a commercial and paying scale had not been determined. One thing made evident was that solid fatty acids more or less mixed with unsaponifiable matters could be extracted in considerable quantity. The presence of these latter substances materially detracted from the value of the product, just as in the somewhat similar case of the "Yorkshire grease" obtained from the soapsuds of wool-scouring works by treatment with sulphuric acid, whereby a mass of fatty acids resulted mixed with unsaponifiable matters derived from the wool. One use for such material was more ingenious than desirable, namely, the adulteration of tallow used for the manufacture of soap and stearine. It was within his own knowledge that difficulties had occurred in a large stearine manufactory from the fact of their making contracts to purchase tripe tallow and such like fatty matters which were subsequently found to contain not far short of 20 per cent. of matters of an unsaponifiable character, and identified as being derived from wool-fat. He therefore felt some hesitation in accepting the statement put forward that material suitable for candles could be extracted from these substances. But as regarded the separation of the more fluid constituents, and their various uses, he thought the members owed a debt of gratitude to the author for bringing the subject before them in so neat a form, and for recalling to their recollection the ancient history of the process, as well as of various old medicaments and other bodies of curious origin. It was probably within the present century that *album Græcum* was an ingredient of the *Pharmacopœia*, substantially consisting of phosphate of lime that had passed through the body of the dog.

Personally he preferred Parrish's food, but that was a matter of taste. As to whether the exudation of the sheep's skin was materially preferable to *album Græcum*, from the sentimental point of view, must be left to personal opinion. But there could be no doubt of the correctness of the Chairman's remarks as to the value of lanolin for introducing medicaments into the system by means of external application.

Mr. HERBERT PORTER wished to ask the author a question with respect to a point which, from his practical experience of wool-washing, appeared to him to constitute a difficulty. He was quite alive to the value of the recovery of lanolin as a means of cheapening the production of wool for spinning. But there was one difficulty common to all such processes, namely, the delay in changing the wool over from the water in which it was rinsed, in order to get the fat from it, into the soap-bath. In the case of the better classes of wool, it was well known that if they got chilled before the dirt was quite removed from them, they never regained their proper colour. If, therefore, the wool had to be put into a centrifugal machine to wash out the fat, and then lifted from that into the soap-bath, unless the mechanical details were perfect there was great danger of the harm which he had indicated occurring, as he had known it to do in more than one experiment.

Mr. WATSON SMITH desired to testify to the correctness of some of Dr. Wright's remarks with respect to what might be called the illicit use of wool-grease. He had certain evidence which proved that there was a considerable excess of supply over demand in the case of wool-grease. Any information, therefore, relating to the production of this body in a useful form would be hailed with gratitude by those manufacturers who were now suffering from its illicit use in the tallow trade. Soap manufacturers had informed him that they at times received, through Hull from the Continent, parcels of tallows and fats from which they could not, by any of the recognised methods of soap manufacture, make hard soap. He had endeavoured to render professional assistance in the matter, but, after numerous practical trials, and after exhausting all the information he could give, it was found that the soap still continued to come out soft. When the manufacturers were prevailed upon to use only first-class materials, no more complaints were heard of.

Mr. H. W. LANGEBECK, in reply, said that he had had no practical experience of candle making, but he had found that the part of the by-products which would prevent soap from giving a good lather were, when decomposed by acids, of a high melting point. Different kinds of wool-grease had different melting points. In the case of ordinary wool-fat imported from France and Germany, the melting point was  $115^{\circ}$  to  $120^{\circ}$ , while that of fat from Leeds was often as low as  $90^{\circ}$ . With respect to the danger of the wool being deteriorated by several washings, he would point out that the first soaking in water was only to remove a part of the dirt and the potash salts. The larger quantity of the fat remained in the wool, and acted as a protector; and it was only by the after-treatment by the soap emulsion that this fat was removed. He had with him for inspection some wool treated by the process, and some of the wash-water in a more highly concentrated condition than it would be drawn from the Yarn. Ten gallons of liquor were concentrated by the Yarn to one gallon. He had also some of the potash salts in a purified state, and some of the ordinary recovered fat, which he had got from France. The wool-fat obtained in the North of England was generally much softer. He would also show a sample of the soap used for washing the wool, its dark colour being accounted for by the fact that it had been dissolved out by diluted spirits, which, containing acetone, form with alkalis a resinous matter of dark colour. He had some of the wool-fat free from soap, and suitable as a lubricant or a leather-grease; and also the same body more highly purified and anhydrous, viz., of light yellow colour. He was quite aware that the fat made from the by-products of wool-washing was unsuitable for giving a hard soap. It would therefore be advisable, as he had already said, to separate those soaps which were insoluble

in cold diluted spirit by filtering from those soluble in that medium, and use them, not by themselves, but in addition to soap. In that way a hard soap would be obtained. In conclusion, he might mention that the soap remaining on the filter was not soluble in water, but was soluble in hot spirits of wine.

Mr. WATSON SMITH exhibited at the close of the meeting an interesting Collection of 72 Alkaloids and Glucosides by Merck, of Darmstadt.

Meeting held Monday, 21st April, 1890.

MR. DAVID HOWARD IN THE CHAIR.

### THE OIL-FIELDS OF INDIA: NOTES ON THE PETROLEUM DEPOSITS OF BIRMA, ASSAM, THE PUNJAB, AND BALUCHISTAN.

BY ROBERTON REDWOOD, F.R.S.E.

DURING the past six months I have from time to time taken steps to put into the form of a paper the available information I have for many years collected in respect to the oil-fields of India, with the results I have obtained in the examination of typical samples of crude petroleum from that country; and as Professor Dewar and I found it impossible to carry out our intention of describing this evening our process of converting heavy oils into hydrocarbons of less density, I thought it best to complete this paper and present it as a substitute, especially as the two subjects are to some extent correlative.

Petroleum occurs in India in Upper and Lower Burma (including the Arakan islands), in Assam, in the Punjab, and in Baluchistan.

#### 1. (a.) ARAKAN.

Just south of Akyab lie the Baranga islands, and still further south the islands of Ramri and Cheduba (see Fig. II.). In Ramri petroleum occurs at Minbyin, on the western side, at Leedaung and Leikmaw, on the south-western and western coasts, respectively, and at Kyauk Phyu. It is also found in Cheduba, in the Barangas (principally in the eastern of the three islands), and elsewhere in smaller quantities. The positions of the wells are shown in the outline map (Fig. II.).

An excellent account of these islands was given in 1878 by Mr. F. R. Mallet, F.G.S., Geological Survey of India.\* The digging of oil wells in Ramri and Cheduba appears to have been an industry for a long time past. About the end of December or beginning of January, when the rice crop has been harvested, some of the villagers employ their spare time in digging wells as a means of adding to their income. The oil season lasts from that time till the rains, when the wells, which are usually sunk in, or close to, the bed of some nalka, get filled with water, and are often choked up entirely by debris washed into them. Mr. Mallet states that the wells are of two classes—those which appear to be in communication with a natural reservoir, from which the oil, generally accompanied by large quantities of gas, rises with considerable rapidity, and those sunk in rock more or less soaked with petroleum, from which the oil slowly percolates into the well. To the former class the wells at Leedaung are said to belong. One of these is about 25 ft. deep and 4 ft. square. It is lined with wood throughout. At the bottom is water covered with a stratum of oil, through which a rather large quantity of gas bubbles up, chiefly from one corner of the well. The oil is drawn morning and evening. Another well of the same kind,

\* Records of the Geological Survey of India, Vol. XI., Part 2.

about 200 yards north-west of the above, is about 40 ft. deep, with water and oil at the bottom which seem to boil gently from the issue of gas. The oil is of a pale yellow colour. Mr. Mallet was informed that these wells had been in existence since the time of the Burmese domination, and that the yield of oil had not diminished. The yield, however, was only a few gallons a day, a quantity that a large reservoir, tapped only by a small duct leading to the bottom of the well, might supply for a very long period.

As regards the future of Ramri as an oil-producing territory, Mr. Mallet makes the following remarks:—"The not unfrequent fiery eruptions from one or other of the mud volcanoes occurring in a line near Tsi Chang (near Kyauk Phyu) leaves but little room for doubt that there is a fissure beneath them in which large quantities of gas are generally stored up, and it is at least highly probable that a considerable quantity of oil is associated with the gas. A lucky boring might strike a spouting reservoir of great capacity, but of course such an undertaking would be of a speculative kind. There are no data to determine at what depth the oil is stored, nor what the inclination of the fissure may be, whether vertical or at a greater or less angle. A boring therefore sunk actually along the line of vents might miss it altogether. A few holes, however, sunk in a line at right angles to the line of vents could hardly fail to strike it if sunk sufficiently deep. The experiment would certainly be an interesting one, whether rewarded with success or not."

Mr. Mallet considers that the wells in the southern part of the Eastern Baranga are of the same class as those at Leedaung already alluded to. They are sunk in grey shale which splits with a rather smooth fracture having a slightly unctuous feel. The bedding is very nearly vertical. Particulars are given respecting the drilling of two wells in this locality. When the bore of the first well had been carried to a depth of 66 ft. from the surface of the ground, "the workmen were surprised and terrified by a sudden outburst of gas and oil, accompanied by loud subterranean sounds, as of distant thunder." The drilling had been commenced at the bottom of an excavated shaft some 30 ft. in depth, and the workmen "had only time to make their escape up the ladders of the well before the oil and gas poured in in quantities that would otherwise have made their position a very perilous one." This well is said to have yielded 1,000 gallons a day for seven days, and afterwards 120 gallons a day, but a great deal of the oil escaped from the well through fissures. In the second well oil was struck at 68 ft., and the yield was 150 gallons a day. The gas escaped with violence, "making a great noise."

Of the second class of wells those near Minbyin, in Ramri, are regarded as the most important. Here, in an alluvial plain in or near to the beds of the nullas, are several scores of wells 4 ft. in diameter, and from 5 or 6 to 10 or 12 ft. in depth, sunk in groups of a dozen or twenty in a tough grey clunch, generally covered with a foot or two of surface soil. In some wells the clunch towards the lower part is entirely soaked with petroleum, but more frequently the oil soaks the rock in patches. The wells are worked from about the beginning of December till the commencement of the wet season, when, being in the beds of nullas, they get filled with rubbish. The aggregate production is only 6,000 bottles, or about 1,000 gallons.

Mr. Mallet is of opinion that the oil-bearing rocks near Minbyin are probably at or near the axis of an anticlinal bend, a position which, as he says, has in other oil regions been found a most favourable one for well sinking. The present wells, he points out, have merely "grubbed at the surface."

Some of the more recent dug wells on this island are lined with bamboo wickerwork.

In Cheduba, according to Captain Halstead, oil is collected by turning up the soil, where oleiferous, to a depth of 2 ft., and surrounding it with a bank of earth, so as to form during the rains a shallow pond, about 20 yards square; gas and oil rise through the water, and the oil is skimmed off and collected. It is worthy of note that this was the method of collection adopted by the Indians in what are now the Pennsylvanian oil-fields.

In reference to the Arakan deposits, Mr. Medlicott,\* commenting upon the fact that so far the business of oil-winning on a large scale in these localities has not been a success, says:—"There are no doubt very large supplies of high class petroleum to be got from this region, but it must be won by suitable methods."

Allusion has already been made to the colour of the crude oil obtained in the Arakan district. Much of it has the appearance of brandy or sherry, and it can be burned in lamps in its crude state. The Baranga oil was exhibited at the Akyab exhibition in 1875, and attracted much attention. Specimens of the oil from the Eastern and Western Barangas were submitted to me for analysis in 1878.

The Eastern Baranga oil was dark brown in colour, and had a pleasant odour. Its specific gravity was .835, and I obtained from it 66 per cent. of excellent kerosene of sp. gr. .810, besides from 2 to 3 per cent. of more volatile products. Owing to the small quantity at my disposal I was not able to ascertain with any approach to precision the percentage of the solid hydrocarbons present, but I formed the opinion that about 3 or 4 per cent. of paraffin, together with lubricating oils, might be obtained from it.

The Western Baranga oil was of similar colour and odour, but of higher specific gravity, viz., .888, and it yielded only 7 per cent. of kerosene of sp. gr. .815. The residue, however, yielded an excellent lubricating oil.

About the same time I examined samples of oil from the mud volcanoes near Kyauk Phyu, and from the wells (10 to 20 ft. deep) at Minbyin. The former was an oil of pale colour, and of sp. gr. .818. It yielded 56 per cent. of kerosene of remarkably high quality, but almost the whole of the material was available for use as burning oil. The Minbyin oil had a specific gravity of .866, and yielded only 15 per cent. of kerosene of sp. gr. .810.

Portions of these samples and specimens of rock from the Barangas, saturated with petroleum, are on the table.

The following interesting comparison of the crude oil obtained in the Arakan district with that produced in Upper Burma ("Rangoon oil") will be found in Mr. Mallet's detailed description of the salses or mud volcanoes of Ramri and Cheduba.† "In this connexion the difference between the petroleum of the Irrawadi valley and of Ramri may be noticed. The mud volcanoes of the former region have been described by Dr. Oldham as very sluggish, and as never exhibiting the fiery paroxysms to which those in Ramri are subject. At the same time the oil is dark coloured, is as thick as treacle, or even solid at 60° F., being indeed often spoken of as 'Rangoon tar,' and contains paraffin to the extent of sometimes more than 10 per cent. The Ramri oils are associated with much gas, and are themselves sometimes as transparent and light coloured as brandy. They have a lower specific gravity than the above, and at 60° are perfectly mobile. Without venturing to assert that the above differences are due to a difference in the temperature at which the oils have been produced, it may be noticed that at Baku, on the Caspian, where there are mud volcanoes subject to fiery eruptions, similar to those of Ramri, the oil is in part of the same pale transparent kind, and is accompanied by immense quantities of gas."

In illustration of these remarks I have placed on the table samples of "Rangoon oil," and of the light oil obtained at Surakhany in the Baku district.

#### (b.) UPPER BURMA.

On p. 261 of the Journal of the Embassy to the Court of Ava, by Major Symes, published in London in 1795, will be found the following account of the wells from which the so-called Rangoon oil was obtained:—

"After passing various lands and villages, we got to Yenangyoung, or Earth-oil Creek, about two hours past

\* Note on the occurrence of petroleum in India, by H. B. Medlicott, Geological Survey of India. Records Geological Survey of India, Vol. XIX, Part 4, 1886.

† The Mud Volcanoes of Ramri and Cheduba, by F. K. Mallet, F.G.S., Geological Survey of India. Records, Vol. XI, Part 2, 1878.



noon. We were informed that the celebrated wells of petroleum which supply the whole Empire and many parts of India with that useful product were five\* miles to the east of this place. The month of the creek was crowded with large boats waiting to receive a lading of oil, and immense pyramids of earthen jars were raised in and around the village, disposed in the same manner as shot and shell are piled in an arsenal. This is inhabited only by potters, who carry on an extensive manufactory and find full employment. The smell of the oil is extremely offensive. We saw several thousand jars filled with it ranged along the bank; some of these were continually breaking, and the contents, mingling with the sand, formed a very filthy consistence."

Additional historical references will be found in the Journal of an Embassy to the Court of Ava, by John Crawford, published in London in 1826, and in the Narrative of the Mission to the Court of Ava in 1855, by Colonel Yule.

I have in my possession a work published in Calcutta in 1826, containing "An account of the petroleum wells in the Burma Dominions: extracted from the Journal of a voyage from Rangoon up the River Erai-Waddy to Amaralpoorah, the capital of the Burma Empire, by Captain Hiram Cox, Resident at Rangoon."

In this journal, dated January 1797, Captain Cox describes with great minuteness the petroleum industry of Burma as it then existed, and states that to him "the smell of the oil was fragrant and grateful." Captain Cox brings his narrative to a close with the following paragraph: "To conclude, this oil is a genuine petroleum, possessing all the properties of coal tar, being in fact the selfsame thing; the only difference is that nature elaborates in the bowels of the earth that for the Burmas for which European nations are obliged to the ingenuity of Lord Dundonald."

The oil district of Yenangyoung (Creek of Oil, or, literally, Creek of Stinking Water) has recently (June 1889) been officially reported upon in elaborate detail by Dr. Fritz Noetling, Palaeontologist, Geological Survey of India,† who points out that the district comprises two oil-fields situated about  $1\frac{1}{2}$  miles to the east of that place, near the villages of Twingauung (Hill of Wells) and Beme. It lies on the eastern side of the river Irrawady, and is distant about 300 miles from Rangoon or about 80 miles from the terminus of the railway at Allaunyo (see Figs. I. and III.). The country forms a tolerably level and flat plateau, rising to 260 feet above the low level of the Irrawady at Yenangyoung.‡ Through the mechanical action of water upon strata of unequal durability, mainly consisting of ferruginous conglomerate and soft sandstone, the surface has become intersected by numerous long, deep, and narrow irregular ravines. The strata unquestionably belong to the tertiary formation, and Dr. Noetling inclines to the belief that they are of no later age than Miocene. They consist chiefly of laminated and clayey sands, sometimes a little indurated so as to form soft sandstones. Some of the beds are highly calcareous, concretionary masses of sandy limestone abounding. Nodular concretions of a very hard quartzitic limestone are also found. The sandstone varies in colour from white or very pale yellow up to dark red and blue. The clays and sand-clays have a bluish-gray tint. Valuable information is furnished by the following section based by Dr. Noetling upon the records of strata perforated in the drilling of Messrs. Finlay, Fleming, and Co.'s first well at Kodaung (between the two native oil-fields, see Fig. IV.).

\* The distance is rather less than  $2\frac{1}{2}$  miles.

† Report on the Oil Fields of Twingauung and Beme, with a map and a plate of geological sections. Rangoon, June 1889.

‡ The Reconnaissance Map (scale, 1 in. to 4 miles), Survey of India, 1888, shows a line of hills running in a N.W. and S.E. direction, parallel to the course of the river, and extending from about 2 miles N.W. of Yenangyoung to about 14 miles S.E. of that place, but Dr. Noetling states that the map is incorrect, no such range of hills existing.

Thickness of the Single Beds in Feet,*	In Descending Order.	Total Depth from the Top.
3-4	Decomposed rock forming the soil .....	3-4
12-14	Soft yellow sandstone.....	16-18
11	Soft red sandstone, probably a thin stratum of soft bluish clay is imbedded	29
79	Soft sandstone of bluish-grey colour, thinly laminated, numerous beds of clay, sandy clay and sandstone alternating; occasionally thin beds of hard sandstone. The strata are feebly soaked with oil up to 50 ft. from the top .....	108
2	Hard grey sandstone.....	110
49	Bluish-grey sandstone of the same quality as before mentioned.....	159
9	Hard grey sandstone.....	159
41	Bluish-grey sandstone, the same quality as before mentioned, but probably at its base the clayey beds grow thicker.	200
20	Stiff blue clay (clunch) .....	220
40	Fine soft sand of greenish colour, frequently alternating with thin beds of dark bluish clunch, a thicker bed of which has been found at an uncertain depth, the sandstone soaked with oil ..	260
4	Hard grey sandstone.....	264
6	Soft sandstone of greenish colour soaked with oil.....	270
1	Hard grey sandstone .....	271
23	Rather hard sandstone of greenish colour alternating with beds of shale.....	294
6	Hard grey (?) sandstone .....	300
65	Soft sandstone of light greenish colour, in beds, unequally charged with oil ...	365
49	Conglomeratic (?) sandstone, or conglomerate of light greenish colour, soaked with oil .....	405

I am particularly fortunate in being able to place before you samples of the strata in question, which Mr. Kirkman Finlay has courteously procured from Burma, for the practical illustration of this paper. These samples were taken at the following depths during the progress of the drilling of the sixth well (A. No. 4).

#### Samples of Strata.

1.	35	to	75	feet from surface.
2.	75	"	139	" "
3.	139	"	160	" "
4.	160	"	185	" "
5.	185	"	190	" "
6.	190	"	244	" "
7.	244	"	272	" "
8.	272	"	280	" "
9.	280	"	330	" "

†

Dr. Noetling classifies the strata, in descending order, into four groups, of which the following are condensed particulars:—

(a.) The upper group, consisting chiefly of sandstones of brown and yellow colour, followed by similar beds of dark red and yellowish-white colour.

(b.) The second group, consisting of bluish-grey sandstones and clays alternating. The lower part of this group is fairly soaked with oil, which actually oozes out of the strata at numerous places in the ravines, but it is not to be considered as the oil-bearing formation proper.

\* In the original report there are evidently some misprints in the recorded measurements.

† A very interesting specimen, distinctly showing the oil *in situ*.



(c.) The third group, consisting of stiff blue clunch. This formation, which is usually found at a depth of 200 ft. when the well is on the top of the hill, or 100 ft. when in the ravine, immediately overlies the true oil-bearing strata.

(d.) The fourth group, consisting of the oil-bearing sandstone. These strata, the thickness of which is by Dr. Noetling considered to be as much as 200 ft., are composed of more or less soft, coarse, or fine micaceous sandstones of bluish-grey colour, changed into a more or less yellowish-green, according to the amount of oil present, interstratified with hard sandstone and blue clunch.

It is worthy of note that the clay beds thin out, and the beds of sandstone grow thicker as the depth increases, and that below a depth of 366 ft. conglomeratic sandstones are found.

Following these statements, there are in Dr. Noetling's report a number of painstaking deductions as to the occurrence and origin of the oil, but as Dr. Noetling himself frankly admits that "no opinion can be formed as to the depth at which the richest beds are to be found," that "none but the vaguest speculations could be formed," and that "bores of greater depth are certain to give further and more exact information," I venture to think that these deductions may be misleading.

The superficial area of the Twingauing oil-field, which lies between the villages of Twingauing and Enausu, is about 90 acres. The total number of the wells of all kinds, new and old, is 375, and of these 166 (44·3 per cent.) are utterly unproductive. The remaining 209 (55·7 per cent.) may be called productive, but these are divided by Dr. Noetling into "productive wells,"\* of which there are 120 (32 per cent. of the whole), and "scarcely-productive wells,"† which number 89 (23·7 per cent. of the whole).

One of the wells is 310 ft. deep (the greatest depth reached by a Burmese dug well), and another 305 ft.; the majority of the finished producing wells do not, however, exceed 250 ft. deep, the difficulties of digging beyond this depth both on account of the presence of petroleum vapour and because of "caving" being very great. It follows, therefore, that these wells drain but a small depth of the oil-bearing sandstone.

The production of the wells varies considerably. The 89 "scarcely-productive" wells probably do not yield more than 10 viss per diem each, on the average. On the other hand, the average daily yield of the "productive" wells may be considered to be not less than 80 to 90 viss, and is probably more than that amount. Some of the wells, however, appear to yield from 100 to 300 viss per day, and one is stated to produce 500 viss. It is obviously very difficult to obtain accurate information as to the production, and the data collected by Dr. Noetling appear scarcely to warrant all the conclusions which he draws from them. It is, however, evident that, as might have been expected, the deeper wells are the more productive.

The wells are shafts 4 to 4½ ft. square. Over the mouth of the well a cross-beam on uprights is erected, and in the centre of this is a drum and axle fashioned out of a single piece of wood and running in naturally grown fork-shaped supports. Over the drum passes a leather rope used to lower and raise the workmen or the earthenware pot (yenanoie) in which the oil is collected. If possible, the well is so situated that the men or women who are drawing up either the pot filled with oil or the digger, walk down an inclined plane along the slope of the hill. The instrument used in digging is about 4½ ft. in length, and consists of a wooden handle deeply notched 6 in. below the top, and provided with an iron shoe. In working, the digger grasps this tool with both hands a little above the shoe, and leaning the notch against his shoulder forces the instrument into the ground, and thus loosens small pieces of the rock, which are brought to the surface in a basket. The shaft is roughly lined throughout with timber. The workman is lowered in a rope sling, and as no artificial light can be used in the well owing to the presence of inflammable gas

or vapour, his eyes are bandaged before his descent, so that time may not be lost in his becoming accustomed to the comparative darkness in which he has to work when the well has reached a considerable depth. The presence of petroleum vapour renders breathing difficult, and Dr. Noetling found that the maximum time during which a young and strong man was able to remain at work was 290 seconds.

As an illustration of the difficulties under which the work is conducted, I may mention that an adventurous European with whom I am acquainted informed me that he caused himself to be lowered into one of the deep native wells, and was so quickly overcome by the gas that he became insensible before he reached the bottom, and did not regain consciousness until some time after he had been drawn up.

I am surprised that some arrangement for ventilating the wells, such as is compulsory in Galicia, is not adopted. Even in the Japanese oil-fields, as I have pointed out on another occasion,\* it is customary to employ a primitive form of blowing apparatus to supply air to the men employed in digging the wells.

As a rule four men are employed in the digging of a well. The oil is drawn up in the special earthenware pot already alluded to, suspended by a cord fastened round the neck, and is poured into another earthenware vessel of the same shape, holding from 10 to 16 viss. Twelve of these are packed in each country cart for conveyance to the riverside.

The whole area of the Beme oil-field is about 35 acres, and the total number of wells does not exceed 151. Of these not more than 72 (47·6 per cent.) are productive. Fifty of the productive wells yield more than 20 viss per day, and 22 less than 20 viss per day, the daily average amounting to 60 to 70 viss. The depth of the Beme wells is not as great as that of the Twingauing wells, and, according to Dr. Noetling, their yield is smaller, not a single well producing more than 165 viss per day, while those giving more than 100 viss are scarce.† The wells are, however, of greater age.

The wages paid for digging range from 1 rupee 5 annas to Rs. 10 per attauing,‡ according to the depth reached. The total cost of sinking a well in the Burmese fashion is from Rs. 1,000 to Rs. 1,500, and the time occupied in the operation is from 8 to 12 months. Each twinza has a right to the ground immediately surrounding his well for a distance of 14 enbits.

Messrs. Finlay, Fleming, and Co. inform me that they estimate the present total production of the Yenangyoung fields at 200,000 to 250,000 gallons per month.

The general configuration of the Yenangyoung oil-fields is shown in the sketch-map, Fig. IV., which is reduced from the map accompanying Dr. Noetling's report. The ground, as will be seen, is very broken, being intersected with choungs or ravines, some of which are from 200 to 250 ft. wide and from 100 to 150 ft. deep. This renders the transport of machinery laborious and expensive. The only wells which have as yet been drilled are those of Messrs. Finlay, Fleming, and Co.; these are located at Kodoung, which, as will be seen, lies about midway between the Twingauing and Beme fields. Drilling was commenced here in 1887, and in all nine wells have been put down, the first two by Canadian drillers, and the remainder by Pennsylvanian drillers, of whom a competent staff, provided with all the modern appliances, have now been at work about 15 months. The first two wells were not successful, one having been abandoned in an incomplete state, and the other yielding but a very small quantity of oil. Of the seven drilled by the present staff, one is a "dry hole," and another yields only a plentiful supply of water containing so much saline matter, largely consisting of magnesium sulphate, that it cannot be used for the steam

\* Yielding from 20 viss upwards per diem. 1 viss = 3·67 lb. avoird.

† Yielding less than 20 viss per diem.

\* Cantor Lectures on Petroleum and its Products. Society of Arts.

† These figures are of more recent date than the records of production included in the tabular statement relating to the Beme wells, to which I shall presently have to direct attention, and the differences between the two estimates are probably due to an actual diminution in the yield of the wells.

‡ One Tawng or attauing = 18 in.

boilers.\* Of the remaining five wells, the depth of which ranges from 225 to 412 ft., it will be sufficient for me to say that the production varies from 3 to 15 barrels per well per 24 hours. Even putting the first two wells out of account, these, it may be said, are not very encouraging results to have achieved during the 15 months that the present staff have been actively at work. It must, however, be borne in mind that in every new oil district there is much to learn, and it often happens that considerable time is occupied in determining what is the method of working best suited to the requirements of the case. Apart, however, from the difficulties attending all pioneer enterprises, there are, no doubt, special obstacles to rapid progress. Until recently, the country round the wells was overrun by dacoits, and at one time the Burma Oil Company had to suspend operations, having had several of its men killed and wounded. Even now the work is carried on under the protection of a squad of armed native troops, who guard the wells day and night. One great cause of delay and expense is the tendency to "caving," which seems very marked. Another trouble has been the scarcity of water, the necessary supplies, not only for drinking, but also for the steam boilers, having to be carried from the river, a distance of nearly 2½ miles. Messrs. Finlay, Fleming, and Co. are, however, just completing a pipe line for pumping water up from the river and oil down to it. As regards the future of the fields, Messrs. Finlay, Fleming, and Co. appear to entertain no doubt whatever of the existence of considerable quantities of petroleum, but their experience is that the yield per well is comparatively small and the cost of drilling very great. In this connexion it should, however, be noticed that the wells yet drilled are of little depth, and that only

what may be regarded as the first oil-sand has been tapped. No one can say what may be found at greater depths, but it is at least possible that far more productive oil-bearing strata may be met with.

Messrs. Finlay, Fleming, and Co. are now making arrangements to drill deeper, in the hope of striking a second oil-sand, and very great interest and importance will attach to the results obtained. I may add, that no flowing well has yet been obtained in this district.

I have already stated how the oil from the native wells reaches the riverside. There it is poured from the earthenware vessels either into the native boats or into the flats\* belonging to Messrs. Finlay, Fleming, and Co., in which it is conveyed to Rangoon by the steamers of the Irrawaddy Flotilla Company. One of the flats is shown in the view of the riverside at Yenangyoung; and, through the kindness of Mr. Kirkman Finlay, I am able to place before you a model of one of the native boats in which a small proportion of the crude oil is carried up river in bulk, for use in lamps of primitive character. The oil is worth about 1½d. per gallon wholesale, delivered at the riverside, but is to some extent retailed to the natives at a very much higher price.

On the subject of the physical and chemical characters of the oil, Dr. Noetling mentions that he had no opportunity of making the necessary examination, and he adds that he considered this "of little importance, as a sufficient supply of samples is easily to be got at any time, and can be examined everywhere." Under these circumstances, I am specially glad that I am able to submit for your inspection a representative series of samples of the crude oil which Messrs. Finlay, Fleming, and Co. kindly forwarded to me a few months ago. These include crude oil from:—

Locality.	Specific Gravity.	Setting Point.	Flashing Point (Abel Test).	Viscosity by Redwood's Viscometer, § Rape Oil at 60° F. = 100.
Yenangyoung (from Twinzats wells) .....	*887	° F. 82	° F. 110	At 90° F. 10.21
Do. do. do. ....	*837	Remains fluid at 0° F.	150	25.86
Do. (from Finlay, Fleming, and Co.'s old No. 1 bore) .....	*869	80	62	..
Do. (from Finlay, Fleming, and Co.'s American bore No. 2, at a depth of 260 ft.) .....	*870	78	80	..
Do. (from Finlay, Fleming, and Co.'s American bore No. 4, at a depth of 272 to 330 ft.) .....	*875	82	83	10.67

‡ Twinzats are native well-owners.

§ Viscosimetry or viscometry. This Journal, 1887, 121 and 148.

It will be noticed that the whole of these oils are opaque in bulk, and of dark greenish colour by reflected light. They possess but little odour, and what there is cannot be considered offensive. Owing to the presence of an unusually large proportion of solid hydrocarbons, many of the samples, as will be seen, are solid at common temperatures.

There is a remarkable absence of uniformity in the character of the oil obtained from the native wells, even where the wells are contiguous and of the same depth. This is very clearly shown in the tabular statement relating to the wells in the Beme field on the following page.

These results are very instructive. They distinctly indicate that there are at least two descriptions of oil, viz., one which has a high specific gravity and viscosity, but containing practically no solid hydrocarbons, and another of much lower specific gravity, but containing a very large proportion of paraffin.

Thus wells 26, 27, 28, 31, and 33, the depth of which ranges from 100 to 160 cubits, yield oils ranging in specific gravity from .915 to .925 which contain little or no paraffin; while, for example, wells 54 to 61 inclusive, the depth of which is from 110 to 115 cubits, yield oils of sp. gr.

.870 to .875 which are very rich in paraffin. Wells 62, 64, and 76 yield oils of .950 to .956 containing no paraffin, but these wells are so shallow that they probably yield only surface oil, though it may be pointed out that well 49, which is of about the same depth, gives an oil of sp. gr. .905 which is very rich in paraffin. To a large extent, no doubt, the lower density of the oils containing paraffin in large quantity is due to the fact that the specific gravity of the solid hydrocarbons is not as great as that of the oils in which they are dissolved, for it has been shown by Beilby† that paraffin in solution in a mineral oil has practically the same specific gravity as when in the molten condition.

I may mention in this connexion that I have found the coefficient of expansion of Burma crude oil containing much paraffin to be considerably lower than those of ordinary American and Russian petroleum.

The heavier oils, containing but little paraffin, are to some extent separately collected and employed in the manufacture of lubricating oil of high viscosity.

The occurrence of oils in this field containing no paraffin is an interesting and important fact which has not, to my knowledge, hitherto been recorded, the Yenangyoung petroleum being always described as extremely rich in paraffin.

\* Since this was written, telegraphic information has been received, and kindly communicated to me by Mr. Finlay, to the effect that this well has been deepened to 675 ft., and now yields a fair quantity of oil, though much difficulty is experienced in excluding the water.

\* These carry about 100,000 gallons in bulk.

† Jour. Chem. Soc., XLIII., 388.

I have exposed the heavy crude oil from this district to a temperature of 0° F. without causing solidification or even the slightest separation of solid hydrocarbons. In this respect the oil in question resembles much of the petroleum obtained in the Baku oil-field.

No. of Well.	Depth.	Daily Yield.	Sp. Gr. of Oil at 60° F.	Remarks.
	Cubits*	Viss†		
1	122	10	*900	Colour very dark.
2	102	10	*860	
6	112	15	*890	Rich in paraffin.
8	155	30	*882	Do.
9	156	80	*877	Do.
10	118	15	*860	
11	117	4	*867	
12	140	5	*862	
13	150	60	*875	Little paraffin.
14	150	70	*890	Rich in paraffin.
15	150	20	*885	Do.
17	145	50	*872	Moderate quantity of paraffin.
18	155	50	*877	Very rich in paraffin.
19	157	20	*880	Do.
20	155	45	*880	Do.
23	150	15	*890	Do.
25	50	50	*890	Do.
26	140	10	*915	Little or no paraffin.
27	120	8	*915	Do.
28	110	2	*920	Do.
31	100	8	*925	Do.
32	160	110	*882	Rich in paraffin.
33	130	2	*925	Little or no paraffin.
34	110	10	*892	Rich in paraffin.
35	100	25	*902	Small quantity of paraffin.
36	80	20	*890	Very rich in paraffin.
37	65	10	*890	Do.
38	50	160	*883	Rich in paraffin.
39	40	150	*881	Do.
40	10	100	*900	Do.
41	67	160	*880	Very rich in paraffin.
42	120	145	*890	Do.
43	15	90	*882	Do.
44	40	120	*875	Small quantity of paraffin.
45	50	140	*880	Very rich in paraffin.
46	45	150	*890	Do.
48	60	150	*872	Do.
49	5	110	*905	Do.
50	10	110	*926	Little or no paraffin.
51	20	145	*868	Rich in paraffin.
53	5	57	*900	
54	140	155	*875	Very rich in paraffin.
55	25	140	*872	Do.

\* 1 cubit = 18 to 20 in.

† 1 viss = 3·65 lb. avoirdupois.

No. of Well.	Depth.	Daily Yield.	Sp. Gr. of Oil at 60° F.	Remarks.
	Cubits	Viss		
56	25	140	*874	Very rich in paraffin.
57	25	120	*871	Do.
58	25	120	*872	Do.
59	90	140	*871	Do.
60	10	130	*870	Do.
61	45	110	*873	Do.
62	4	30	*956	No paraffin. Oil of high viscosity.
63	5	30	*919	Little or no paraffin.
64	4	30	*950	Do.
65	5	83	*919	Do.
66	210	120	*886	Very rich in paraffin.
72	80	140	*881	Do.
73	80	140	*881	Do.
74	40	140	*890	
75	5	80	*949	No paraffin. Oil of high viscosity.
76	3	50	*956	Do.
77	40	140	*894	Very rich in paraffin.
79	70	137	*904	Do.
80	60	145	*890	Do.
81	60	140	*880	Do.
84	30	110	*884	Rich in paraffin.
86	40	80	*890	Very rich in paraffin.
88	40	100	*876	Rich in paraffin. <sup>66</sup>
89	7	120	*880	Do.

The great variation in the quality of oils obtained from neighbouring wells of the same depth appears to indicate that considerable uncertainty must, for the present at any rate, necessarily prevail as to the description of oil obtainable from a drilled well in this locality.

It is worthy of note that the oil obtained from Messrs. Finlay, Fleming, and Co.'s drilled wells is not of lower specific gravity than that yielded by some of the dug wells of the natives. This is not in accordance with experience in other petroleum-producing countries, and in this connexion it may be noticed that the No. 4 drilled well gives at somewhat greater depth slightly denser oil than the No. 2 drilled well. It may, however, be found that still deeper wells will give lighter oil than any yet obtained in this locality, presuming, of course, that oil-bearing strata are found at considerably greater depths, of which, at present, there is no direct evidence.

I have subjected a portion of the sample from No. 2 well to fractional distillation, and I find that under atmospheric pressure it begins to distil at 260° F., but less than 30 per cent. distils within the range of the mercurial thermometer. The specific gravity of the first tenth by volume is ·779. The total distillate amounts to 95 per cent., and the last half of this solidifies at 50° F. Twenty-seven and a half per cent. by volume (equal to about 26 per cent. by weight) of kerosene of sp. gr. ·823 and flashing point 73° F. is obtainable, and about 1·4 per cent. of the more volatile hydrocarbons has to be eliminated in order to get an oil of this flashing point. The kerosene is easily refined and is of good quality. The heavy oil contains paraffin amounting to from 10 to 12 per cent. of the crude oil, and the carbonaceous residue, when the distillation is conducted to dryness, amounts to 2·15 per cent., the loss (incondensable gases, &c.) being equal to 2·75 per cent.

By the adoption of the process devised by Professor Dewar and myself (which we hoped to have been able to describe this evening), it has been found practicable very

largely to increase the yield of burning oil (kerosene) of low specific gravity. We have, in fact, succeeded in obtaining from 70 to 80 per cent. of this product (a sample of which is on the table) from Messrs. Finlay, Fleming, and Co.'s crude oil, but we shall be in a better position to discuss this point when we have completed further experiments on a large scale.

The native Rangoon oil industry is well known to be one of great antiquity, but until within the last 35 years the oil was used in the crude state. In 1854 Mr. Warren de la Rue obtained a patent\* for improvements in treating products from petroleum, the crude oil with which he experimented being imported from Rangoon. For some time afterwards the refining of this material was carried on by Price's Patent Candle Company, and by Messrs. Charles Price and Co. In 1857 Messrs. De la Rue and Müller published an "Account of the Chemical Examination of Burmese Naphtha or Rangoon Tar,"† and on the 26th March in the following year, the Rev. John Barlow, vice-president and secretary of the Royal Institution, described at a meeting of that institution the "mineral candles" and other products manufactured at Belmont and Sherwood.‡

Messrs. De la Rue and Müller state that the raw material which they examined has, at common temperatures, "the consistence of goose-fat; it is lighter than water, has usually a greenish-brown colour, and possesses a slight odour, peculiar, but not disagreeable. For the purpose of the investigation several tons of the oil was carefully collected at the source and transmitted to Europe in well-secured vessels, and even this large supply was not found sufficiently ample for the complete examination of several constituents such as organic bases, "the presence of which in minute proportion was established beyond a doubt."

The raw material was distilled in a current of steam (first of a temperature of 212° F., and subsequently superheated), and thus furnished 96 per cent. of volatile products, fluid and solid. Steam at 212° F. volatilised 11 per cent. of oil perfectly free from solid hydrocarbons; between 230° and 293° F. 10 per cent. of a further distillate almost free from solid hydrocarbons was obtained. On raising the temperature of the steam to the fusing point of lead, an additional 20 per cent. distilled over, and this retained its fluidity at 32° F., although it contained an appreciable quantity of solid matter. At this stage of the process the products of distillation began to solidify on cooling, and about 31 per cent. of a crystalline material was obtained sufficiently consistent to be submitted to pressure. After that the consistency of the products of distillation began to diminish, and at a considerably higher temperature 21 per cent. of a mixture of solids and liquids distilled, the latter predominating, especially towards the latter part of the operation. In the last stage of the process the distillate completely changed its character, becoming very dark in colour, of a pitch-like consistence, and exhibiting scarcely an indication of the presence of crystalline matter. When this product, which amounted to about 3 per cent., had passed over, there remained in the still a coke-like mass, which contained a small quantity of earthy impurities. By exposing the distillates obtained beyond the temperature of 293° F. to a freezing mixture, nearly all the crystalline matter solidified and became removable by means of filtration and pressure. It was thus ascertained that the raw material operated upon contained from 10 to 11 per cent. of solid constituents (paraffin).

The solid product was purified by being subjected to the action of boiling concentrated sulphuric acid and then washed, first with an alkaline solution, then with water. On redistillation it was obtained quite white.

In order to purify the liquid products they were twice redistilled in a current of steam. They were then subjected to fractional distillation, but it was found impossible thus to separate the various hydrocarbons, notwithstanding the

large quantity of material operated upon, and recourse was had to the action of sulphuric and nitric acids. The proportion removable by these acids was found to be in most cases relatively small, but it increased with the boiling point of the fluid, varying from less than one-tenth to nearly one-third. In this manner the nitro-compounds of various members of the benzene series were isolated.

The novelty of the substances described was stated by Mr. Barlow to consist in the material from which they were obtained, the process of manufacture, and their chemical constitution. The raw material was described as the semi-fluid naphtha drawn up from wells sunk in the neighbourhood of the river Irrawady, in the Burmese Empire, and it was stated that this material was used by the natives as lamp-fuel, as a preservative of timber against insects, and as a medicine. The crude oil was imported in hermetically-closed metallic tanks. The physical resemblance of the products to those previously obtained by Reichenbach, Christison, Gregory, Reece, Young, Wiesman (of Bonn), and others from peat, coal, &c. was referred to, but it was pointed out that the latter were obtained by the decomposition of the raw material, while De la Rue's process from first to last was one of "simple separation without chemical change."

In the process of manufacture as carried out by Mr. George Wilson at the Belmont and Sherwood works, the crude oil was first distilled with steam at 212° F., about one-fourth being thus separated. By a second or even a third distillation the distillate was fractionated into products ranging in specific gravity from .627 to .860, and in boiling point from 80° F. to upwards of 400° F. These products were all colourless and free from solid hydrocarbons. The vapour of the most volatile was found to be highly anæsthetic. The more volatile distillates were commercially employed for detergent purposes under the name of Sherwoodole, and the less volatile as lamp oil. A small percentage of products of the benzene series was obtained.

The residue of the distillation with steam at 212° F. (amounting to three-fourths of the whole) was fused, treated with sulphuric acid, washed, and distilled with superheated steam. The boiling points of the oils thus obtained ranged from 300° to 600° F. The distillate from 430° F. and upwards contained the solid paraffin, for which the commercial name of Belmontine was proposed. The oils from which the paraffin was separated were found to possess valuable lubricating properties.

In 1865—66 Messrs. C. M. Warren and F. H. Storer,\* in the United States, obtained from Rangoon petroleum the olefines  $C_{10}H_{20}$  to  $C_{13}H_{26}$ ; also probably  $C_5H_{12}$ , and the paraffins  $C_7H_{16}$  to  $C_9H_{20}$ . They also found naphthalene, and their experiments indicated the probable presence of xylene and cumene. In this investigation only the more volatile portions of the crude oil were dealt with, and the results obtained appear to indicate a preponderance of olefines over paraffins.

The developments now in progress in the oil-fields of Upper Burma may therefore be regarded as the revival of an old industry under the altered conditions of improved methods of obtaining the crude oil, and the adoption of the principle of manufacturing the commercial products at the port of shipment, a principle which prevails in competitive petroleum industries. This revival dates from 1870, when the Rangoon Oil Company, Limited, erected a refinery at Rangoon. The oil wells being, however, situated in the King of Burma's territory, great difficulty was experienced in obtaining supplies of the raw material at prices which admitted of carrying on a profitable industry, and in 1876 the company was wound up. Shortly after the annexation of Upper Burma in 1886, the work having been carried on under great difficulties in the interval of 10 years by Messrs. Finlay, Fleming, and Co., the Burma Oil Company, Limited, was formed, and at once proceeded with the extension of the refinery on the bank of the river at Dumdledaw, close to Rangoon.

\* "Improvements in treating products arising from the distillation of a certain tar or naphtha, to render the same suitable for dissolving or removing fatty or resinous substances."

† Phil. Mag. (4) XIII., 512; Proc. Roy. Soc. VIII., 221.

‡ Proc. Roy. Inst. II., 506.

\* Mem. Am. Acad. IX.

This factory is capable of refining about 500,000 gallons per month, but at present the total yield of the oil-fields is less than half that quantity. The crude oil is found to yield on the average about 27 per cent. of fair standard kerosene, but the company manufacture principally a burning oil of higher specific gravity for use by natives as well as Eurasians, and of such oil a much larger percentage is obtained. The sample of this product on the table has a specific gravity of .840 and a flashing point of 121° F. (Abel test). Much of the crude petroleum from the Yenangyoung field contains, as already stated, from 10 to 12 per cent. of solid hydrocarbons, but it is important to note that, in consequence of the unfavourable conditions under which the work is necessarily

conducted, Messrs. Finlay, Fleming, and Co. do not practically obtain from the average raw material more than 4½ per cent. of paraffin, samples of which in the crude and refined states are before you. The "melting point" (English test) of the former is 125½° F., and of the latter is no less than 132° F. The company has a candle-making department, but finds it impossible to compete against the Dutch stearin candles, which are sold at an extremely low price, and the paraffin is accordingly exported to London. The other products include "naphtha" of sp. gr. .813 and flashing point 67° F. (Abel test) as well as intermediate and lubricating oils. I find that samples of the latter which are on the table furnish the following results on examination:—

	Specific Gravity at 60° F.	Flashing Point.		Viscosity.* Rape Oil at 60° F. = 100.		Cold Test.
		Closed.	Open.	At 70° F.	At 140° F.	
Lubricating oil (3) .....	.920	° F. 238	° F. 266	34.23	9.24	Ceases to flow at 30° F.
Lubricating oil (4) .....	.930	330	354	73.76	12.47	Ceases to flow at 30° F.
Lubricating oil (5) .....	.931	330	360	121.02	15.71	Ceases to flow at 42° F.
"Valvoline" (6) .....	.949	400	422	25.95 (at 200° F. .02)		Ceases to flow at 45° F.

\* By Redwood's Viscometer.

The colour of the first sample (3) by Lovibond's tintometer, in a 2-in. cell, series 500, is 130; and of the second sample (4) is 224. The specific gravity of these oils is in each case very high in relation to the viscosity, and the viscosity is affected to somewhat more than the average extent by alterations in temperature.

More than once during the past few years I have had occasion to report upon the commercial products obtained by the Burma Oil Company, and I am in a position to state that the difficulties which at first attended the manufacture have to a large extent been overcome. The extreme heat of the climate, however, is a great drawback, and necessarily renders costly the operations connected with the separation and purification of the paraffin.

The only other locality (see Fig. III.) in Upper Burma where petroleum has been actually collected in notable quantity is Pagan-Kyet, about 10 miles above Pagan, or about 50 above Yenangyoung, on the opposite or west bank of the river Irrawady. Here there are 14 wells which about a year ago were officially stated to yield about 8,000 viss of oil per month. For some time past Messrs. Finlay, Fleming, and Co. have refined the produce of these wells together with the Yenangyoung oil at Rangoon, but the yield of the Pagan wells has been steadily diminishing and is now very small. The firm in question have, however, obtained a concession and are about to commence drilling in the Pagan oil-field. I find that the oil from this locality, a sample of which is on the table, has a specific gravity of .837, a setting point of 60° F., and a viscosity of 5.91 at 90° F. (rape oil at 60° F. = 100). It is therefore of considerably less density than the Yenangyoung oil, and it yields a larger percentage of kerosene, but a very much smaller percentage of paraffin.

At Minbu, about 18 miles below Yenangyoung on the opposite or west bank of the river, petroleum also occurs, but the deposits have never been commercially worked, though here, as near Pagan, Messrs. Finlay, Fleming, and Co. have arranged to drill. The oil occurs at Minbu, just as it does in the island of Ramri, in the immediate neighbourhood of mud volcanoes. The general character of the locality is clearly shown in the series of four photographs for which I am indebted to Mr. Finlay. The oil springs are in a valley running north and south, bounded on the east side by a range of hills behind Minbu cantonment, and on the west by low hills and broken ground covered with cactus and scrub as at Yenangyoung. Photograph No. 1 shows a sort of tank made by the natives at the upper end of this valley. In

this tank gas and oil are constantly bubbling up, and around it are innumerable small springs where gas is freely emitted and may be lighted with a match. Photograph No. 2 represents an active mud volcano about half a mile from the oil springs already referred to. The conical hill has been formed by the ejected mud, and at intervals of a few seconds there is a distinctly audible outburst of gas accompanied by a little water and mud. The gas can be ignited at the apex of the cone. The mud consists of a very fine bluish coloured clay mixed with a little gravel. Photographs Nos. 3 and 4 represent other similar volcanoes situated at a distance of 200 yards and 100 yards respectively from the one already described. It is said that a Burman commenced digging a well in this spot many years ago, but had to desist "as it was too hot for him."

Among the other specimens before you are samples of the mud from these volcanoes containing petroleum, and of the oil from the neighbourhood partially freed from sand.

Petroleum is found in various parts of the Yaw country (west of Pagan), where it is collected to some extent in pits 3 or 4 ft. deep. This oil is said to resemble Arakan oil, but I have not seen a sample of it. Petroleum is also reported to have been observed in the Chindwin Valley, which lies due north of Pagan.\*

Other localities in which there are promising indications are at Padaukpen, west of Thayetmyo (where a small quantity of oil slightly lighter than the Pagan oil is obtained), and at Yenatong, west of Myanong. These are both in Pegu, and cannot therefore be included in the Upper Burma deposits.

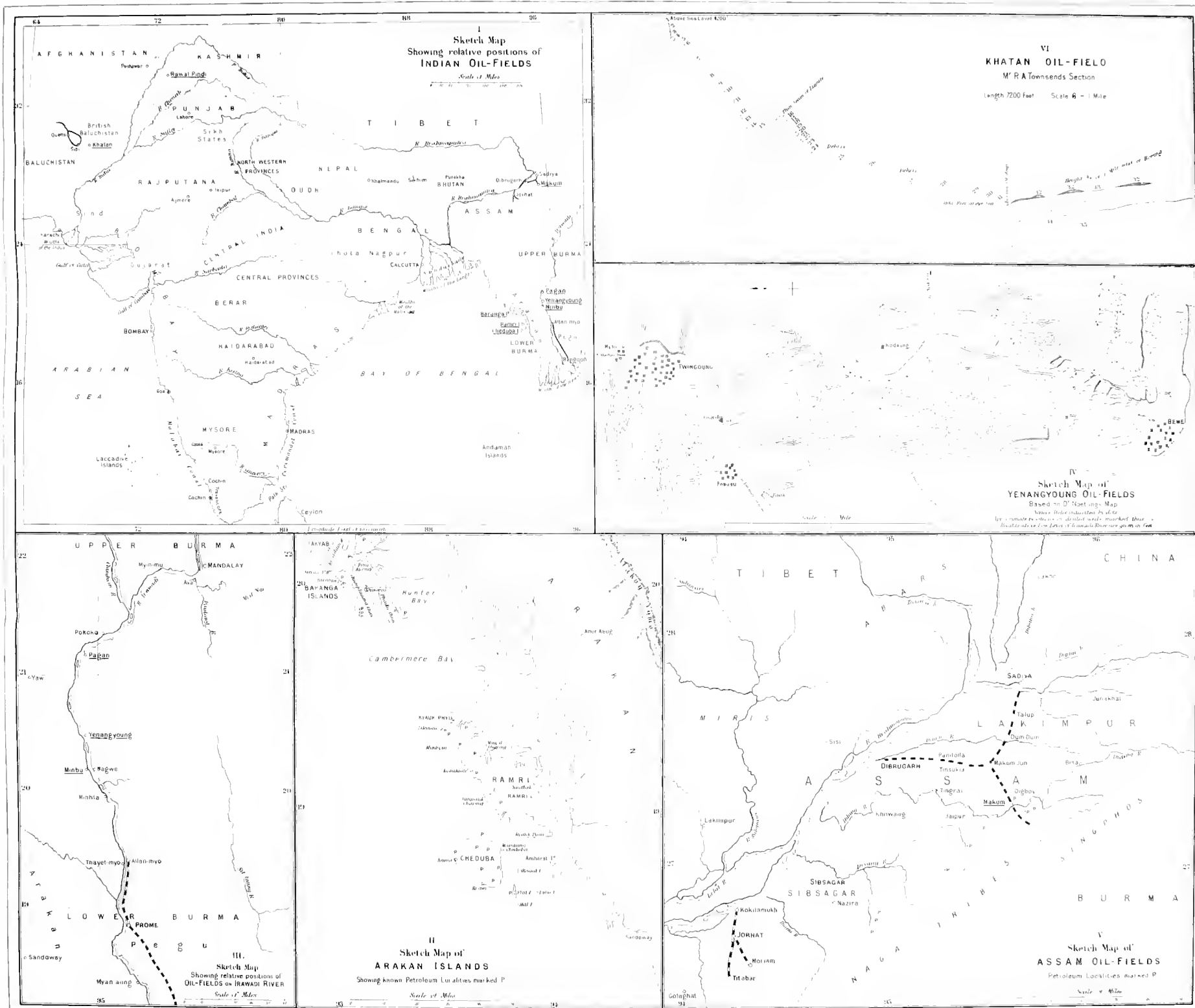
I cannot conclude my remarks on the oil-fields of Upper Burma without a cordial expression of thanks to Mr. Kirkman Finlay, of the Burma Oil Company, Limited, who, in the course of a lengthened residence in that country, has acquired an intimate practical knowledge of the subject, and has kindly furnished me with much valuable information and a large number of highly instructive illustrative specimens.

I should also like to take this opportunity of saying that our knowledge of the various petroleum deposits in India has been very effectively popularised by the pen of Mr. Charles Marvin, and I have no doubt that the intelligent

\* It was reported in Yenangyoung, on the 26th February last, that Dr. Noetling had found springs of oil resembling those of Minbu at a place called Indin, some 50 miles inland from the Chindwin river (see Fig. III.).

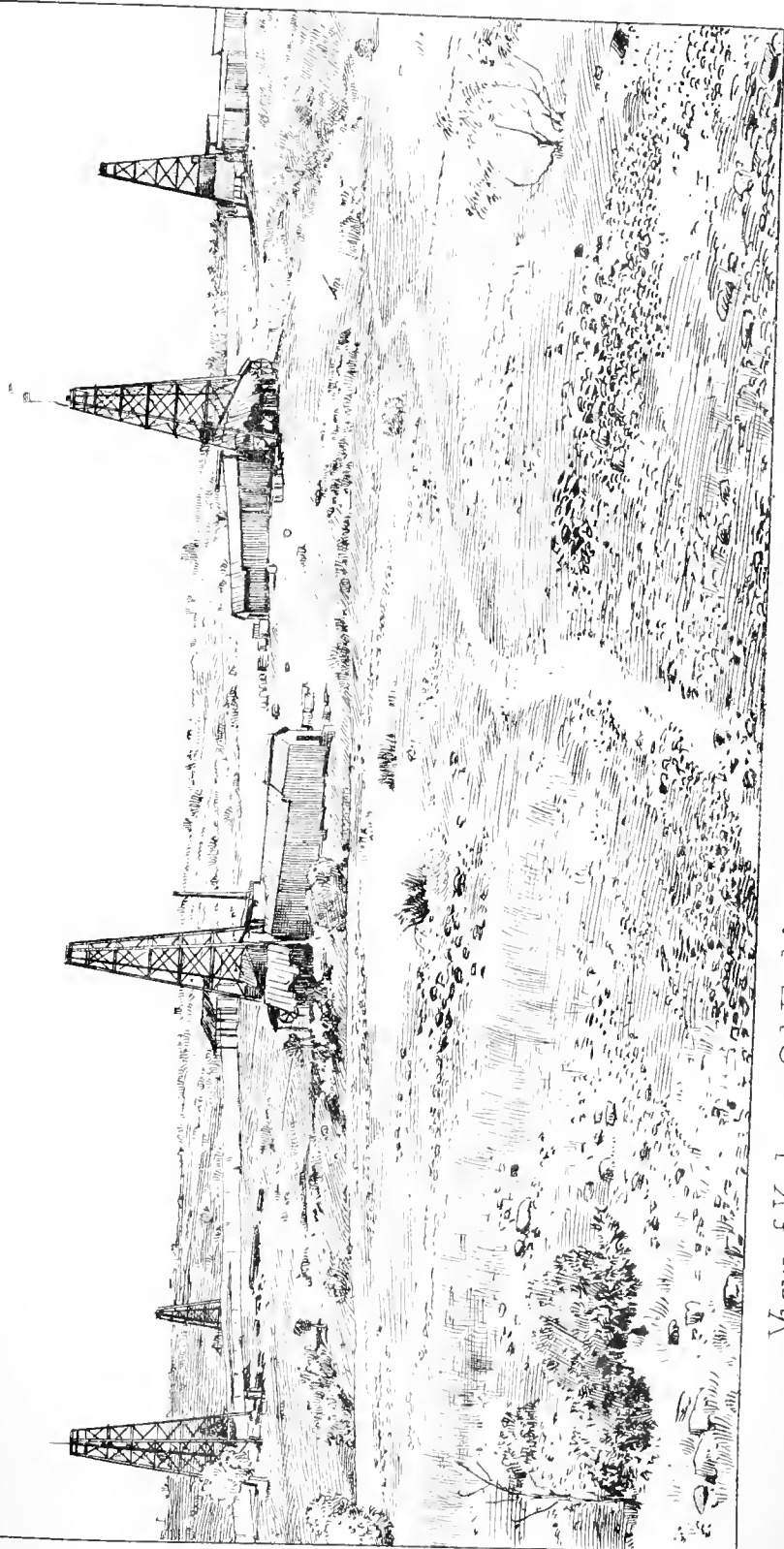
SKETCH MAPS TO ILLUSTRATE PAPER ON  
OIL-FIELDS OF INDIA.  
by Boverton Redwood

366



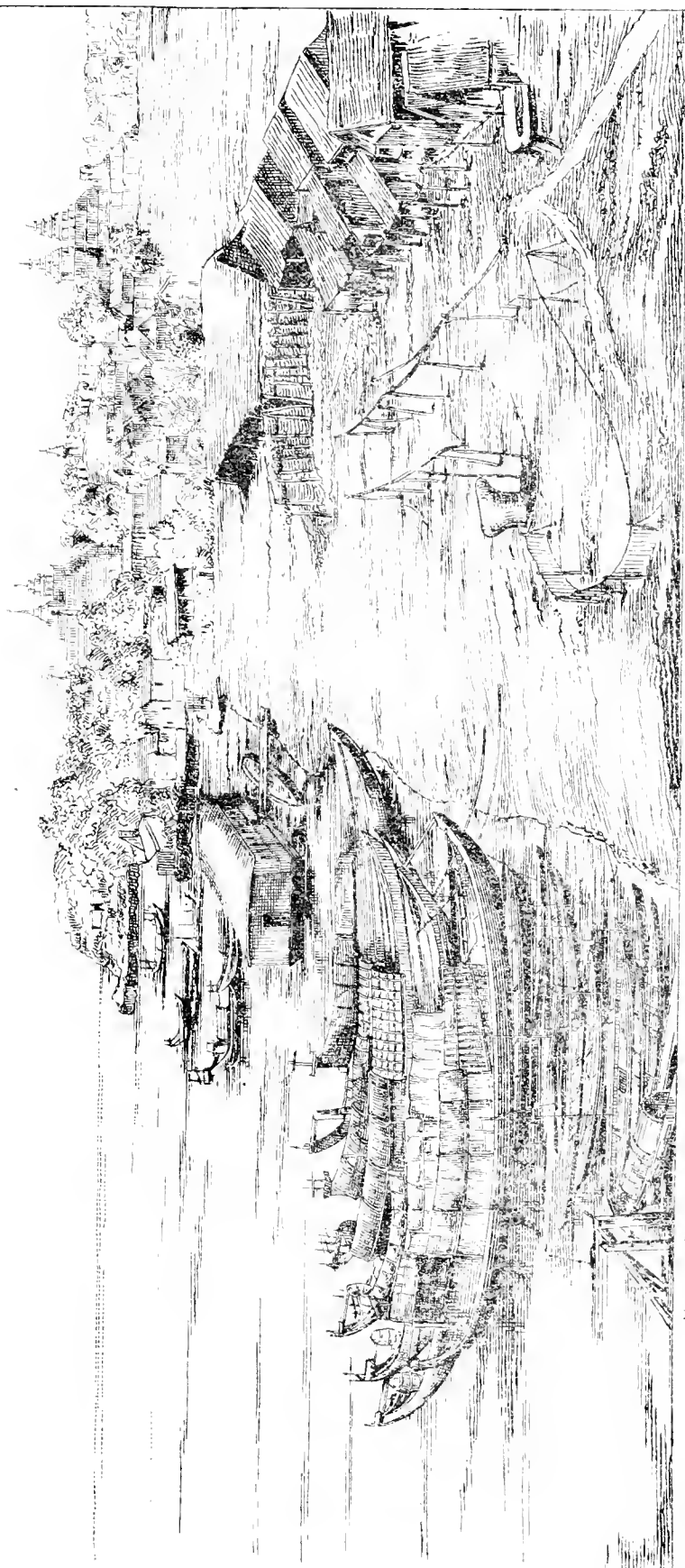






View of Kodung Oil Field, shewing derricks used in drilling the wells.



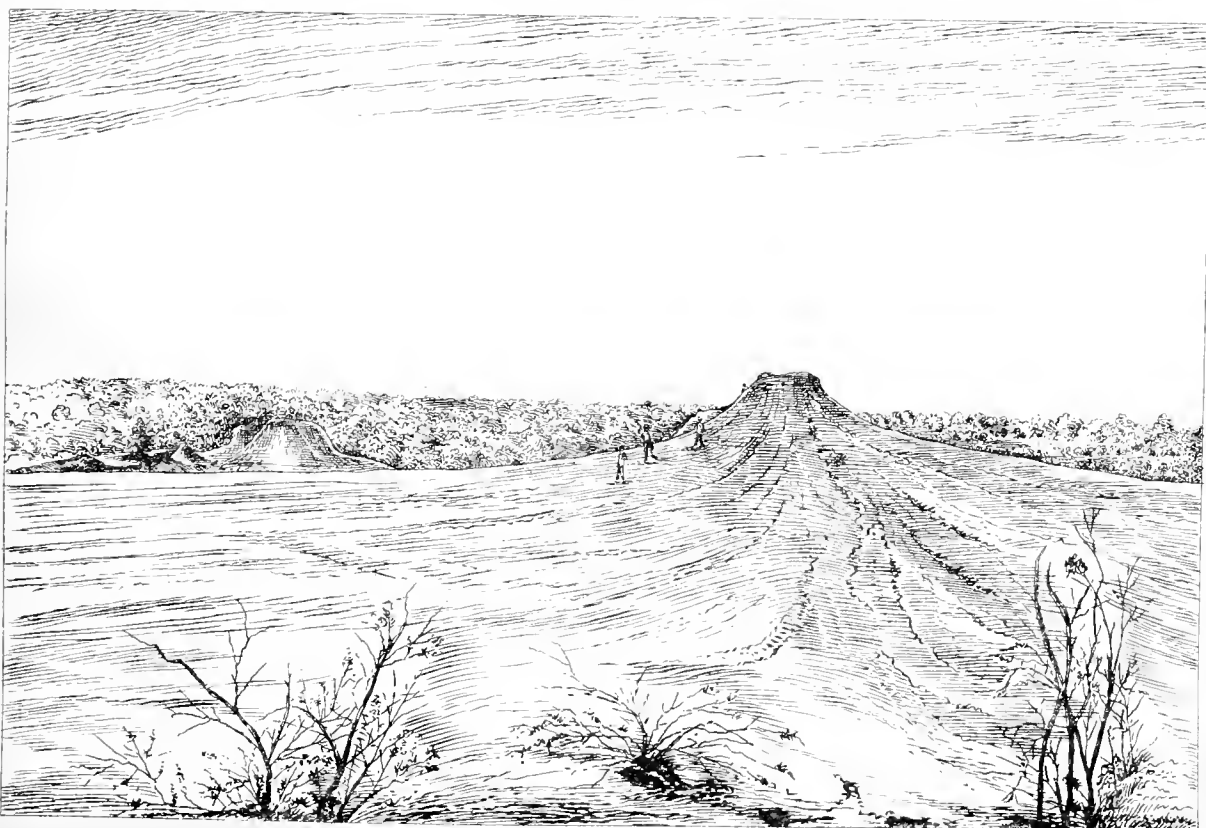


View of Yenangyoung on the Irrawaddy, showing Flat and Native Boats, used in transport of Petroleum.

123456789101112131415161718192021222324252627282930313233343536373839404142434445464748495051525354555657585960616263646566676869707172737475767778798081828384858687888990919293949596979899100



Nº 1. Oil Spring at Minbu.



Nº 2. Mud Volcano at Minbu, half a mile from Nº 1.



interest which is now taken in the subject is very largely due to the information which that graphic writer has made public.

## 2. ASSAM.

As long ago as 1825, Lieut. Wilcox, in an expedition up the Dihing River, observed a seam of coal in the bed of the Buri Dihing at Súpikong, in Upper Assam, near which petroleum rises to the surface.\*

In 1828, Mr. C. A. Bruce observed petroleum springs in more than one locality, and in 1837 Major White discovered several springs of petroleum close to the camp on the Námúp River, which had hitherto been unknown to Europeans, and apparently almost unused by the neighbouring Singphos.†

In 1837—38 Captain S. Hannay noticed petroleum rising from some of the coal outcrops, and in 1845 he described his search in the neighbourhood of Jaipur for petroleum. He also visited the Námchik River, and of this locality he writes:—"At Namtchuk Pathár, near the mouth of the river, the petroleum exudes from the banks, and a bed of very fine eaking coal runs across the bed of the Namtchuk. The hills here are also intersected by ravines, and in one spot an extensive basin or hollow is formed at some height, which contains muddy pools in a constant state of activity, throwing out, with more or less force, white mud mixed with petroleum. This is indeed a strange-looking place, and I am told by the Singphos that at times there is an internal noise as of distant thunder, when it bursts forth suddenly with a loud report, and then for a time subsides."‡

In 1865, a brief account of the petroleum springs in connexion with the coal-fields in Upper Assam was given by Mr. H. B. Medlicott.§ The most abundant springs visited were those near Makum, where Mr. Medlicott recommended that trial borings should be made, the copious discharge of gas and non-discharge of water being regarded as favourable indications.

In November of the following year, Mr. Goodenough, a member of the firm of McKillop, Stewart, and Co., having been granted certain rights over a large tract of land on both sides of Buri Dihing, extending from Jaipur to the effluence of the Noa Dihing, commenced boring at Nahor Pung. In addition to several holes drilled by hand, one of which was 102 ft. in depth, a well was carried down to a depth of 195 ft. by the use of a Mather and Platt's steam-boring machine, but according to the account published by Mr. T. H. Hughes in 1874|| (dated Raiganj, 1st December 1873), no good results were obtained in this locality, though a few signs of gas were noticed. While these borings were in progress, others were begun at Makum.¶

Oil was struck in one hole on the 26th March 1867, at 118 ft., and it immediately rose 74 ft. in the bore, being 44 ft. below the surface. About 300 gallons were drawn, after which it was found not to flow continuously, a circumstance which it was hoped would be remedied by sinking deeper. Eight holes were put down in the Makum district, and they were nearly all successful in tapping oil, though the yield varied in each. In January 1868, 100 to 125 gallons a day were collected from No. 4, while No. 5 yielded from 550 to 650 gallons. The action of No. 5 bore was intermittent. Pure water was spouted for three or four hours, then almost pure oil for 15 to 30 minutes, after which all action ceased for an hour, or sometimes longer, and then activity set in again. The discharge from No. 5 bore was at times so copious that there was not storage accommodation for the oil, and the flow was accordingly diminished by fixing a valve on the well pipe. The pressure exerted by the oil during the flow was 30 lb. to the inch.

The following interesting table gives the times of occurrence and duration of the flows or "blows" from No. 5 bore, with the quantity of oil discharged:—

Date.	Com- menced to run.	Day and Hours running.	Gallons per Day of 24 Hours.	Remarks.
1868.				
Jan. 8	9 a.m.	6 15	530	Stopped gradually.
" 16	11 a.m.	0 12	1,500	1,500 galls. in 12 hours, very strong blow.
" 17	11 a.m.	0 14	2,100	2,100 galls. in 14 hours, very strong blow.
" 20	Midnight	0 9	500	500 galls. in 9 hours.
" 21	10 a.m.	0 20	450	450 galls. in 20 hours.
" 23	11 a.m.	1 13	300	300 galls. per day, ran slowly.
" 27	9.30 a.m.	0 23½	300	do. do.
" 29	9.30 a.m.	6 15	250	Running very slowly.
Feb. 5	9 a.m.	0 19	500	500 galls. in 19 hours.
" 7	9 a.m.	2 0	700	
" 13	2 p.m.	0 19	1,400	1,400 galls. in 19 hours.
" 16	9 a.m.	1 6	1,600	1,600 galls. in 30 hours.
" 17	11 a.m.	1 5	900	In 20 hours,
March 2	9.30 a.m.	1 6	1,700	In 30 hours.
" 7	9 a.m.	2 0	3,000	3,000 galls. in 18 hours.
" 11	9 a.m.	1 11	3,500	In 35 hours; very strong, burst pipes.
" 31	10 a.m.	31 22	500 per day	Valve very little open, to reduce the flow as much as possible.
May 14	6 p.m.	60 0	450 per day	
July 14	11.30 a.m.	3 22½	100	
" 19	9 a.m.	4 21	550	
" 27	1 p.m.	0 15	600	In 15 hours.
" 28	10.30 a.m.	3 1	700	A strong blow.
Aug. 8	9 a.m.	0 3	500	In 3 hours, very strong flow.
" 11	2 p.m.	3 10	1,500	
" 22	9.30 a.m.	0 8	400	In 8 hours.

Here the man who kept the account fell ill, and the one who relieved him never kept any.

Notwithstanding these highly promising results, the great difficulties of transport appear to have brought about the failure of the undertaking; but in recording this Mr. Hughes remarks that "the prospect of an abundance of mineral oil in Assam has been proved, and if this splendid province should ever be opened up, fortunes will yet be made in this branch of mining."

In his description of the coal-fields of the Naga Hills, published in 1876,\* Mr. Mallet enumerated the places where oil has been observed in this district, and the approximate positions of these springs or surface indications are shown in the sketch map of the Assam oil-field (Fig. V.). In all cases the oil rises either on or close to the outcrop of the coal-bearing group, and usually near the outcrop of one or more seams of coal; indeed, Mr. Mallet records one instance in which he saw the oil oozing out of the coal itself, though he points out that this may have been accidental, the coal being merely the last rock through which the oil passed on its way to the surface. Thick soft

\* Asiatic Researches, XVII.

† Jour. Asiatic Soc. Bengal, VI.

‡ Jour. Asiatic Soc. Bengal, XIV.

§ Mem. Geol. Survey of India, I., 44-45.

|| Petroleum in Assam, by Theodore H. Hughes, A.R.S.M., F.G.S., Geol. Survey of India Records, VI.

¶ In lat. 27° 18' N., and long. 95° 40' E.

\* On the coal-fields of the Naga Hills, bordering the Lakhimpur and Sibsagar Districts, Assam, by F. R. Mallet, F.G.S., Geological Survey of India. Mem. Geol. Surv. of India, XI., Part 2.



sandstone is the rock principally met with in boring, but blue clay also occurs. The strata, which are much disturbed, belong to the tertiary epoch.

The most likely sites for productive wells appear to lie within the area of the immense concessions granted to the Assam Railways and Trading Company, and it is well known that this company has for some time past been drilling at Digboi (see Fig. V.).

Mr. J. B. White, a director of the company, has just returned to this country from a visit to Assam, and has very courteously given me full information as to the drilling operations. Digboi is situated six or eight miles to the west of Makum, and drilling was commenced here last autumn, the spot having been selected on account of the good surface indications. At a depth of 176 ft. oil and gas were met with in soft sandstone (subsequently found to be immediately overlying shale), and it was anticipated that the well might yield as much as 80 barrels per day. The production however rapidly diminished, and it became evident that the oil occurred in a small "pocket" which was soon emptied. Drilling was accordingly recommenced, and the well is now in process of being deepened. The indications are regarded by the driller as exceedingly promising, but drilling is here, as elsewhere in India, difficult, and there is considerable tendency to "caving." The oil from this well is, in Mr. White's opinion, less viscous than that obtained at Makum, and contains more paraffin, a large amount of solid hydrocarbons separating from it at 38° F., which was the minimum temperature during his stay. I hope shortly to give the results of an examination of this oil. Mr. White, who was concerned in the work carried out by Mr. Goodenough, informs me that the official accounts from which I have quoted under-stated the yield of the wells. From one bore there was a continuous flow for 18 months after the project was abandoned. He adds that surface indications of oil occur over an area some 60 miles square, and in places the soil is so saturated with petroleum that it is sold for use in protecting timber from the attacks of the white ant.

Mr. R. A. Townsend has recently made an official report\* upon the Assam oil-field which is in the main confirmatory of the foregoing statements. Mr. Townsend's visit of inspection was made in the wet season, and therefore in some respects under conditions unfavourable to accurate observations; but he appears to have satisfied himself that the prospects of success in boring for oil in Assam are extremely good, and he points out that to a large extent the difficulty of transport has been removed.

On more than one occasion within the past 12 years I have examined samples of Assam crude petroleum. The oil, as will be seen from a sample on the table, is dark brown in colour, of rather high viscosity (the viscosity of a sample of sp. gr. .940 was 14.2 at 90° F., rape oil at 60° = 100), and has a slight and not unpleasant odour. Its specific gravity appears usually to range from .933 to .940, and its flashing point is sometimes as high as 212° F. (Abel test). It begins to distil freely at 280° F., but considerably less than 20 per cent. volatilises within the range of the mercurial thermometer. The oil contains none of the kerosene hydrocarbons, but it yields by the ordinary process of distillation 89 per cent. by weight of lubricating oil distillates. The proportion of solid hydrocarbons is not large. The carbonaceous residue varies, according to my experiments, from between 3 and 4 per cent. to over 8 per cent.

In the paper by Mr. Hughes already referred to, particulars are given of the examination of a sample of the Assam crude oil. The material had a specific gravity of .971, and commenced to boil at 460° F.; it was therefore of greater density than the samples I have distilled, but it appears to have yielded more paraffin. As, however, a series of the distillates obtained, ranging in specific gravity from .873 to .918, are characterised as suitable for lamp oil, "although of rather higher specific gravity than that obtained from American petroleum," I do not consider the results worthy of much consideration.

### 3. PUNJAB.

Accounts of the Punjab oil-springs were published by Mr. A. Fleming in 1848\* and in 1853;† by Mr. MacLagan in 1862‡ and by Mr. A. Fenner in 1866.§

A few years later Mr. Lyman was deputed to examine the deposits, and his reports were issued collectively in 1871.¶ From these it appears that in the Rawalpindi district (see Fig. 1.) there are some 16 spots at which indications of petroleum are met with in the tertiary rocks.

Mr. Lyman states that in every case the oil seems to come from a deposit of very small horizontal extent, "sometimes only a few feet, seldom as much as a few hundred yards." Some of the natural springs are described as yielding "as much as three quarts per day," and a boring of 75 ft. deep at Gunda is asserted to have produced about 11 gallons of oil a day for a period of six months. Mr. Lyman gives the density of the oil as 25° B. (=907), but there appears to be some variation in the quality of the oil, for while that obtained at Gunda is said to be burned by the natives "with a simple wick resting on the side of an open dish," the product of the Pannolia springs is described as being more inflammable, and as needing a tube for the wick.

Mr. John D. Noble, of Petrolia, a well-known Canadian petroleum producer and refiner, recently spent some time in this district, and thinks so highly of the prospects of establishing a successful petroleum industry in the Punjab that he has secured a concession of a large extent of territory with a view to drilling for oil.

### 4. BALUCHISTAN.

The oil-field of Khātan is situated in the Mari Hills of Baluchistan, about 40 miles in a direct line to the east of Sibi station on the Quetta branch of the North-Western Railway running from Ruk junction to Quetta. The position of the petroleum wells is shown on the sketch-map of India (Fig. 1.).

The oil occurs in the Eocene formation, and is found exuding in many places, much of the oil which has thus escaped having been converted by exposure into a hard mass.

Borings were first made here on behalf of the Indian Government by Mr. R. A. Townsend, Superintendent of Petroleum Explorations, in the cold season of 1884—85, and it was found that immense quantities of petroleum were obtainable at moderate depths. The wells drilled by Mr. Townsend are five in number, and are situated in a valley surrounded by mountains about 4,000 ft. high. Their diameter is only 4½ in., and their depth does not much exceed 500 ft. The geological features of the locality have been carefully dealt with by Mr. Townsend in an official report,¶ and the section reproduced from that document (see Fig. VI.) clearly indicates the character and disposition of the strata.

The following is an explanation of the section:—

- 1, 2, 3, 4. Nummulitic limestone.
- 5, 7, 9, 11, 13, 15. Light-brown earthy limestone, interstratified with clay and shale and (in 6, 8, 10) gypsum.
- 8 consists of a massive stratum of pure white gypsum.
- 16, 18, 20, 22, 24. Chunchy limestone, interstratified with soft shale.
- 25, 27, 29, 31. Soft olive shale.
- 26, 28, 30. Dark brown flint.
32. Fine-grained hard blue limestone.

"It is in the two deposits of shale 25 and 27 that the first traces of petroleum are discovered, and in several places where vertical faces are exposed to the direct sun's

\* Jour. Asiat. Soc. Bengal, XVII.

† Jour. Asiat. Soc. Bengal, XXII.

‡ Supplement to the Punjab Government Gazette.

§ Proc. Punjab Government Public Works Department.

¶ Reports on the Punjab Oil Lands, by E. S. Lyman, Government Press, Lahore.

¶ Report on the Petroleum Explorations at Khātan, by R. A. Townsend, Superintendent of Petroleum Explorations in Baluchistan. Records of the Geological Survey of India, Vol. XIX, Part 4, 1886.

\* Report on the Assam Petroleum Deposits, by R. A. Townsend, Simla, 14 September 1888.

rays, bituminous drops and threads mark them with a jet blackness."

The following record of one of the borings, of 524 ft. in depth, indicates the order of succession downwards of the strata perforated by the drill:—

	Thickness.	Depth.
	Feet.	Feet.
(1.) Gravel, with boulders and bitumen..	12	12
(2.) Jointed blue limestone .....	20	32
(3.) Hard, marine conglomerate, with abundance of flint .....	195	27
(4.) Alternating bands of soft, bluish shales and hard flinty limestone with iron pyrites.....	30	257
(5.) Rather hard shale with pyrites.....	217	474
(6.) Dark grey limestone without fossils..	2	476
(7.) Soft grey shales.....	48	524

Oil was obtained at 28, at 62, at 92, at 115, at 125, at 133, and at 374 ft. Mr. Townsend states that, "both in drilling and pumping, a considerable quantity of sulphuretted hydrogen gas escapes, but it is not in sufficient quantities to cause a natural flow of oil from the tubes."

Owing to the disturbed character of the strata, drilling is extremely difficult; the conglomerate is fractured in all directions, and through the fissures "the oil finds its way upwards, borne on the top of the warm waters which accompany it; but while these fractures afford a ready means to the miner of 'striking oil,' they sadly interfere with his progress in boring, as the drilling tool in descending must inevitably enter many of these crevices at an acute angle to their planes, and it is almost impossible to prevent the tendency of the tool to follow the vagaries of such crevices, and thus produce a 'crooked hole,' which is fatal to further progress, unless straightened."

Unfortunately the oil obtained is of remarkably high specific gravity and viscosity, as will be seen from the sample on the table. Its density is, I find, practically identical with that of water, and it is in consequence freed with very great difficulty from the water with which it is associated as it comes from the well. Even when the oil is warmed the water does not readily subside. If an attempt be made to distil the oil containing water, the contents of the still froths up and passes over bodily. By prolonged exposure in a capacious vessel to a temperature somewhat above the boiling-point of water, the oil can be sufficiently dehydrated, but a far better system has been suggested, which I hope before long to have the opportunity of describing. The oil is black or extremely dark brown in colour by transmitted light, with comparatively little fluorescence, and it possesses very little odour. Its flashing point is 280° F. (Abel test), and it contains no hydrocarbons available for use as ordinary burning oil. If 50 per cent. be distilled off, the first third of the product will be found to have a specific gravity of about .910, and the remaining two-thirds of about .930, but the viscosity of these distillates is extraordinarily low in relation to the specific gravity. By careful distillation with superheated steam, about one-fourth of the product (one-eighth of the crude oil) may, however, be obtained of sp. gr. .958 and viscosity 168 at 70° F. (rape oil at 60° F. = 100). This oil would be available for use as a lubricant. The residue in the still, when 50 per cent. of the volatile matters have been removed, forms on cooling a moderately hard pitch of jet black colour and lustrous fracture, as the specimens before you indicate. This material might be employed in the manufacture of "patent fuel" and for other purposes. When distilled to dryness the crude petroleum yields rather more than 70 per cent. by volume of a heavy oil containing very little paraffin, and the residue then consists of about 26 per cent. by weight of hard coke. The last portions of the distillate when exposed to a temperature of 32° F. assume the consistence and appearance of vaseline.

Khátan petroleum is by far the most refractory oil to which Professor Dewar and I have applied our new process of distillation, but we have succeeded in obtaining from it about 40 per cent. of kerosene, which has a specific gravity of .810.

It is, however, as a fuel for use in locomotives and otherwise that the petroleum in question is likely to become specially valuable, and from this point of view the importance which attaches to the development of the Khátan oil-field is very great. On a former occasion\* I have referred to the extent to which liquid fuel is employed in Russia, and I anticipate that in those districts of India where solid fuel is very costly, as is the case in Baluchistan, we shall before long see this example followed. The Khátan oil-field lies at an elevation of 1,400 to 1,500 ft. above the railway at Sibi, and it has been proposed to transport the oil to the railway by pipe-line; but as I have already said, the oil is very viscous, and experiments made led to the apprehension that great difficulty would be experienced in pumping it. Mr. Townsend is of opinion that the oil might be pumped so long as its temperature did not fall below 70° to 80° F., but at certain seasons of the year it would be exposed to a temperature far below that. About two years ago I had occasion to investigate the subject, and I found that the viscosity of the oil at 85° F. was no less than 430 times that of ordinary Baku crude petroleum, the viscosity of which is 11.6 at 90° F. (rape oil at 60° F. = 100); I, however, arrived at the conclusion that by the employment of a pipe-line of large diameter, with a sufficient number of pumping stations and powerful pumps, the oil could be transported during at any rate the greater part of the year. The consistence of the oil is not due to the presence of a high percentage of solid hydrocarbons as is the case with much of the Yenangyoung oil, but arises from the large amount of asphaltum present. Hence the fluidity of the oil is not very largely increased by a moderate elevation of temperature.

As an alternative scheme to the pipe-line it has been proposed to connect Khátan with the Sind-Pishin State Railway at Babar-Kuch, and thus provide a means for the transport of the oil, but such a line would be very costly owing to the difficulties of the country through which it would pass.

Mr. Townsend, with whom I had some conversation during his recent visit to this country, evidently appreciates fully the importance of the fuel question, but he regards the comparatively inaccessible position of the field and the trying character of the climate during the hot season (when no European could work there) as serious obstacles to the utilisation of the Khátan petroleum deposits. That petroleum may be obtained there in apparently immense quantities is clearly shown by the yield of the wells. According to the official report of Colonel Conway-Gordon† experiments made by pumping four of the wells (the fifth had not then been drilled) showed that the yield of each well was from 400 to 600 barrels of oil in the 24 hours. "Thus any one of the existing wells is more than competent to deliver the entire supply of 50,000 barrels of oil a year, which is estimated to be the amount required for the Sind-Pishin section of the North-Western Railway."

In this connection I may perhaps be permitted to reproduce the following extract from the "Indian Engineer," which was published last year in the Journal of this Society,‡ under the erroneous heading of "Petroleum works in the Punjab":—"The Khátan petroleum works have, it appears, now passed beyond the experimental stage. An agreement has been definitely entered into to supply the works at the Khojak tunnel with about 3½ million gallons of the oil. The wells, which are 42 miles from the railway, have been connected with Babar-Kuch on the Sind-Pishin line. The saving to the State from the use of the petroleum on the Khojak works alone will, it is estimated, be enough to cover all the cost of the Khátan experiments, and leave a balance of over 5 lakhs of rupees,

\* Russian Petroleum Industry. Jour. Soc. Chem. Ind., Vol. IV.

† Administration Report on the Railways of India for 1887-88, by L. Conway-Gordon, Lieut.-Col., R.E., Director-General of Railways.

‡ 1889, 314 (Trade Report).

And there is no reason to anticipate that the benefits of the apparently inexhaustible supply should end here. On the contrary, in a country like Beluchistan, where every stick of wood is worth its weight in silver, the demand for the new fuel must be very great; and in addition to other advantages its use may be expected to put a stop to the destruction of the forests, which until recently was going on at an alarming rate. It is now intended to replace the expensive and wasteful system of camel carriage by laying a pipe-line from the wells along the Babar-Kuch road; and should Government decide on sanctioning this, it may be confidently expected that the capabilities of Khātan will emerge once for all from the cold shade of neglect and scepticism in which an exaggerated idea of its remoteness has left it so long."

An oil similar to that obtained at Khātan occurs at Shoran, in Kalat, in the province of Kach Gandawa, in Hind Baluch country. Shoran is about 42 miles from the railway at Belpat, and it is a question which of the two fields it would be best to develop for fuel purposes.

#### GENERAL CONCLUSIONS.

Throughout India, petroleum occurs in the tertiary formation, as in Russia and Galicia. The strata in the oil-producing localities are greatly disturbed, and drilling is everywhere in India more or less difficult.

Apparently petroleum occurs in the greatest abundance in the Khātan oil-field in Baluchistan, but the oil is not of satisfactory quality even regarded as liquid fuel, the locality of production is comparatively inaccessible, and the climate is bad.

Undoubtedly the best oil from the point of view of the kerosene refiner is that which is obtained in the Arakan islands (the eastern Baranga and Ranni). On the other hand, the Yenangyoung oil, or much of it, though not yielding kerosene largely by the ordinary process of manufacture, contains much paraffin, which is a valuable product, and, as has been pointed out, the yield of kerosene from such oil is capable of being largely increased.

As regards geographical position, the Arakan deposits are placed at an advantage over those of Upper Burma, and in like manner the Upper Burma deposits, or most of them, can be developed more advantageously than those of Assam, in consequence of the transport facilities afforded by the Irrawaddy river.

Far too little is known about the oil-fields of India to admit of a confident prediction as to their future, in respect either to area or to potential productiveness, being uttered. Even as regards Upper Burma, we cannot say whether the future centre of most successful development will be Yenangyoung, Pagan, Minbu, or elsewhere, and the actual results of drilling can alone remove the uncertainty at present existing. Moreover, it by no means follows that the precise spots selected by the natives as giving readiest access to the oil through the primitive methods of working available to them will prove to be the most productive ones when the drill is substituted for the spade.

The Indian Government appear disposed to encourage competitive private enterprise, especially in the oil-fields of Upper Burma, and so long as this principle is legitimately applied no exception can be taken to it; but in the interests of the Empire it is to be hoped that the work of petroleum exploration and development will in no case be attempted without a full appreciation of all that it involves. It is in the highest degree important that this work should for some time to come be carried out under skilful, energetic, and above all, experienced management, and that ample means should be provided. On any other basis it is highly probable that much money would be wasted without obtaining satisfactory results, or even conclusive negative evidence; much disappointment and discouragement would ensue, and the development of the oil-fields of India would be considerably retarded.

Hitherto the practice of the Government has been to grant a prospecting licence for a period of 12 months over four square miles of territory. On the expiration of the specified period the licensee is at liberty to select one square mile, over which he has the right to drill for oil, on the basis of

the payment of a royalty of 20 per cent. on the oil raised. It is now, however, reported from Rangoon that the Government has decided to grant no more prospecting licences or concessions, at any rate in the Yenangyoung district, and that as soon as Dr. Noetting has completed the marking out of the oil-bearing lands in this locality into square mile blocks, these will be offered for sale by auction. The necessity for purchase should afford some guarantee for the work of development being efficiently carried out.

I am indebted to the India Office, and especially to Mr. Arthur N. Wollaston, C.I.E., for affording me exceptional facilities in referring to various reports dealing with the subject of this paper.

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The following have been selected to fill the vacancies, and will take office in July next:—*Chairman: A. Norman Tate; Vice-Chairman: F. Hurter; Secretary: Chas. A. Kohn; Treasurer: W. P. Thompson; Committee: C. L. Higgins, A. H. Knight, E. K. Muspratt, and A. Watt.*

#### SESSION 1889-90.

May 7th:—*Dr. F. Hurter. "Photo-chemical Investigations."*

Notices of Papers and Communications for the Meetings to be sent to the Local Secretary.

*Meeting held on Wednesday, 2nd April, 1890.*

DR. F. HURTER IN THE CHAIR.

### AN INSTRUMENT FOR THE MEASUREMENT OF DIFFUSE DAYLIGHT AND THE ACTINOGRAPH.

BY F. HURTER, PH.D.

THE actinometer which I have devised for measuring diffuse daylight depends upon entirely different principles than the instrument Mr. Ballard has just brought to our notice. In most instruments designed for measuring light the eye has to decide when two things are equal, in Mr. Ballard's instrument, when the luminosity of the paint is equal to the light coming through the blue glass, in the ordinary silver paper actinometers the eye decides when two tints are equal.

Once when examining the spectrum of nitric dioxide the thought struck me that the light absorbed by that gas must do some molecular work, and as this was not chemical

decomposition, it could only result in a rise of temperature. I saw the application which could be made of this, and I found that if a differential gas thermometer was filled with nitrogen dioxide, one bulb exposed to diffuse light, the other kept in the dark, the gas exposed to the light expanded. But owing to the difficulty of finding a suitable separating fluid, one which should not absorb the gas, I could not obtain satisfactory results. Equally unsatisfactory I found an instrument filled with bromine vapours. The idea that all coloured substances would in diffuse light assume a slightly higher temperature than white or colourless transparent substances led to the construction of differential thermometers, the bulbs of which contained a red substance in one, and a white one in the other. Very sensitive instruments were obtained with red and white wool, next in sensitiveness was cotton and paper. The red substances absorb most of the rays of the spectrum, except the red ones, whereas the white substances absorb very little of any of the rays, and the absorption of the light causes a slight rise in the temperature of the red substance, which expands the air of the bulb containing this substance. The consequent increase in pressure is read off on the scale behind the syphon gauge, and as experiments in this direction proved, this rise of pressure was proportional to the chemical action of the light on silver salts.

But instruments so made were very unsatisfactory. Beside the temporary alterations in pressure in the bulb containing the red substance there was also a permanent change, which increased with every exposure to the light, and though little perceptible in one day, in the course of a few months became so large as to completely destroy the

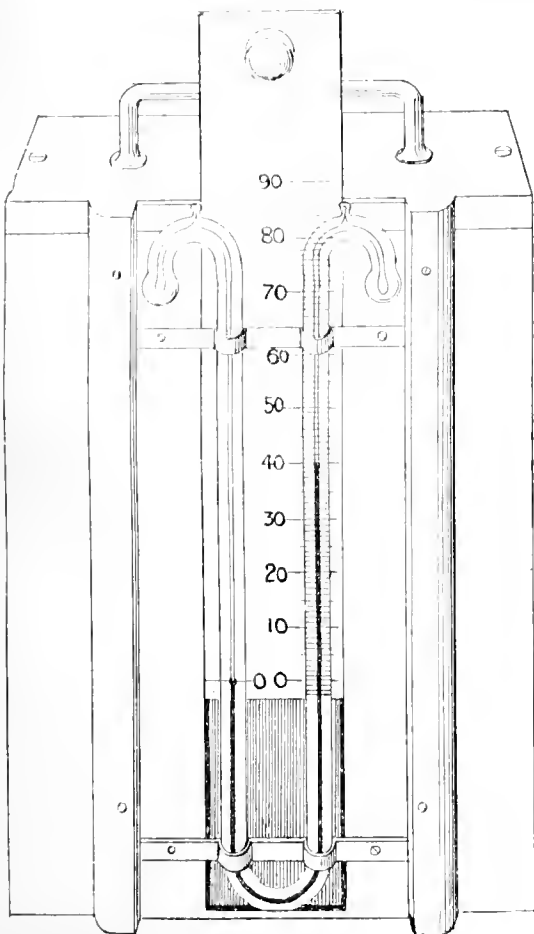
instrument by driving the liquid out of the syphon gauge into the bulb containing the white substance.

An investigation of what this was due to revealed the fact that the oxygen of the enclosed air gradually vanished, and it disappeared faster in the white tube than in the red one. What becomes of the oxygen I am unable to say. It was clear I had to abandon organic substances.

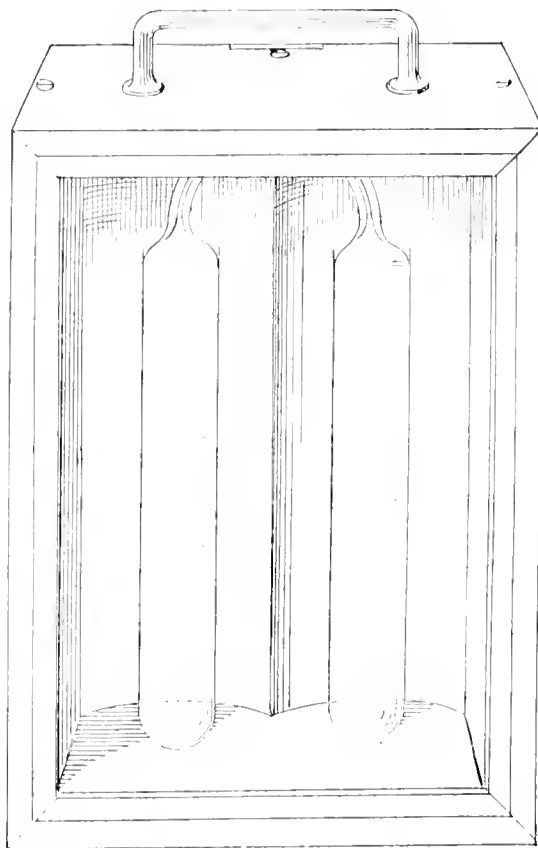
But even when I began to use coloured glass the same difficulty still arose. I used either alcoholic solutions or benzine solutions of alkanet as indicator in the syphon gauge, and even after I had abandoned them also I still found the zero points of the instrument unsteady. It varied very little, but in the course of a year the change was quite perceptible.

A good instrument can however be made as follows:—Two test tubes holding from 10 to 15 cc. are chosen; one of them must be made of very thin glass flashed inside with copper ruby glass, the other one must be of similar thin clear white glass. They should both be of the same weight and the same capacity, a slightly larger capacity for the white tube is favourable. These tubes must be well cleaned with nitric acid and caustic potash, carefully rinsed with boiled distilled water, dried and fused on to capillary glass tubes. When this has been done they must be repeatedly exhausted under the air pump to remove all products of combustion which enter during the operation of fusing to the capillary. They are finally made red hot and when cool a single drop of pure distilled water is allowed to enter into each tube, and is shaken into the bulb. The syphon gauge is filled either with a solution of potassium bichromate, or better with a solution of potassium carbonate, to which is

#### HURTER'S ACTINOMETER.



Back View.



Front View.

added sufficient potassium iodide and iodine to give it a deep colour. The bulbs are then sealed on to the syphon gauge, two very fine hair tubes being drawn out first, through which the air can escape and enter, on either side of the liquid in the syphon gauge.

When the instrument is so far completed it is again exhausted repeatedly to remove products of combustion, and the hair tubes are sealed up, the bulbs being at the time immersed in cold water to keep their temperature alike. The instrument has then to be tested to see that it is absolutely air-tight, and that both bulbs behave alike when the temperature is raised.

The position of the liquid in the gauge must not move under the receiver of the air pump, nor when the bulbs are immersed in hot water. A temporary change during the latter operation always occurs, but it must be only temporary.

If the instrument be found good it is mounted in a wooden box with a glass front, and to render it more sensitive the bulbs are placed in front of two concave reflectors, which are best made of silvered sheet copper and very thin. The side of the box opposite the reflectors is closed by a sheet of thick patent plate glass, coated on the outside with transparent gelatin and varnish, which helps to prevent the entrance of heat rays into the box.

A millimetre scale behind the syphon gauge, which is outside the box at the back, completes the instrument. (See illustrations.)

Yellowish-brown glass is almost as good as ruby glass and easier to procure.

An instrument of this description will indicate the brightest diffuse daylight obtainable in this country, as reflected by the northern sky by a variation in the level of the liquid of about 80 millimetres. It is therefore possible to measure the diffuse light to one per cent. of its greatest intensity with ease, and this is more than sufficiently accurate for all photographic processes.

By means of this instrument it is quite possible in steady light to time very accurately the exposure of a photographic plate. But my friend Mr. Driffield soon discovered that a very accurate measurement of the light was not necessary, and the reason for this we decided to ascertain, and he has been my fellow worker in all subsequent investigations.

We made self-recording actinometers by registering the position of the liquid in the syphon gauge photographically

upon gelatino-bromide paper, which was fastened on a revolving drum driven by a clock. We continued to take observations for every day of the year 1885—86, and some of these photographs of the intensity of diffuse daylight are represented by the diagrams.

When there was a steady light throughout the day, which seldom occurred, the curve so clearly indicated that the light was a function of the altitude of the sun, that I had no difficulty in recognising the sinus curve. This curve showed itself really in almost every diagram. It is broken up by passing clouds. The dotted curve in the diagram of March 4th shows the sinus of the altitude of the sun on that day for latitude  $53^{\circ} 8'$ . By measuring at the same time the diffuse light with the actinometer, and the sinus of the altitude of the sun by well-known means, we obtained the connexion between the altitude of the sun and the degrees of our actinometer.

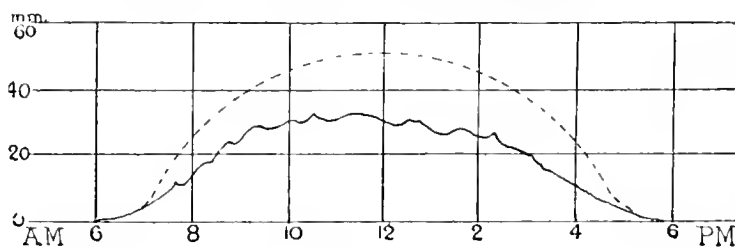
We thus found that the diffuse light at any given hour of any given day is seldom less than 25 per cent. of the maximum light possible at that hour, except when it is foggy or raining, but under those circumstances no one thinks of taking pictures outside.

It is therefore clear that exposures of photographic plates at any particular moment under ordinary conditions vary only in the ratio 1:4. But as other investigations which Mr. Driffield and myself made showed that the latitude of exposure within which a good picture is possible is in many cases comprised in exposures varying from 1:2, it will be seen that accurate measure of the light is quite unnecessary.

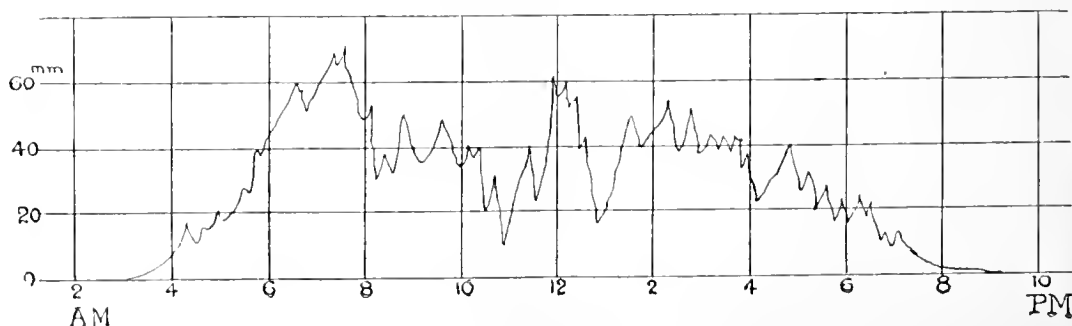
We therefore together devised the little instrument called the actinograph, which Mr. Ballard recommended as a calculating machine. The fact is, it is not only a calculating machine, it is in reality a machine for telling at any hour of the year the maximum light possible, and for converting that indication directly into times of exposures of plates of varying speed, with various lenses.

The manipulation of the instrument is exceedingly simple, and it has this great advantage over actinometers, it tells beforehand what the exposure on any day at any hour has to be, according to the condition of the atmosphere, and the eye soon judges that. With this instrument photographic exposures can be ascertained with so great a degree of probability as to be almost equal to certainty.

DIFFUSE LIGHT REGISTERED BY HUYTER'S ACTINOMETER.



March 4th, 1886.

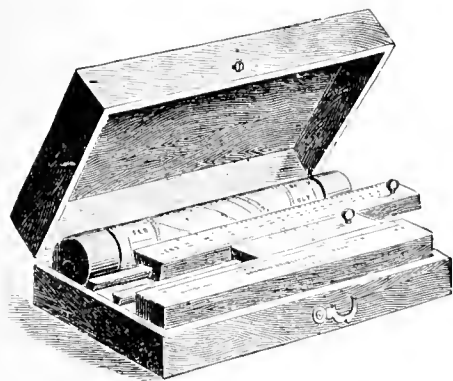


June 12th, 1886.

The instrument has four scales: a light scale on a revolving cylinder, a slide carries the lens scale and the time scale, and on a fixed scale is marked the speed of the plate. The speed of the plate must be ascertained by the operator by a simple experiment.

The lens scale is set to the hour and date at which an exposure is to be made, the speed index is set to indicate the speed of the plate; when that is done five different exposures in seconds can be read off at once, corresponding to five different conditions of the light: Very bright, bright, mean, dull, and very dull.

The sketch shows the instrument.



THE ACTINOGRAPH.

To give an idea of speed, I will add that Wratten's slow plates are about 10 on our scale, and Ilford Ordinary about 15 to 20.

The degree of light and the unit of speed are chosen for the instrument as follows:—The highest possible diffuse light corresponding to the altitude of the sun =  $90^\circ$ , is divided into 100 equal parts, one of which we call an actinograph degree. The unit of speed is the speed of a plate which with one such degree of light will take an ordinary landscape in as many seconds as the square of the ratio of the focal length and aperture indicates. Thus a plate would be speed 1, which with one actinograph degree of light, and a lens of  $F \div 4$  it would take 16 seconds to produce a good landscape negative.

This system of degrees of light and speed of plates I venture to say is the only scientific one which has yet been proposed, and the actinometer I have described I believe to be the only satisfactory instrument for measuring diffuse light yet devised. The photographed records show that it is somewhat slow in following the light. Indeed the variations of light are frequently so sudden that the use of actinometers for photographic purposes will always remain unsatisfactory, because some time must necessarily elapse between the observation of the light and the exposure owing to a calculation having to be made.

The actinograph, on the other hand, avoids all calculation, takes into account variations in light, in lenses, and in plates, and will be a truly scientific substitute for actinometers, so long as the earth does not deviate from her time-honoured journey round the sun.

## NOTE ON A CRYSTALLINE BOILER DEPOSIT.

BY A. F. FRYER,

*Sheridan-Muspratt Scholar, University College, Liverpool.*

A few weeks ago, on the removal of a boiler from a steam tug, which had been working in the River Mersey some 12 years, an interesting deposit was found, especially incrusting the iron stays of the boiler. A sample of this was kindly sent to our laboratories by Mr. T. L. Miller.

It was thought that the sample would be worthy of your interest, on account of the thickness and the convoluted appearance of its upper surface, but especially because the greater part of it is distinctly crystalline.

The substance consists almost entirely of the sulphates of calcium and magnesium, as will be shown by the percentages of my analysis:—

CaO .....	33.3
MgO .....	7.3
Al <sub>2</sub> O <sub>3</sub> .....	1.4
SO <sub>3</sub> .....	51.7
SiO <sub>2</sub> .....	0.6
Moisture .....	2.6
	<hr/> 96.9

There is also a trace of iron and the faintest trace of chloride.

The presence of the water is, I think, interesting. The sulphate of magnesia is undoubtedly present as kieserite, which crystallises with one molecule of water. The percentage of water necessary for my 7.3 per cent. of magnesia would be 3.3 per cent., which you see is a little above the actual amount found.

From an examination of the crystals, they appear to consist of the rhombic anhydrite  $\text{CaSO}_4$ , along with a little kieserite.

The specific gravity I also determined and found to be 3.05 (that of anhydrite is 2.8—3.0).

Hoppe-Seyler and G. Rose have shown that gypsum when strongly heated in a solution of sodium chloride is converted into anhydrite. The conditions under which this deposit has accumulated point to the probability that its crystalline structure may have arisen from this cause. It is curious also that at Stassfurt anhydrite crystals occur in the kieserite, crystallising in rhombic prisms with pinnakoid domes. 8.6 per cent. of the substance is soluble in water, and the calcium sulphate is more soluble than the magnesium sulphate, the proportions being 88.71 per cent. to 11.29 per cent. respectively.

No alumina was found in the soluble portion.

## DISCUSSION.

Dr. Kohn said it would be interesting to hear if any of the members had come across a very marked crystalline boiler incrustation, similar to the one shown.

Mr. W. P. THOMPSON said it was very rarely that they did not find the deposit crystallised when it was largely sulphate of lime. He had repeatedly had to clean out boilers in his younger days, and he had repeatedly seen the stuff crystallised when it had plenty of sulphate of lime. He had seen the crystals with  $\frac{1}{4}$  in. long needles when they were at the end of a nut or stay. They were found in locomotive boilers in the form of radiating nodules.

Dr. HERTER said that as a rule boiler incrustation was always of a crystalline texture, but he had never seen such beautiful specimens as those shown.

## Newcastle Section.

*Chairman: T. W. Stuart.*

*Vice-Chairman: P. P. Bedson.*

### Committee:

A. Allhusen.  
G. T. France.  
G. Gatheral.  
*John Glover.*  
T. W. Lovibond.  
*John Pattinson.*

H. R. Proctor.  
B. S. Proctor.  
W. W. Proctor.  
W. L. Remondson.  
*C. H. Ridsdale.*  
J. E. Stead.

*Hon. Local Secretary and Treasurer:*  
Dr. J. T. Dunn, The School, Gateshead.

The names in *Italics* are those of members of Committee who retire at the close of the current Session. The following have been elected to fill the vacancies and will take office in July next:—*Chairman: John Pattinson; Vice-Chairman: T. W. Stuart; Committee: P. P. Bedson, C. H. Hills, and T. W. Hogg.*

Notices of Papers and Communications for the Meetings to be sent to the Local Secretary.

## Manchester Section.

The Committee of this Section is unchanged.

## Nottingham Section.

The Committee of this Section is unchanged.

## Glasgow and Scottish Section.

*Chairman: R. R. Tatlock.*

*Vice-Chairman: Wm. Pittman.*

### Committee:

G. Beilby  
Wm. Boyd.  
*A. Crum Brown.*  
J. Christie.  
*W. S. Curphey.*  
D. B. Dott.  
Wm. Foulis.  
J. Gibson.

*R. Irvine.*  
G. McRoberts.  
E. J. Mills.  
*J. M. Milne.*  
T. L. Patterson.  
E. Smith.  
*E. C. C. Stanford.*  
*A. Whitclaw.*

*Hon. Treasurer: W. J. Chrystal.*

*Hon. Local Secretary:*

Dr. G. G. Henderson, Chemical Laboratory, University of Glasgow.

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### SESSION 1890.

May 6th (Edinburgh):—

Dr. J. B. Readman. "The Manufacture of Phosphorus, Part II."

Mr. G. McRoberts. "Blasting Gelatin and other Explosives."

June 3rd (Glasgow):—

Dr. J. Clark and Mr. McGhie. "A new Cobalt Mineral."

Notices of Papers and Communications for the Meetings to be sent to the Local Secretary.

*Meeting held in the Societies' Rooms, 207, Bath Street, Glasgow, on Tuesday, 1st April 1890.*

MR. R. R. TATLOCK IN THE CHAIR.

## NOTE ON THE FATTY ACIDS OF OLIVE AND OTHER OILS.

BY ROBERT R. TATLOCK, F.R.S.E., F.I.C., F.C.S.

IN estimating the fatty acids in olive-oil soap I have always and very frequently noticed that after the acids were dried till every trace of water had been expelled they continued to lose weight by exposure to the temperature of the water-oven, which is usually about 90° C. (194° F.). That this loss resulted from the volatilisation of some substance, probably of an acid character, which had been in combination with soda, there could be little doubt, and subsequent experiments confirmed this view. In any case the substance dissipated was not water, and as its amount was considerable the question arose as to whether it should be regarded as a portion of the fatty acids, or merely as an inoperative substance incapable, even in combination with soda, of contributing to the detergent properties of the soap, and consequently of little or no value. At first it was thought probable that this volatile body existed only in the oil which had been extracted from the olives by chemical means or solvents (disulphide of carbon), and which is commonly employed for the manufacture of this class of soap, and that it would not be present in the oil expressed by mechanical means. It was soon found by experiment, however, that it existed in these two varieties to an equal extent as nearly as possible. On extending the investigation to other oils I soon found that while the fatty acids of some of these behaved in a similar manner to those of olive oil—giving off a volatile body at or under steam heat—there were other oils whose acids gave an opposite result, as they gained weight, undoubtedly due to oxidation, under similar circumstances, as did olive oil itself, and more particularly when it was deprived of the considerable proportion of free oleic acid almost invariably present in the commercial article. Of course it is quite possible—indeed highly probable—in the case of fatty acids of olive oil and those which behave under heat in a similar manner, that oxidation and consequent increase of weight goes on simultaneously with volatilisation and consequent decrease of weight, but that the latter goes on at a greater rate than the former, thus making it alone apparent. On the other hand it is hardly possible that the fatty acids such as stearic, that show but a slight decrease in weight after many hours' exposure, can contain any appreciable proportion of volatile substance, as if they did it would pass off at any early stage of the heating process, at least to a large extent, and would thus be observed.

The subjoined table shows the weights of 100 parts of fatty acids from various kinds of commercial olive oil, as well as those from castor, rape, cotton-seed, linseed, and stearine, and also of commercial and neutral olive oil, when exposed for various periods to a temperature of 90° C. (194° F.).

The fatty acids were in each case prepared by saponification with alcoholic potash and liberation by normal hydrochloric acid in slight excess. They were then dried until they ceased rapidly to lose weight, and no film of water could be condensed on glass by the cooling of their vapours.

It is unnecessary to interpret the results, as the figures speak so plainly for themselves. It should be noted, however, that the olive oil No. 10 was the one employed to produce the fatty acid No. 4 as well as the neutral oil No. 11, which was obtained by shaking up the oil with weak solution of caustic potash to saponify the free fatty acid, dissolving out the neutral oil by ether and recovering by evaporation.

Of course the volatile substance may be a decomposition-product of the fatty acid from which it is evolved by heat,



and judging from the tardiness with which its evolution begins and progresses, it is more likely to be such than a volatile acid pre-existing in the oil. On account of this consideration I have hitherto in analysis of soaps and oils returned it as a portion of the true fatty acid.

It is obvious from these results that the determination of fatty acids in soaps is, in many instances, a delicate operation, and one requiring as much care, skill, and observation as any other part of the analysis of that material.

TABLE SHOWING LOSS (OR GAIN) IN WEIGHT OF DRY FATTY ACIDS, DURING DIFFERENT PERIODS, AT 90° C.

Time heated at 90° C. (194° F.).	1. Fatty Acids from Olive Oil.	2. Fatty Acids from Olive Oil.	3. Fatty Acids from Olive Oil.	4. Fatty Acids from Olive Oil.	5. Fatty Acids from Castor Oil.	6. Fatty Acids from Rape Oil.	7. Fatty Acids from Cotton- seed Oil.	8. Fatty Acids from Linseed Oil.	9. Stearic Acid.	10. Olive Oil containing 9.42 per Cent. Free Oleic Acid.	11. Olive Oil from which Free Fatty Acid was removed.
Dry .....	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00
24 hours ...	99.22	99.33	99.18	99.50	99.18	100.50	99.26	101.25	100.08	100.24	100.58
48 ..	98.88	98.92	98.85	99.06	98.51	100.30	99.04	101.23	100.06	100.52	101.42
72 ..	..	..	..	98.70	97.85	99.89	..	..	99.72	100.52	101.32
96 ..	98.18	98.20	98.17	..	..	..	98.42	100.42	..	..	..
120 ..	..	..	..	98.09	96.82	99.46	97.87	100.19	98.92	100.40	100.36
192 ..	96.96	97.08	96.97	..	..	..	..	..	..	..	..
360 ..	95.45	95.50	95.42	..	..	..	..	..	..	..	..
528 ..	94.14	94.17	94.10	..	..	..	..	..	..	..	..
720 ..	92.62	92.67	92.57	..	..	..	..	..	..	..	..

#### DISCUSSION.

MR. STANFORD asked what acid was used in precipitation? His reason for asking that was because he had found it extremely difficult to wash some organic substances free from acid, and he thought it was possible some acid might have been left and become a little concentrated. He was also inclined to think there might be a small portion of glycerin left. With regard to the loss of about 8 per cent. that occurred, it seemed to him that the volatile substance might be recovered, and he hoped Mr. Tatlock would try and devise some method for doing so.

DR. MILNE asked Mr. Tatlock if he had not employed sulphuric acid as well as hydrochloric in precipitation?

MR. TATLOCK, in reply, said that the first three or four results were got by the use of sulphuric acid, but in the later experiments hydrochloric acid was considered to be the best. With reference to Mr. Stanford's suggestion about some of the acid being left, he should have said in his paper that the hydrochloric acid was added in very slight excess, so that the volatile substance got amounted to very many times the entire excess of acid employed. As regarded glycerin being left, considering the complete saponification with alcoholic potash, he was of opinion the glycerin must all have been washed away.

to manipulate, which is a serious matter in a works laboratory where many analyses have daily to be made.

It must be admitted that outside of those directly connected with the subject, very few even competent chemists will be found from whom reliable results can be obtained. There is evidence of this in a case which came before my notice some time ago, in which a sample of cobalt material agreed upon by the buyer and seller at 6 per cent. was reported by a competent French chemist to contain 15 per cent. This is certainly an exaggerated instance, but there is doubtless necessity for more light being thrown on this subject, and I think I am justified in putting on record a complete process for the separation and estimation of cobalt and nickel, which, although not quite new, may still be unknown to the majority of the members of this Society.

The process consists of two operations:—

1. Separation and determination of the cobalt by the "phosphate" method.

2. Determination of the nickel by electrolysis.

The "phosphate" method was originally devised by M. Dirvell, and published in 1879, but, as then described, proved very unworkable, and it was not until modified on several important points by Dr. Clark in 1883 that its value as a means of separation was really appreciated. It has now been in daily use for several years, and has given complete satisfaction both as to accuracy and the facility with which the separation can be made.

Before proceeding to its description it may be well to describe briefly the procedure usually adopted leading up to the separation which is applicable to ores and mattes containing metals removable by  $H_2S$  in acid solution, and any iron, alumina, or manganese which may be present. A quantity of the sample is taken according to its approximate contents, but that will be found most suitable which gives about .5 gm. or thereabouts of mixed metals. This is dissolved in  $HCl$  or aqua regia and evaporated to dryness, the residue moistened with  $HCl$ , dissolved in hot water, filtered from silica, &c., and in the filtrate metals of the second group are removed with  $H_2S$ . After oxidation with  $HNO_3$  the filtrate is nearly neutralised with  $Na_2CO_3$ , and the iron and alumina twice precipitated with acetate of soda to ensure perfect separation. The mixed filtrates, thoroughly freed from iron and alumina, are slightly acidified with acetic acid, and the cobalt and nickel thrown down with  $H_2S$  gas, filtered, washed with  $H_2S$  water, dried, calcined, and weighed as a guide to the approximate quantity of the two metals present.

#### THE ESTIMATION OF COBALT AND NICKEL.

BY JAMES HOPE.

THE separation of cobalt from nickel has always been a matter of difficulty to the chemist, especially due perhaps to the extremely analogous properties of these two metals.

Many processes, however, have been devised for this purpose, but the majority have been abandoned as being in various respects faulty. The "nitrite" process, which is that most commonly recommended by text-books, is no doubt found by some to give fairly good results, but in the hands of inexperienced operators very often fails to give any results at all when cobalt is certainly known to be present. The chief objection, however, is that it is long and tedious

The cobalt and nickel being thus obtained as sulphides and free from all other metals, are dissolved in aqua regia in a small covered beaker, any sulphur which may separate being removed by heating with a few drops of bromine, and the solution evaporated to dryness with the addition of a few drops of dilute  $\text{H}_2\text{SO}_4$  to convert the metals into sulphates. The presence of chlorides would interfere with the subsequent electrolysis of the nickel. The residue after dissolving in hot water and diluting to about 50 cc. is then ready for the separation. A quantity of phosphate of ammonia ( $\text{NH}_4\text{H}_2\text{PO}_4$  containing about 61 per cent.  $\text{P}_2\text{O}_5$ ) equal to about four times the weight of metallic cobalt and nickel present, is now weighed off, dissolved in the smallest possible quantity of water and boiled for a minute or two with a few cc. dilute  $\text{H}_2\text{SO}_4$ . This is afterwards added to the solution containing the cobalt and nickel which has been previously heated nearly to boiling.

Dilute ammonia (2 parts '880 to 1 of water) is now cautiously added until the light blue precipitate which at first forms partially dissolves, and finally with constant stirring and addition of the ammonia drop by drop the cobalt comes down as a pink crystalline precipitate of ammonio-cobaltic-phosphate  $\text{CoNH}_4\text{PO}_4$ . The beaker is placed on the top of the water-bath for 5 or 10 minutes with occasional stirring and finally allowed to settle. The clear blue filtrate containing the nickel is decanted off through a small filter, and the precipitate in the beaker dissolved in a few drops dilute  $\text{H}_2\text{SO}_4$ ; a small excess of phosphate of ammonia again added and the cobalt reprecipitated with ammonia exactly as already described. The precipitate is now transferred to the filter, washed well with small quantities of hot water, and dried at  $100^\circ\text{C}$ . After removal of the precipitate the filter is burned in a platinum capsule, the precipitate added and ignited for 15 or 20 minutes over a good Bunsen burner. The ammonio-cobaltic-phosphate is thereby converted into the purple coloured pyrophosphate  $\text{Co}_2\text{P}_2\text{O}_7$ , and as such is weighed, the metallic cobalt being found by multiplying the weight obtained by the factor (0.403).

In many cases the reprecipitation of the cobalt might be dispensed with, but it is absolutely necessary where the nickel is largely in excess of the cobalt. As it only occupies a few minutes it is always safer in every case to make the second precipitation to ensure perfect separation.

The proper quantity of ammonia to be added is also a point which requires careful attention, as an insufficiency would still leave nickel with the cobalt, and too large an excess would likely dissolve a little of the cobalt. Before proceeding therefore to the estimation of the nickel, it is customary to examine the mixed filtrates by gently heating for a few minutes on a sand-bath, and if a slight pink precipitate forms it is immediately filtered off and added to the main precipitate. Cobalt, however, is rarely found with the nickel as, with careful observation, the exact point at which the addition of the ammonia should be stopped is easily discerned. Prolonged heating of the nickel filtrate when free from cobalt might precipitate a small quantity of the nickel as the yellow-coloured phosphate. This may be easily removed by acidifying with  $\text{H}_2\text{SO}_4$  and making again alkaline with ammonia.

The clear blue filtrate now ready for the determination of nickel is contained in a tall form of beaker, and should bulk about 200 cc. About 10 cc. strong ammonia are added, to render distinctly alkaline, and the solution, after heating to about  $70^\circ\text{C}$ ., is immediately ready to be electrolysed. For this purpose a battery of two 1½-pint size Bunsen cells is found to give the requisite strength of current, and is capable of depositing about 0.15 to 0.20 grm. of nickel per hour.

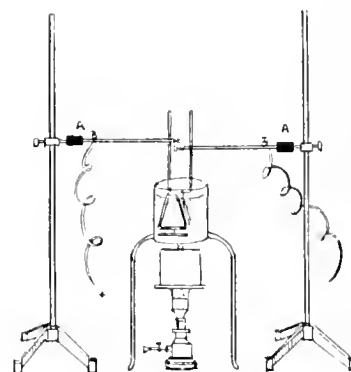
The electrodes employed consist of a sheet of platinum shaped in the form of a cone, to the side of which a piece of thick platinum wire is riveted, and a spiral formed of the same kind of wire terminating in a straight piece, both being of the dimensions usually supplied by Johnson, Matthey, and Co., of London.

The spiral being now inserted in the solution, the cone is also suspended to cover, without touching, the spiral, taking care to have it wholly immersed in the solution. The battery can now be connected, the cone with the zinc or negative

pole and the spiral with the carbon or positive pole, and the beaker covered with two halves of a watch glass. If the battery is in good working order, the nickel will be found firmly deposited on the cone as a bright metallic coating in from two to three hours. The complete deposition of the nickel may easily be ascertained by drawing off a few cc.'s of the solution with a pipette, which should not give the slightest colouration on addition of a drop of colourless ammonium sulphide. As soon as this is the case, the battery is disconnected, the cone removed from the solution, thoroughly washed in distilled water, then in rectified alcohol, dried in a water-bath at  $100^\circ\text{C}$ ., and weighed. The weight of the cone having been previously noted, the increase found gives the weight of metallic nickel.

The whole process of separation and estimation need not occupy more than four hours, and of course several may be carried on side by side. I think it will be recognised that this is a decided advance on the older processes as regards time; and as to its accuracy, I have made many estimations, using solutions of carefully-prepared pure salts of cobalt and nickel mixed in various proportions, of which I give here a few of the results:—

Taken.		Found.		Per Cent. Found.	
Cobalt.	Nickel.	Cobalt.	Nickel.	Cobalt.	Nickel.
1236	1155	1242	1155	100.5	100.0
1236	05775	1232	0575	99.7	99.6
2172	05775	2149	0585	99.1	101.3
3708	05775	3701	0580	99.8	100.4
0618	3465	0619	3454	100.1	99.7
0618	2310	0625	2295	101.1	99.4
0618	1155	0621	1155	100.5	100.0



A A Blocks of Vulcanite.



Cone.



Spiral.

In conclusion, I have here shown a rough sketch of the apparatus employed, which may be more easily understood by those not specially conversant with electrolytic work.

# DISCUSSION.

Mr. STANFORD asked if the solution of the nickel salt was heated during electrolysis?

Mr. ROUGER said it had been his fortune to have a little to do with cobalt and nickel, and he was glad to hear that Mr. Hope had been so successful with his process—a process which he had something to do in bringing forward. He noticed one or two alterations Mr. Hope had made, which he could quite understand would be advantageous. One of these, especially, was the conversion of the salts into sulphates before the operation was commenced. In his time they were in the habit of working upon them as chlorides after the cobalt had been precipitated. By Mr. Hope's plan the process had been shortened a step.

Dr. MILNE said that some time ago Mr. Hope mentioned to him that he was making some experiments upon a volumetric method of determining cobalt, and he would like to know if he had had any reliable results in that way.

The CHAIRMAN said that if familiarity with the subject entitled a person to speak upon it, he was sure Mr. Hope must be considered as an authority upon the determination of cobalt and nickel. He could not of course give, off-hand, any opinion with regard to the details of the methods, but could only judge by the results. These results, considering the minute quantities operated upon, were satisfactory. He was glad to notice particularly that Mr. Hope had kept in view the necessity of testing the accuracy of the process by operating with a small quantity of cobalt and a large amount of nickel, and *vice versa*. This he regarded as an instance of the extreme accuracy which nowadays was obtained in technical laboratories. It was not so many years ago that professional chemists had to instruct technical chemists, but now he was very glad to be able to recognise that in many cases it was the reverse. The professional chemist could not possibly have a perfect knowledge of all processes used in manufacture, for the field was far too vast to admit of him gaining such a diversified knowledge, and it was to the technical chemist he must look for the extreme accuracy and correct methods for working out special processes. He was glad to say that in any instance where he had applied to works chemists for a description of the special methods employed for the determination of substances in connexion with manufacture, they at once responded to his request.

Mr. HOPE, in reply to Mr. Stanford, said the solution was kept at a temperature of 70° during the electrolysis. With reference to Dr. Milne's remark, when he promised to give a paper before the Society, he intended it to be on a volumetric process for the determination of cobalt, but not having the necessary time to work out the process, he thought it might be interesting to give the paper he had read to-night. He intended, however, to give the other paper on some future occasion.

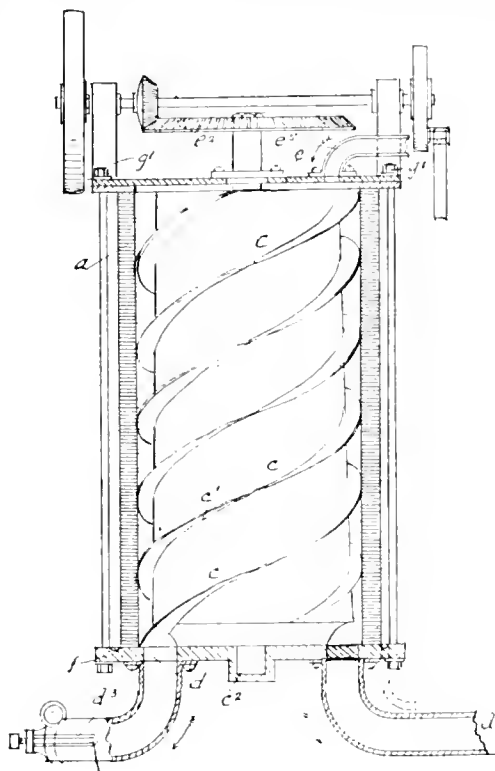
## Journal and Patent\* Literature.

### I.—GENERAL PLANT, APPARATUS, AND MACHINERY.

#### PATENTS.

*Improvements in Means or Apparatus for Separating Solid Matters from Liquid.* F. H. Danchel, Maidstone. Eng. Pat. 499, January 10, 1889. 8d.

The apparatus described in this specification is practically a continuous filter from which the solid matter is continuously removed. A cylinder (*a*) is formed from rings of thin



sheet metal held between the end plates *e* and *f* by means of tie bolts *g*<sup>1</sup>. In the interior is a cylinder *c* having formed upon its periphery the screw thread *c*<sup>1</sup>, and this can be rotated by the shaft *c*<sup>2</sup> by means of the gearing shown. The material to be filtered is forced into the cylinder through the pipe *c*<sup>2</sup> under pressure; the liquid escapes through the small interstices between adjacent plates, leaving the solid matter on the internal surface, from which it is scraped by the screw *c*<sup>1</sup> and forced from the machine through the pipes *d*, *d*<sup>1</sup>, which are closed by spring-loaded valves shown at *d*<sup>2</sup>. The operation of the whole is therefore continuous.

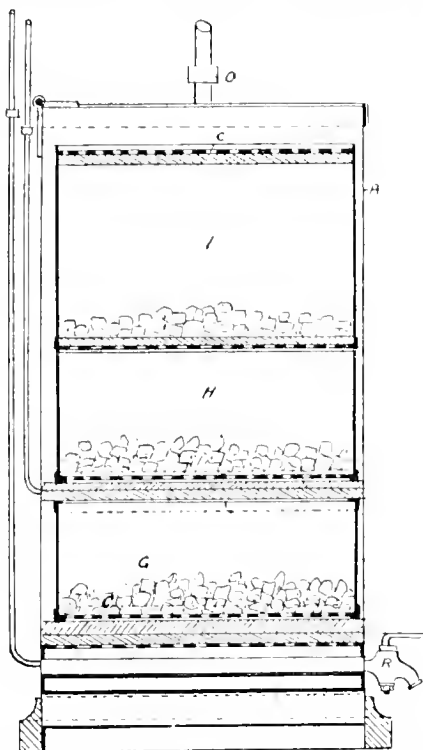
—C. C. H.

\* Any of these specifications may be obtained by post, by remitting the cost price, plus postage, to Mr. H. Reader Luck, Comptroller of the Patent Office, Southampton Buildings, Chancery Lane, London, W.C. The amount of postage may be calculated as follows:—

If the price does not exceed 8d. ....	1d.
Above 8d., and not exceeding 1s. 6d. ....	1d.
" 1s. 6d., " " " 2s. 4d. ....	1½d.
" 2s. 4d., " " " 3s. 4d. ....	2d.

*Improvements in Filters.* P. de Mol and A. Gerken, Brussels, Belgium. Eng. Pat. 676, January 14, 1889. *Sd.*

The filter consists of an outer casing A provided with a cover O fitted with an inlet pipe. In the interior of A three



separate vessels, I, H, G, are superposed. Each of these has a perforated bottom on which rests a disc or discs of cotton fibre; the bottom of G is covered with broken gas carbon, that of H with wood charcoal, and that of I with sponge. The perforated cover C is also sometimes covered with cotton waste. Filtration takes place through the whole depth of the apparatus, the unfiltered liquid entering at O, and leaving at R filtered.—C. C. H.

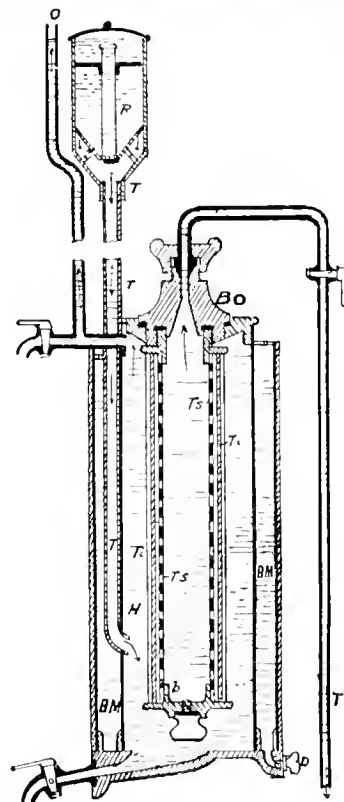
*Improved Filtering Apparatus.* E. Dueret, Paris, France. Eng. Pat. 1462, January 26, 1889. *Sd.*

T's is a perforated tube round which is wound the filtering material T', the whole being supported by the cap B o. The unfiltered liquid enters the chamber R, on the shelves of which some of the grosser matter is separated; passing by the pipe T it enters the vessel H, filters through the material T' and passes from the apparatus by the pipe T''. The stand pipe O allows any air which may be in the apparatus to escape. A jacket B M may also sometimes be used so as to raise the temperature of the liquid during filtration. Decolourising material is also sometimes used between the layers of the filtering material.—C. C. H.

(For drawing see top of next column.)

*Improvements in Apparatus for Automatically Softening, Cleansing, and Filtering Water Continuously or in Quantities as required.* J. Wright, London. Eng. Pat. 2695, February 15, 1889. *Sd.*

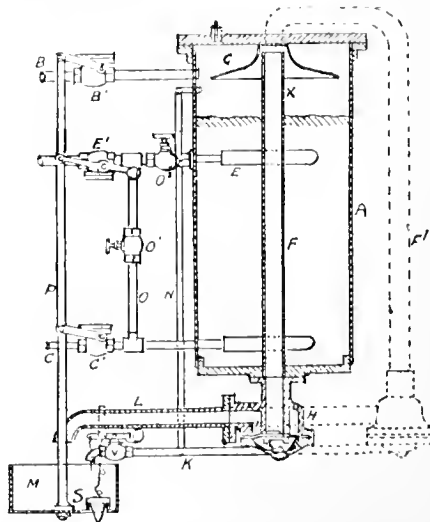
This invention has for its object the provision of such means as will ensure the operations of cleansing or softening and filtering the water being carried on jointly or separately without the expenditure of any power other than that supplied by the water itself; while the apparatus is so constructed as to cause the supply of water to it to automatically govern the starting and stopping of the operations as required; the quantity of water supplied to be cleansed,



softened, or filtered being capable of minute regulation." For the mode by which this is effected the two sheets of drawings attached to the specification must be consulted. There are three claims.—E. S.

*Improvements in Filters.* W. Mather, Salford. Eng. Pat. 3125, February 21, 1889. *Sd.*

This invention is applicable to that class of filters shown in the accompanying Figure, in which the material arrested in the filtering material is subsequently removed by a current of water passed through it in the opposite direction to that of filtration. A is the body of the filter, B the pipe conveying



the unfiltered water, C pipe conveying filtered water from the filter, E, E cleansing pipe. During the time the filter is in operation the pipe N transmits the pressure of the water to the flexible diaphragm I, and closes the end of wash-out pipe F. As soon, however, as the resistance in the filter increases the increased pressure due to such resistance lifts the weighted valve V, and thus allows the water in pipe K to escape into the suspended tank M, which then falls, closes valves B' and C', and opens valve E. Filtration then ceases, and the washing out of the accumulated impurities commences. To ensure the complete removal of the impurities, the tank M is kept down by a further rapid escape of water from pipe L, for as the pressure in K is diminished the diaphragm I falls and allows the dirty wash-water to be rapidly syphoned out of the filter. A reversal of the action occurs by the breaking of the syphon at G, and as the suspended plug S lightens the tank M by allowing the water to escape the counterpoise on V, reverses the valves and filtration again commences.—C. C. H.

*Improvements in Wooden Vessels for Carrying or Holding Corrosive Substances.* P. W. Turner, Glasgow. Eng. Pat. 5373, March 27, 1889. 4d.

The staves of barrels or other vessels are steeped in a mixture of solid paraffin and india-rubber or gutta-percha. —E. G. C.

*Improvements in the Construction of Filters and other Vessels.* G. Cheavin, Boston, Eng. Pat. 6222, April 11, 1889. 6d.

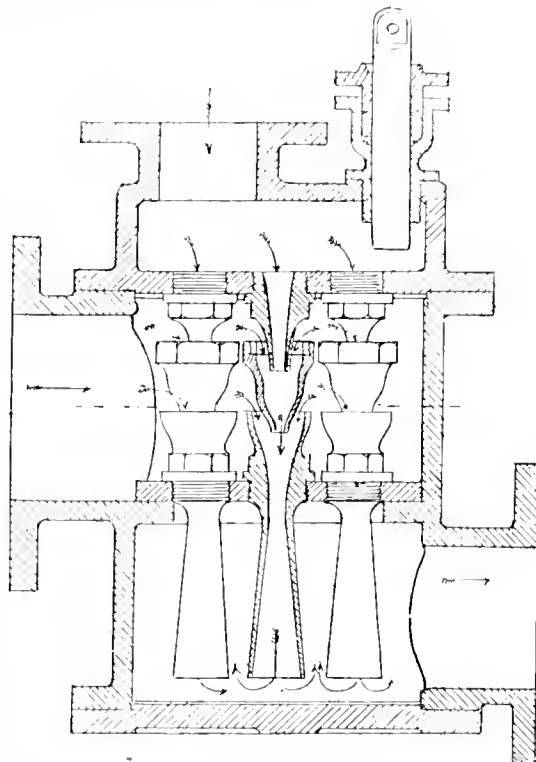
This invention relates to a method of securing plates or diaphragms forming the partitions and covers of filters and other vessels. The edges of the plate, instead of being of the same thickness all round, are made thickest at the end of one diameter and thinner at the end of the diameter at right angles to the first, the thickness varying between the two extremes. A notch is cut at one of the thinnest places. The seat upon which the plate rests consists of a ring fixed inside the vessel, and above the ring are two lugs diametrically opposite to each other. In putting the plate in place, the thinnest part opposite to the notch is first inserted between the ring and one of the lugs, the notch allowing the plate to pass the other lug and drop into its place, when a partial turn of the plate causes it to be wedged between the lugs and ring. The filtering medium employed is preferably "idiocharites," the top and bottom layers being coarser than the middle, and is placed between two discs of pure asbestos cloth.—E. S.

*Improvements in the Construction and Arrangement of Mechanical Retorts for the Treatment of Animal, Vegetable, and Mineral Matters.* R. Haig, Paisley. Eng. Pat. 6242, April 12, 1889. 6d.

This is a modification of a patent for mechanical retorts previously described by T. Rouet in Eng. Pat. 11,584 of 1886. The retort has a vertical shaft, to which are affixed plain rotary discs, one above the other, with stationary partitions between them. The material to be treated, whether animal, vegetable, or mineral, reaches the upper disc from a feeding apparatus, falls over the outer edge of that disc on to the partition, is there acted on by scrapers which sweep it towards the centre, where it falls through an opening to the next rotary disc, is there driven or swept again over the outer edge to reach the next division, and so on until delivered at the lowest point. On its passage through the retort it is exposed to any desired degree of heat. The improvements covered by the present patent consist in arrangements for: 1. Allowing the materials to reach the upper disc from an opening in the cover of the casing, made as near to the centre of the shaft as practicable. 2. Allowing the volatile products to escape through openings in the bottom of the retort instead of at the sides. There are two sheets of drawings.—B.

*Improvements in Apparatus for the Lateral Action or Induction of Fluids.* A. Morton and W. R. M. Thomson, Glasgow. Eng. Pat. 6932, April 23, 1889. 11d.

This is an improvement in the mechanical arrangement of ejector-condensers. The principal object of the improvements is to obtain greater ease and accuracy in the adjustment of the action of the ejector to various quantities and pressures of steam, to temperature and quantity of condensing water, and to height of lift.



The sketch shows an arrangement of several nozzles fitted to one case. For further details the specification and five sheets of drawings must be consulted.—B.

*Process and Apparatus for Cleaning the Filtering Bodies of Chamberland and other similar Filters.* O. André, Paris, France. Eng. Pat. 7050, April 27, 1889. 1s. 1d.

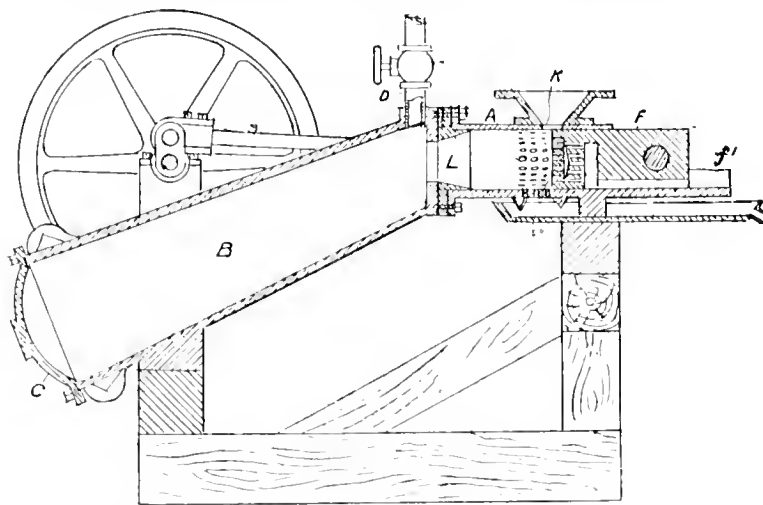
The filtering apparatus, composed of Chamberland tubes, clog rapidly, and consequently are not so convenient for use as they otherwise would be. In the form of filter described by the patentee means are provided to overcome this defect. The Chamberland tubes are arranged in the interior of a cylinder feeding into concentric pipes. Water is forced into the cylinder, passes through the tubes, and is led away by the collecting pipes. The accumulated impurities on the exterior of the tubes are removed by means of jets of water issuing from small tubes contiguous to the filtering tubes, and these are driven with considerable force against the filters. At the same time a current of water or steam may be forced through them from the interior to the exterior. After being fully cleansed filtration can be again resumed as before described.—C. C. H.

*Improvements in Method and Apparatus for Extracting the Liquid or Soluble Constituents from Disintegrated Vegetable Materials.* J. E. Seales, jun., Brooklyn, U.S.A. Eng. Pat. 19,002, November 26, 1889. 11d.

The apparatus described in this specification is intended for the extraction of the soluble constituents from disintegrated vegetable matters. F is a plunger fitting the tube A;

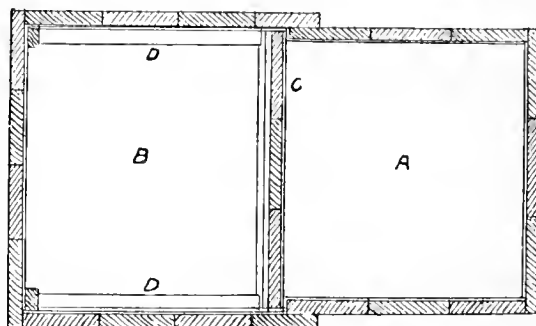
reciprocation of F is secured by the connecting rod and crank as shown. Opening from A through the contracted die L is an inclined strong vessel B with a tight cover C, and fed with water or other solvent by pipe D. The chopped vegetable matter, which is of course more or less fibrous,

is introduced through the hopper K. Reciprocation of plunger F drives the fibrous matter forward, and, aided by the friction of the sides of tube A, assisted by the resistance of the die L, a solid pellet is formed. Each motion of the plunger drives this against the liquid anvil in B, and whilst



the hydraulic buffer so produced compacts the mass the liquid displaced is driven through the interstices of the pellet and so removes the solid constituents. The liquid escapes through perforations shown in the walls of the tube and in the face of the plunger. Such a form of apparatus, the patentee states, is well adapted for the treatment of sugar canes, &c.—C. C. II.

A is the tower, B the shaft for access, C the removable side, D D struts for wedging the several parts of the side to



*Improvements in the Construction of Filters and Reagent-Feeders for the same.* J. W. Hyatt, Newark, New Jersey, U.S.A. Eng. Pat. 19,862, December 10, 1889. 11d.

THE invention consists chiefly in improved means for filtering fluids through a bed of granular materials by introducing the unfiltered fluid at the bottom of the filter bed. A series of inlet strainers is provided in the bottom of the filter bed, the water being forced through the bed to a series of outlet strainers, which may be located in another portion of the bed at its bottom, or in the upper portion a little below the surface. A special strainer is constructed by holding separate rods side by side, with slight interspaces, and preferably in the form of an annular bundle. Means also are provided for supplying a regulated volume of a coagulating substance to the unfiltered fluid in its passage to the filter. For details of the invention the specification and the seven sheets of drawings which accompany it must be consulted. There are five claims.—E. S.

the framework of the tower for the purpose of making tight joints when at work.

There are two sheets of drawings and four claims.—B.

*An Improved Boiling and Precipitating Tower.* G. A. Hanisch, Beuthen, Upper Silesia. Eng. Pat. 20,719, December 24, 1889. 8d.

THE invention refers to washing and boiling towers as used in many chemical processes, and which generally require a periodic cleaning out. The inventor proposes to construct them in such a manner as to ensure a considerable saving of time and materials in the cleaning process. For this purpose he makes one of the sides of the tower completely removable in parts, having previously built an upright shaft to adjoin the tower on that side, in order to allow access thereto without exposing the materials and gases to the outer atmosphere.

The accompanying diagram represents one of the arrangements in horizontal section.

*Improved Method of and Apparatus for Recovering Chemicals from Solutions, or when mixed with Substances which are Volatilizable or Destructible by Heat, especially adapted for Recovering Soda from Waste Liquors of Pulp Digesters.* M. B. Mason, S. D. Warren, and F. Warren, Boston, U.S.A. Eng. Pat. 20,908, December 31, 1889. 8d.

See under XIX., page 408.

## II.—FUEL, GAS, AND LIGHT.

*Note on a Treatise by Carl Otto on Improvements in Heating by Means of Gas at Constant Volume.* Schreier-Kestner. Bull. Soc. Ind. Mulhouse, 1889, 453—458.

ST. CLAIR DEVILLE, in 1868 (Compt. Rend. 67, 1089), advocated the heating of steam-boilers by fires supplied with compressed air; he considered that the heating surface

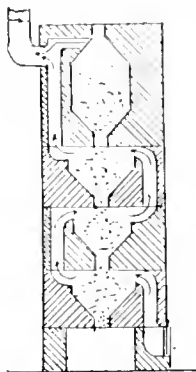
might then be lessened. Bessemer, however (Engineer, July 1869, 19), declared against the system of employing compressed air, holding that the compression to be effected would necessitate the employment of a machine which would in itself consume more work than corresponded to the actual gain subsequently effected, and so, on the whole, a loss would result. It is against this statement that Otto protests. In fact, on theoretical grounds, he is led to the conclusion that working under a pressure of 4 kilos. an economy of 23 per cent. is obtained, from which must be subtracted only the expense of compression, which, however, is compensated for by other advantages, such as more complete combustion, reduction in quantity of smoke, possibility of reducing size of apparatus. The author proceeds to point out that from calorimetric study of the combustion of oils it has been ascertained that steam-boilers can actually furnish 85 per cent. of their heat in steam. Otto thus obtains from the fuel more energy than it is by any means capable of giving. Otto arrives at a false result in taking count of the loss resulting from work absorbed by the chimney draught; instead of taking the temperature of the gases at the base of the chimney, he takes it at the fire.

—T. L. B.

#### PATENT.

*Improvements in Apparatus for the Combustion of Fuel.*  
F. Dinz, St. Jean des Vignes, France. Eng. Pat. 4830,  
March 19, 1889. 11d.

SEVERAL modifications are described and illustrated in the specification, but the essential features will be understood from the accompanying Figure. The arrows show the course



of the gases which come in intimate contact with the fuel. The latter is fed continuously into the uppermost central chamber from which it drops through the contracted orifice into the chamber next below it and so on, the ash ultimately falling into the ash box at the bottom of the series.—O. H.

result of the prospecting was that a thick stratum of rock salt was struck, and the salt-mining industry has in consequence been greatly developed.

This satisfactory outcome to his former labours, and the proof it affords of the value of the assistance which the Imperial Association is in a position to render, encourage the author to hope that his present report may be of some use.

As much as 12 years ago it was pointed out by different engineers that the Balakan naphtha differs considerably from that of Pennsylvania; thus, only about 37 per cent. of kerosene is obtainable from the Caucasian naphtha, while the Americans succeed in getting as much as 70 per cent. from theirs.

The main object of owners since the opening of the Baku industry has been to obtain from Apshehon naphtha a product resembling American kerosene as nearly as possible, and so prevent the latter from flooding the Russian markets. The Baku owners have been blamed for this by some authorities, it being thought that they ought to work out a special grade of oil. This objection, however, appears to the author unjust, competition necessitating the production of an oil undistinguishable by the consumer from American kerosene. The object at present aimed at in the Russian refineries is to obtain 37 per cent. (nearly) of kerosene and 55—50 per cent. of naphtha residuum, which is much used along the Volga for fuel. This last is so important as to be no longer a mere by-product, and hence naphtha has become a heating material as well as a lighting material. Up till 1878 this residuum or refuse was procurable for  $\frac{1}{2}$ —1 kop. per pud. Now it fetches 1 kop. per pud. The question is not yet settled as to whether this method of treatment is the most advantageous; for 12 per cent. of oil can be got by simple distillation from the residuum. These oils are heavier than petroleum (kerosene), but will burn in lamps. They are the so-called "solar oils" (heavy naphtha oils). The object of the writer is to introduce them into general consumption as a substitute for kerosene. The advantages of these oils are their exceptional safety, due to high flashing point. The author recommends their separation from the refuse, because he considers that to employ naphtha for lighting purposes is the most suitable use to which it can be put, and as for the employment of naphtha for heating purposes, that has already reached such a pitch of development that there is no danger of its ever being abandoned. This is due to the owners not knowing what use to make of the refuse. The specialist, however, desires to find out the most suitable use for a material.

It is not proposed in the present paper to deal with the products obtained by fractional distillation ("cracking") from heavy naphtha constituents. This process is too slow for commercial requirements. The product here to be considered is obtained by ordinary distillation, and is intermediate between kerosene and lubricating oils. It has been largely obtained in lubricating oil works, but has not hitherto been utilised. The reasons for this are—(a) the difficulty of obtaining a suitable lamp; (b) the difficulties and expense of refining. Its specific gravity is 0.870, and its flashing point from 100°—110° C. This is the so-called solar or intermediate oil.

As regards the lamp question, Kumberg obtained a prize for a suitable lamp in 1883, but his lamp has not been generally adopted. Since then several cheap lamps have been invented, and this has caused the author to re-direct his attention to the application of solar oil for lighting purposes. Last autumn Voïslar introduced a lamp said to burn the oil well, and another has since been invented by Bluman. This difficulty would doubtless be overcome if the oil came into more general use.

This is, however, not the only difficulty, for the trouble of purification renders the solar oil as yet more expensive than kerosene, and though it has the advantage of greater safety, this does not make up for the difference in price. The duty on solar oil is 30 kop., that on kerosene 40 kop.; but this difference is more than swallowed up by cost of refining. Mendeleeff states that the refining of solar oil is very difficult. The author used sulphuric acid (sp. gr. 1.835) and soda-lye (sp. gr. 1.23), employing 4 per cent. sulphuric acid and such an amount of caustic soda as to give 1—1 $\frac{1}{4}$

### III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

*A Recommendation for the General Employment of Heavy Naphtha Oils for Lighting Purposes.* K. Lissenko.  
Bull. Soc. Impér. Poly. Russie, 1890, 24, 1—14.

THIS is a paper read before the Imperial Russian Technical Association. The writer commences by observing that some 15 years ago he presented a report on the subject of the rock-salt deposits situated between Bakkmoït and Slavausk, with the view of inducing the Ministry of Finance to make grants for the purpose of prospecting. The Imperial Association received his proposals with favour, and made a grant of 50,000 roubles for that object. The



per cent. in the oil, found that it could be easily purified. The precipitation must take place hot, and there is a greater loss but no special difficulty. The oil, though burning well in the Makaroff lamp, varied in colour. M. Ropps found the purification satisfactory, and a sample was sent through the Nobel Co. to Mr. Redwood in England. On another occasion the author tried to obtain oil of the same degree of purity, using sulphuric acid of sp. gr. 1.835, but did not succeed, and consequently concluded that he had not got to the bottom of the question. The ordinary criterion for kerosene, *i.e.*, to stir it with sulphuric acid of sp. gr. 1.50, when it should remain colourless or nearly so, does not hold for solar oil.

The oil submitted to the lamp committee by the Baskoff works, though it burned well in some lamps, was badly purified. To get a practical result the heavy oils will have to be refined to a corresponding equality with kerosene. At Baku it is held that the degree of purity obtained depends on the strength of the acid and the temperature employed. It is for this purpose of lowering the temperature to the desired point that such large coolers are used in that town. The author's experiments have confirmed those of other experimenters in Baku.

The result of a number of experiments with acids of different strengths, and also with Nordhansen sulphuric acid, and at various temperatures, went to show that the degree of purity attained depended on—

1. *Strength of Acid.*—Thus, when 4 per cent. of acid, sp. gr. 1.835 or 1.840, and cold treatment, were employed, the result was to give an oil of the standard sup. W. 21 mm. and 35 mm. respectively. Thus the small difference in specific gravity of 0.005 exercises an enormous influence on the result.

2. *Quantity of Acid.*—Oil refined with 3 per cent. and 4 per cent. of strong acid gave sup. W. 35 mm. and 30 mm. More extended experiments will doubtless show that there is a limit beyond which the influence of quantity will not be so apparent.

3. *Temperature.*—When treated with acid hot the resulting oil is heavier.

In Baku it is thought that the colour may be effected by the presence of nitric acid in the sulphuric acid. This is doubtless true, and hence the rule ought to be observed that only sulphuric acid free from oxides of nitrogen should be employed.

From these experiments it is clear that the cost of refining depends on the standard adopted. Ropps' purified oil is sold, it would seem, at 4 rh. 50 kop. per pud wholesale. The author considers that sup. W. 30 mm. ought to be a sufficient degree of purity, for which 4 per cent. of sulphuric acid of sp. gr. 1.84 may be used. The acid supplied at Baku will therefore require careful concentration, and he considers that the Baku acid works ought to give their attention to the manufacture of both fuming and anhydrous acid.

It must be remembered that the refining of the heavy oils requires three times as long as that of kerosene, besides the provision of a greater number of tanks and more elaborate heating apparatus, and consequently the difference of 10 kop. duty will be more than counterbalanced by the extra cost.

As there are so many grounds which make the general introduction of the heavy oils advisable, *e.g.*, their greater safety and the more economical exploitation of naphtha which would result, and as the cost of refining cannot, as far as can be seen at present, be much reduced, the author submits the following proposals:—

- (1.) That the Ministry of Finance be requested to make an alteration in the duty upon the various oils of such a kind that those hitherto rated at from 40—50 kop. should be in future rated at from 20—30 kop.
- (2.) That the latter should be dyed red, and that the employment of the dye should be understood as guaranteeing a sufficient degree of purity.

These proposals, if carried out, the author thinks, would lead to the general employment of the heavy oils for lighting purposes.

## PATENT.

*Improvements in and connected with Retorts for the Destructive Distillation of Shale or other Oil-yielding Minerals.* N. McFarlane Henderson, Linlithgow. Eng. Pat. 6726, April 20, 1889. *Sd.*

The minerals are first treated in a series of upper cast-iron retorts for the purpose of obtaining the crude oil, after which they are passed into a common intermediate chamber and thence into a series of lower fire-brick retorts, where they are acted on at a higher temperature by steam in order to drive off the carbonaceous and nitrogenous constituents remaining in the minerals as completely as is practically possible. The vapours evolved in the lower retorts are passed upwards and may proceed through the same outlet pipes as the oily vapours from the upper retorts, or through separate outlet pipes. On their way up from the lower retorts these highly heated vapours act on the minerals temporarily lodged in the common intermediate chamber, and distil off therefrom any oily vapour remaining or requiring for its evolution a greater heat than that acting in the upper retorts.—D. B.

## IV.—COLOURING MATTERS AND DYES.

*Production of Indigo.* Centralb. f. die Text. Ind. No. 3, 1890.

INDIGO in India is sown towards the end of February, and when the young plant has attained a height of from 6 to 8 in., it remains stationary till a rainy season, when it continues its growth. If this rain begins after the time of the hot winds, about the end of April, a good season may with certainty be counted upon, but if the rain only comes, as is usually the case, at about the middle of June, an average season will be the result, for which the plant will be ripe for cutting during the first week in June. After the plant has been cut to about 6 in. from the ground, it is brought in carts to the factory. There it is laid in the fermenting vats, which, when completely full, are covered over with boards, in order to prevent any of the plants being washed away when the water is run off. Water is then allowed to enter these vats, and the process of fermentation commences, causing the soluble colouring matter to separate out from the plant. The fermentation is caused by a micro-organism, and so long as there is the necessary nourishment for the growth of these bacteria, the production of the colouring matter will continue.

The fermentation lasts about 10 or 12 hours. After this time the nourishment for the bacteria begins to fail, and butyric acid fermentation, and afterwards putrid fermentation, set in, neither of which permit of the further formation of colouring matter. At the moment when the colour-forming fermentation is over, the yellowish-green liquid is run into another set of vats standing underneath the first series, and provided with a mechanical apparatus for heating the liquid, in order to bring it in contact with the atmosphere. The colouring matter, now insoluble in water, sinks to the bottom, forming a thick layer. If the water be now run off, crude indigo paste is obtained, which, by various methods of treatment, is freed from moisture and impurities. The colouring matter is pressed into sheets 1 metre square and 3 in. thick; then cut into cubes about 3 in. large, and lastly subjected, for several months, to an air-drying process, after which it is ready for packing and exporting.

The complaint which is often made as to the small yield of indigo from the plant is founded upon the comparatively short time allowed (10—12 hours) for the fermentation, during which the formation of any colouring matter was possible. The improved process discovered by Schrottky is based upon the lengthened period of fermentation, and the prevention or deferring of the butyric acid and putrid

fermentation. He found that those factories using water saturated with vegetable or animal matters obtained both less and an inferior quality of indigo than those which used pure water or water containing saltpetre. In the course of his experiments, therefore, Schrottky added nitrates to the water in the fermentation vats, in order to afford nourishment to the bacteria for a greater length of time, with the result that he obtained a yield of 12 to 15 per cent. more than he previously obtained. Further experiments were made to ascertain how long putrid fermentation might be postponed, and how long it was possible for the formation of colouring matter to take place. In this case the addition of carbolic acid was very successful, and the period of extraction was thereby so much lengthened that a much larger quantity of the colouring matter was obtained from the plant than was possible before. The increased production appears, however, to take place at the expense of quality. (See also this Journal, 1890, 224.)

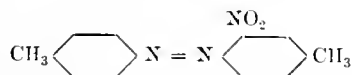
*Azo- and Azoxytoluenes.* J. V. Janovsky. *Monatsh.* **10**, 585—601.

MONONITRO-PARAZO-TOLUENE is best prepared by treating azo-toluene with 5 parts of nitric acid of sp. gr. 1.43; the temperature should not rise above 30°, otherwise the dinitro-compound is also formed. It is readily soluble in ether, acetone, and light petroleum, and melts at 80°. When reduced with tin and hydrochloric acid it gives para-toluidine (m.p. 43°—44°) and meta-para-tolylenediamine, melting at 88°.

The most important reactions of the two tollylenediamines obtained from para-toluidine are given below; in both cases a neutral or slightly acid solution of the hydrochloride is employed:—

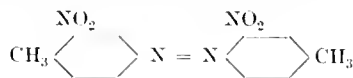
—	<i>o</i> -Tolylenediamine [CH <sub>3</sub> :NH <sub>2</sub> :NH <sub>2</sub> =1:2:4].	<i>β</i> -Tolylenediamine. [CH <sub>3</sub> :NH <sub>2</sub> :NH <sub>2</sub> =1:3:4].
Ferric chloride.	No change at first, after standing for a long time an orange colouration.	A wine-red colouration.
Potassium bichromate.	A yellowish-brown colouration.	A reddish-brown precipitate.
Potassium ferricyanide.	Olive-green crystalline plates.	A dark red colouration.
Bromine water.	A yellowish-white precipitate.	Brown flocks and a magenta-red solution.
Platinum chloride.	A yellowish-brown colouration.	A reddish-brown precipitate.
Gold chloride.	A brown precipitate.	A red solution with blue reflex and metallic mirror in the cold.
Potassium nitrite.	In very dilute solutions a golden-brown colouration, in concentrated a brown precipitate.	No colouration, but a salmon-coloured precipitate.
Solution of bleaching powder.	A reddish-brown colouration and then a light brownish-yellow precipitate.	A dark red colouration, then an olive-green precipitate.

Since the tollylenediamine (m.p. 88°) obtained (together with *p*-toluidine) from nitro-parazo-toluene gives all the reactions of *β*-tolylenediamine, the two compounds are identical, and the nitro-compound has the constitution—

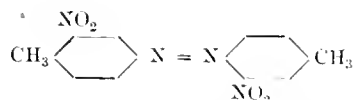


The reactions given above can be employed, even in presence of other substances, for the detection and identification of tollylenediamines, compounds which so frequently result from the decomposition of azo-dyes.

Dinitro-azo-toluene (m.p. 114°) is obtained by treating parazo-toluene with nitric acid of sp. gr. 1.51. When reduced with an alcoholic solution of stannous chloride it yields an intensely yellow diamido-compound; this base dyes yellow, but the hydrochloride is colourless. When dinitro-azo-toluene is treated with tin and hydrochloric acid it gives meta-para-tolylenediamine (see above), so that its constitution is represented by the formula—

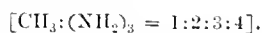


or

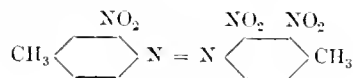


The two trinitro-azo-toluenes, melting at 138° and 189°, respectively, yield meta-para-tolylenediamine (m.p. 88°) and triamidotoluene on reduction with stannous chloride and fuming hydrochloric acid; the two bases can be separated by converting them into the sulphates and adding alcohol to the aqueous solution of the mixed salts, whereon the meta-para-tolylenediamine sulphate is precipitated.

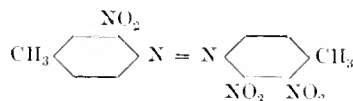
The triamidotoluene obtained, crystallises in plates and becomes coloured on exposure to the air; the hydrochloride is crystalline and gives the following very characteristic reactions:—In dilute solutions ferric chloride produces no change at first, but after some time the solution turns brownish red. Potassium bichromate gives an olive-brown colouration and bleaching-powder solution, a rose-red colouration, and then a brown precipitate. Potassium nitrite, in very dilute solutions, produces a rose-red colouration, in concentrated solutions a dark brown precipitate. Sulphuric acid, mixed with a few drops of nitric acid, produces a steel-blue colouration analogous to that obtained with *α*-triamidobenzene. This triamidotoluene does not yield an eurdine, and has the constitution—



Since both the trinitro-azo-toluenes give meta-para-tolylenediamine and the same triamidotoluene they must be represented by the formula—



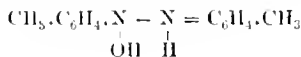
and



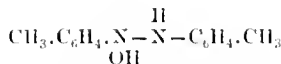
respectively.

Para-nitrotoluene gives two azoxytoluenes, an *α*-compound melting at 70° and a *β*-compound melting at 75°. The two substances are best prepared by gradually adding a quantity of zinc dust, rather less than that theoretically necessary to produce azotoluene, to a warm mixture of nitrotoluene and caustic soda (25°—30° B.). The reduced mass is then dissolved in hot glacial acetic acid, and the solution cooled to 20°—25°, whereon parazotoluene crystallises out. The mother-liquors deposit the *β*-azoxytoluene on standing, mixed with a small quantity of a compound melting at 109°, and on adding water to the filtrate the whole of the *α*-compound is precipitated. Both compounds can be easily purified by recrystallising from light petroleum. The relative quantity of the two products depends on the concentration of the soda and on the quantity of zinc dust used. These two azoxytoluenes have the same molecular weight, and both give paratoluidine on reduction with tin and hydrochloric acid. When treated with stannous chloride in warm alcoholic solution they both give yellow crystalline

compounds which are insoluble in acids, and are probably hydroxy-derivatives of the constitution—



and



respectively.

When  $\alpha$ -azoxytoluene is treated with nitric acid of sp. gr. 1.42 it gives a golden mononitro-compound which crystallises well from alcohol, melts at 51°, and on reduction gives an amido-azoxytoluene.  $\beta$ -azoxytoluene yields under the same conditions a crystalline nitro-compound melting at 82°. Both azoxytoluenes dissolve in fuming  $\text{HNO}_3$  (sp. gr. 1.51) yielding one and the same trinitro-compound which crystallises in prisms and melts at 196°.—F. S. K.

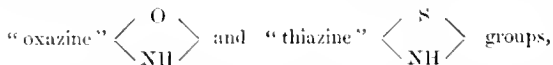
*Patent Blue and Acid Violet N.* E. Illich. Chem. Zeit. **13**, 1702—1704.

A NOTICE of patent blue has already appeared (this Journal, 1890, 53), and the present article deals with the technical uses of new shades of the original colour which appear in commerce as calcium salts of the sulphonated colouring base. Their advantages are stated to be:—1. Characteristic brilliant shade. 2. Very even dyeing. 3. Great fastness to alkalis and lime. 4. Considerable fastness to light. Whilst ordinary patent blue dyes a greenish blue, the shade “superfine and N” gives a pure blue, which shows to advantage by artificial light. The shade “I” closely approaches that of indigo carmine, and possesses many advantages over the ordinary substitute for indigo carmine, namely a mixture of acid violet and acid green. Acid violet N has a different chemical composition from ordinary acid violets, and possesses very similar qualities to patent blue. All these colouring matters dye wool in an acid bath with or without glauher salt or alum. On a chrome mordant they will dye in a neutral bath or in presence of acetic acid, which fits them for use in the same bath with alizarine dyes. The shades produced will stand a moderate amount of milling if care be taken to rinse well immediately afterwards, otherwise the colour will slowly bleed into the white. The dye baths should be preferably of wood, and the presence of copper should be avoided for the finer shades, especially on silk.

—G. H. B.

*Azo-Colouring Matters containing the Quinone Group.* F. Kehrman. Chem. Zeit. **14**, 93 and 146—147.

According to Witt's nomenclature *chromogens* are bodies generally belonging to the aromatic series which yield colouring matters having an affinity for animal and vegetable fibres on the introduction of *chromophors*, of which the nitro, azo, and quinone groups are examples. Although the function of chromophors is generally ascribed chiefly to the azo—



yet there appears reason to view certain colouring matters containing these groups as owing their characteristic properties to other possible chromophors which they may contain; thus Nietzki (Chem. Zeit. Rep. **13**, 343) ranks Weselsky's resorcinol colouring matters not as owing their essential qualities to their being “oxazine” or “phenoxazine” derivatives but to their containing the quinone imido group.

The author here remarks that the “oxazine” group in such a body as “phenoxazine” (oxydiphenylamine)  $\text{C}_6\text{H}_5 \cdot \text{ON} \cdot \text{H} \cdot \text{C}_6\text{H}_5$  and the “thiazine” group in thiodiphenylamine  $\text{C}_6\text{H}_5 \cdot \text{SN} \cdot \text{H} \cdot \text{C}_6\text{H}_5$  allow of the comparison of these bodies with hydrophenazine (diphenylene hydrazine)  $\text{C}_6\text{H}_5 \cdot \text{N}_2 \cdot \text{H}_2 \cdot \text{C}_6\text{H}_5$  better than with phenazine (azophenylene)

$\text{C}_6\text{H}_3\text{N}_3\text{C}_6\text{H}_4$ , the former corresponding with quinol  $\text{C}_6\text{H}_4(\text{OH})_2$ , the latter to quinone  $\text{C}_6\text{H}_4\text{O}_2$ . It is therefore conceivable that phenazine may be viewed as a chromogen, a position not held by “phenoxazine” and “phenthiazine” (thiodiphenylamine). Although attention has been paid to the effect of the introduction of various chromophors into artificial colouring matters, yet hitherto none has been prepared which can be viewed as a combination of an azo compound with a quinone, on account of the difficulty of synthesising any such body. The author has attempted to attain this object by starting with hydroquinone, but has been unable to diazotise it on account of its suffering oxidation in the process.

In conjunction with Tiesler (J. prakt. Chem. **40**, 480) the author has found that the hydrogen atom attached directly to the nucleus in chloro-*p*-dihydroxyquinone  $\text{C}_6\text{H}(\text{OH})_2\text{ClO}_2$  is easily replaceable by halogens and ( $\text{NO}_2$ ). Arguing from this he expected the ready formation from it of mixed hydroxyazoquinones, and verified this deduction not only in the case of monochlorodihydroxyquinone, but also in that of the paradihydroxyquinone of Nietzki and Schmidt and its substitution products, chlorodihydroxyquinone and nitrodihydroxyquinone, as well as in that of the dihydroxytoluquinone of Zincke and Hagen and hydroxynaphthoquinone. The reaction does not occur, however, in those bodies in which the (OH) group is not in the same nucleus as the quinone group. Hydroxyanthraquinones apparently yield no azo-colouring matters.

The class of substances thus produced are generally yellow-red to brown-red bodies soluble in the cold in alkalis and forming crystallisable salts.

An example of the mode of formation of one will serve as a type:  $\text{C}_{10}\text{H}_7(\text{OH})\text{O}_2 \cdot \text{N}_2 \cdot \text{C}_6\text{H}_5$ , *hydroxynaphthoquinone azobenzene*, is formed by dissolving the finely powdered sodium salt of hydroxynaphthoquinone (1 mol.) in 40 to 50 times its weight of boiling water, and stirring in a solution of diazobenzene chloride (1 mol.) to which has been added sodium acetate to remove free hydrochloric acid. The separation of the new substance begins at once and is quantitatively complete in half an hour. It is yellowish-red in colour, insoluble in water, slightly soluble in boiling alcohol and benzene, fairly soluble in boiling glacial acetic acid, from which it crystallises in bright red felted needles melting at 220—225° C.; dried at 120° C. it gave on analysis the following figures C 68.60, H 3.93, N 10.73 parts per cent. respectively instead of the theoretical values, C 69.06, H 3.59, N 10.07. The shade of colour similar to but intenser than that of hydroxybenzene azobenzene and the ready solubility of the substance in dilute alkalis forbid the supposition that it should be regarded as a “hydrazide” (hydrazone)  $\text{C}_{10}\text{H}_7\text{O}_3 \cdot \text{N} \cdot \text{NH}(\text{C}_6\text{H}_5)$ . The presence of the quinone group appears therefore to have an inconsiderable influence on  $\beta$ -naphthol azo-colouring matters; the same has recently been shown to be true of the ketone group by Witt (Ber. **21**, 321). Tetrazodiphenyl from benzidine (paradiamidodiphenyl) gives when treated with excess of hydroxynaphthoquinone a dark brown-red colouring matter which is doubtless tetrazodiphenyldihydroxynaphthoquinone  $\text{C}_{10}\text{H}_7(\text{OH})\text{O}_2 \cdot \text{N}_4 \cdot (\text{C}_6\text{H}_5)_2\text{N}_2\text{O}_2(\text{OH})\text{C}_{10}\text{H}_7$ . By not using an excess of hydroxynaphthoquinone but supplying the place of a portion of it with some amine or phenol mixed tetrazo-compounds can be obtained.—B. B.

#### PATENTS.

*A Process for the Production of Azo-Colours from the Para-diamines of Stilbene and Fluorene.* C. A. Martius, Berlin, Germany, May 31, 1886. Amended, November 30, 1889. 6d.

The revised claim is as follows:—“The production of azo-colours which dye cotton direct from a soap-bath, by combining one molecule of the tetrazo-compounds of stilbene or fluorene . . . . . with two identical or different molecules of an amine or phenol, such as  $\alpha$ -naphthylamine,  $\beta$ -naphthylamine, dimethylamine, diethylaniline, diphenylamine, phenyl-naphthylamine, tolyl-naphthylamine, carboic acid, cresote, resorcin,  $\alpha$ -naphthol or  $\beta$ -naphthol.

or their sulphonic or carbonic or sulphocarbonic acids." For further details the amended specification itself must be consulted. The original specification has been abstracted in this Journal, 1887, 437.—O. H.

*Processes for the Production of Azo-Colours that Dye Cotton direct from a Soap-Bath.* C. D. Abel, London. From the "Actiengesellschaft für Anilin-Fabrication," Berlin, Germany. Eng. Pat. 6687, May 6, 1887. Amended, November 30, 1889. 8d.

THE processes have already been described in this Journal, 1888, 43. The emendations, which in no way affect the claims, are chiefly literal, except in the example B (1) (see abstract already cited), viz., the production of a mixed azo-colour from benzidine,  $\beta$ -naphthylamine disulphonic acid R, and  $\beta$ -naphthylamine. It was stated in the original specification that "instead of using naphthylamine in a hydrochloric aqueous solution it can also be used as a free base in an alkaline solution." The word *alcoholic* is now substituted for *alkaline*.—O. H.

*Improvements in the Manufacture of Colouring Matters.* O. Murray, London. From G. C. Zimmer, Mannheim, Germany. Eng. Pat. 13,798, September 24, 1888. 6d.

"This specification has been reprinted in consequence of an amendment made by order of the Comptroller prior to the sealing of the patent." The emendations are not, however, shown. (Compare this Journal, 1889, 610.)

—O. H.

*Improvements in the Manufacture of Red Colouring Matters suitable for Dyeing and Printing.* I. Levinstein, Manchester. Eng. Pat. 3048, February 20, 1889. 6d.

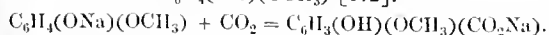
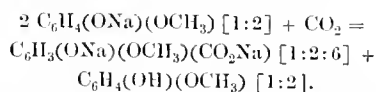
THIS specification describes the preparation of a base by heating *m*-xylydine with sulphur and combining its diazo-compound with phenols or their sulphonic acids, or using the sulphonic acid of the base for diazotisation and combinations. (See also this Journal, 1889, 980.)—T. A. L.

*Improvements in the Manufacture of Picric Acid.* E. Eisenmann, London, and A. Arche, Vienna, Austria. Eng. Pat. 4539, March 15, 1889. 4d.

CARBOIC acid is converted into phenoltrisulphonic acid by the aid of sulphuric acid, and this is transformed into picric acid by means of sodium nitrate. The temperature need not exceed 100°, whilst the yield of pure picric acid is equal to about 155 per cent.—D. B.

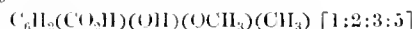
*A Process for the Production of Orymethoxybenzoic Acids.* J. Y. Johnson, London. From Dr. F. von Heyden Nachfolger, Radcben, Germany. Eng. Pat. 5785, April 4, 1889. 6d.

THIS is an application of Kolbe's salicylic acid synthesis to guaiacol, cresol, and eugenol, in order to obtain the carboxylic acids of these compounds. The process consists in either passing carbonic acid over an alkaline salt of guaiacol heated to a temperature above 100° C. or in performing this operation under pressure. The reactions can be respectively represented as follows:—

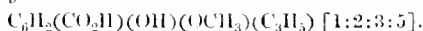


When the reaction is complete the product is dissolved in water and a mineral acid added when the guaiacol carboxylic acid crystallises out with 2 molecules of water. When anhydrous it melts at 148°—150° C. In an aqueous solution it gives a blue colour with ferric chloride and is decomposed on heating into guaiacol and carbonic acid. By substituting

cresol and eugenol for guaiacol in the above process, the following compounds are respectively obtained:—*Cresol carboxylic acid*—



and *Eugeninic acid*—



The acids are to be employed in the manufacture of azo-dye-stuffs.—T. A. L.

*Production of an Imide Compound of Fluorescein Chloride and of Colouring Matters therefrom.* O. Murray, London. From "The Farbwerke vormals Meister, Lucius and Brünig," Höchst, Germany. Eng. Pat. 6134, April 15, 1889. 6d.

WHEN fluorescein chloride is heated with concentrated aqueous ammonia to 200° C. for 6—8 hours, water is given off and an imide is formed having the composition  $\text{C}_{20}\text{H}_{11}\text{Cl}_2\text{NO}_2$ . The same compound is obtained by heating fluorescein chloride with sal-ammoniac in presence of lime and zinc chloride, but in this case coloured basic products are also formed, part of the chlorine being replaced by amidogen. The imido-compound of fluorescein chloride is colourless and melts at 235° C. It is insoluble in water and aqueous acids or alkalis, but dissolves easily in warm alcohol, benzene, or chloroform. It is not acted on by concentrated hydrochloric and sulphuric acids nor by boiling with zinc powder in an alkaline alcoholic solution. When fused with fatty and aromatic bases or their hydrochlorides in presence of zinc chloride to 270°, it forms red to bluish-violet colouring matters soluble in alcohol which on sulphonation can be rendered soluble in water.—T. A. L.

*Improved Apparatus for the Production of Colouring Matters.* J. E. Bedford, Leeds. Eng. Pat. 6778, April 23, 1889. 6d.

AN apparatus devised for the purpose of subjecting the lichens known as orchella weed to the prolonged action of ammonia and atmospheric air in presence of water in order to produce colouring matters. The process is considerably accelerated by using oxygen in place of air or together with it. In order to expose as large a surface as possible to the action of the gases, a vertical vessel is employed containing a number of trays, the edges of which are turned downwards so that they retain a certain portion of the gases pumped in at the bottom of the vessel. Scrapers fixed to a vertical shaft prevent the deposit of colouring matter on the upper surface of the trays and also serve to agitate the liquid. The deposition of the colouring matter is further aided by the application of heat. A vertical section of the vat accompanies the specification.—T. A. L.

*The Manufacture of Dyestuffs from Oxyethers of Alpha-Naphthylamine and their Sulpho Acids.* S. Pitt, Sutton, Surrey. From L. Casella and Co., Frankfort-on-the-Maine, Germany. Eng. Pat. 7067, April 27, 1889. 6d.

THE oxyethers of  $\alpha$ -naphthylamine are obtained from the  $\alpha$ -nitronaphthols by first converting them into the ethers and subsequently reducing the compounds, or the naphthol ethers may be nitrated and then reduced. According to the latter method the process is performed as follows:—17·2 kilos. of naphthol ethyl ether are added to 30 kilos. of nitric acid of 40° B. at a temperature below 40° C. The nitro-compound is washed with water and reduced with 20 kilos. of iron in presence of hydrochloric or acetic acid. The reduction product may be purified by precipitation as hydrochloride or sulphate. The methyl, amyl or benzyl ethers may be substituted for the ethyl ether above mentioned. The bases are scarcely soluble in water, but dissolve easily in alcohol and ether, giving solutions having a violet fluorescence. To convert the bases into sulphonic acids they may be directly sulphonated or the nitro-naphthol ethers can be sulphonated and reduced. A better method, however, is to convert the naphthol sulphonic acid into the

ether and nitrate, and reduce it, or the naphthol ethers may be sulphonated, nitrated, and reduced. According to the former method 24.6 kilos. of sodium naphthol sulphonate are dissolved with 4 kilos. of sodium hydrate in 200 litres of 50 per cent. alcohol. 11 kilos. of ethyl bromide are added and the mixture is heated for several hours to 60°–80° C. The naphthol ethyl ether sulphonate crystallises out on cooling in large silvery crystals, and after drying is added to cold nitric acid of 40° B. in which it dissolves with a dark yellow colour. The solution is poured into water and the nitro-sulpho-naphthol ethyl ether precipitated by the addition of salt. This on reduction yields ethoxy- $\alpha$ -naphthylamine sulphonic acid in the form of colourless needles sparingly soluble in water. These products are all capable of combining with diazo-compounds to form dyestuffs, and as they themselves contain a free amido group the compounds can be rediazotised and combined with phenols and amines. The two following examples describe the preparation of the dyestuffs. (1.) 30 kilos. of naphthylamine disulphonic acid are diazotised and combined with a solution of 21 kilos. of methoxy- $\alpha$ -naphthylamine hydrochloride, when the dyestuff forms immediately and precipitates. (2.) 34 kilos. of benzidine disulphonic acid are diazotised and combined with amido naphthol ether. The product is rediazotised and forms a sparingly soluble tetrazo-compound which is combined with 50 kilos. of  $\alpha$ -naphthol- $\alpha$ -sulphonic acid. The colouring matter dyes wool, silk, and cotton a dark blue shade. The colouring matters derived from diazo-compounds and the naphthol ether sulphonates give red to violet shades. On diazotisation they form yellow to brown tetrazo-compounds which when combined with phenols or amines produce dyestuffs of dark greenish blue shades.

—T. A. L.

*Manufacture of Green and Violet Colouring Matters.* C. D. Abel, London. From "The Actiengesellschaft für Anilinfabrication," Berlin, Germany. Eng. Pat. 7550, May 6, 1889. 6d.

A PROCESS for condensing methyl or ethylbenzylsulphonic acid with aromatic aldehydes and oxidising the leucosulphonic acids thus formed, so as to obtain green and violet dyestuffs. 50 kilos. of methyl or ethylbenzylaniline are gradually added to 120 kilos. of fuming sulphuric acid at 40°–50° C., and the melt is heated to 80° C. until a test dissolves in dilute caustic alkali. 100 kilos. of water are then added, and the sulphonic acid is precipitated by adding 100 kilos. of caustic soda of 40° B., after which it is filter-pressed and dried. 80 kilos. of ethylbenzylsulphonic acid, 34 kilos. of benzaldehyde, and 30 litres of water are heated, under an inverted condenser, at 110° C. for 78 hours, with constant stirring, 1,000 litres of water are then added, the solution neutralised with caustic soda, and the leucosulphonic acid precipitated at 30° C. with hydrochloric acid. In order to oxidise it, 100 kilos. of the dry acid and 31 kilos. of caustic soda of 40° B. in 400 litres of water are mixed with 120 kilos. of peroxide of lead (containing 34 per cent. of  $PbO_2$ ) suspended in 1,500 litres of water. The mixture is kept at 20° C., and the oxidation effected by adding, as quickly as possible, 45 kilos. of sulphuric acid of 66° B. in 300 litres of water. After stirring for some time, 30 kilos. of soda in 200 litres of water are added, the lead removed by filtration, and the colouring matter precipitated by the addition of salt. It is again dissolved in 1,000 litres of cold water, filtered and precipitated with brine, after which it is filter-pressed and dried. The colouring matter is readily soluble in water, less so in alcohol. Dilute mineral acids precipitate the free acid, whilst concentrated acids turn the green solution of the colouring matter yellow. Free alkalis destroy the colour of the solution.—T. A. L.

*Improvements in the Manufacture of Blue Colouring Matter for Laundry, Bleaching, and other Purposes.* M. Williams, Bootle, and J. Aseough, Handsworth. Eng. Pat. 9442, June 7, 1889. 4d.

FARINA, sago or rice flour, starch, china clay, or other suitable substances are to be mixed with a soluble blue,

and subsequently treated with borax, boracic acid, or other boron compound for use as "antiseptic blue" for household purposes.—T. A. L.

*Process for the Production of Mixed Azo-Colours.* P. Jensen, London. From "The Actiengesellschaft für Anilinfabrication," Berlin, Germany. Eng. Pat. 15,296, December 12, 1885. Amended December 27, 1889. 6d.

THIS amended specification more clearly describes the processes. For the ambiguous words "*acting afterwards upon*," which repeatedly occur both in text and claim, are substituted the more definite "*combining the products obtained with*." Also in Example 1 (see abstract of original specification, this Journal, 1886, 595) a correction is made in the weight of the sodium acetate employed in the preparation of the colouring matter from benzidine, naphthionic acid and  $\beta$ -naphthylamine sulphonic acid.—O. H.

## V.—TEXTILES: COTTON, WOOL, SILK, Etc.

### PATENT.

*A Process and Apparatus for freeing Wool from Grease.* L. Pinagel, Aachen, Germany. Eng. Pat. 20,936, December 31, 1889. 8d.

IN the valuation of wool the quantity of moisture and wool-grease it contains must be considered, and the apparatus patented is for estimating the latter. A vessel containing ether, light petroleum or carbon disulphide, is heated by a water-bath and the vapour of the solvent passed into the sample to be extracted and thence to a vertical condenser, whence it drops back on to the wool and is returned to the vessel at intervals. A gauge glass fixed to the portion of the apparatus containing the wool serves to show when the extraction is complete by the solvent ceasing to be coloured yellow.—B. B.

## VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

*The Composite Indigo-Indophenol Vat.* G. Galland. Bull. Ind. Soc. Rouen, 1889, 330–331.

THE substance of this report has already appeared in this Journal, 1889, 618; also 1889, 189, 282, and 539.—E. B.

*Firing of St. Denis Red.* H. Koechlin. Bull. Soc. Ind. Mulhouse, September 1889. Appendix, 38–39.

THE mordant used consists of—

500 cc. of magnesium acetate of 30° B.,  
500 cc. of water,  
250 cc. of aluminium acetate,  
or of—  
150 grms. of magnesium sulphate,  
50 grms. of alum,  
1 litre of water.

(The alumina may be replaced by chrome alum, which is also a good mordant for these colouring matters.)

The mordant is fixed by passing the material prepared with it through an alkaline solution of a metal, the following solution having the preference:—

1,000 cc. of water,  
50 grms. of zinc sulphate,  
100 grms. of caustic soda of 38° B.

Dyeing is performed at 80° with 2 to 3 grms. of St. Denis Red—

2,500 cc. of water,  
5 grms. of lime,  
500 grms. of salt.

These quantities suffice for 1 metre of calico. For subsequent use the bath needs replenishing only with colouring matter and lime. Alum added to the dye-bath in small quantity is very beneficial. The above mordant is suitable for fixing Ponceaux, Bordeaux, and other similar colours, such as the new blue of Meister, Lucius, and Brünig.—T. L. B.

*Note on the Bleaching of Cotton by Hydrogen Dioxide.*  
H. Koechlin. Bull. Ind. Soc. Rouen, 1889, 332.

*An Improvement in Bleaching.* H. Koechlin. Bull. Soc. Ind. Mulhouse, September, 1889. Appendix, 39—40.

A NEW factor is introduced, viz., magnesia, as well as the use of hydrogen peroxide in cotton bleaching. The author treats five pieces of calico of 100 metres each for six hours at the boil with—

100 litres of water,  
10 kilos. of fused caustic soda,  
30 kilos. of soap,  
5 kilos. of calcined magnesia, and  
50 litres of hydrogen peroxide.

The method is too costly for general use, but is exceedingly useful, when pressed for time. The bleaching is much better than that obtained by the ordinary processes, and the fibre suffers less.

The operation may be performed in Mather and Platt's apparatus. For 220 pieces of 100 metres each, or 2,200 kilos. of material are necessary—

3,000 litres of water,  
400 litres of hydrogen peroxide (12 vol. strength),  
20 kilos. of calcined magnesia, and  
50 kilos. of soap.

After six hours' boiling, wash, acid, wash and dry. It is advisable to give the goods a preliminary souring in sulphuric acid of 2°, and then to allow them to lie in pile 12 hours, and wash before bleaching.

Tussah silk is bleached a much better white when magnesia is added to the hydrogen peroxide, than when ammonia is added.

Better results are obtained in bleaching calico by Mather and Platt's method, when magnesia is added to the caustic soda. In every case it is necessary to exclude air; observing this precaution cotton may even be treated in a Mather and Platt's keir with a relatively strong solution of bleaching powder.—T. L. B.

*On a New Chromium Mordant for Cotton.* E. Kur.  
Chem. Zeit. 13, 1669.

THE new mordant is chromium bisulphite, which may be obtained in solution by passing sulphurous acid gas to excess into water holding in suspension chromium hydrate obtained by precipitating a cold solution of chrome alum with ammonia. About 24 hours' digestion are required for complete solution of the hydrate. On the large scale the mordant is best prepared by dissolving chrome alum in twice its weight of boiling water, and, after cooling to 15° C., adding a concentrated solution of sodium bisulphite until the colour of the mixture changes from bluish-green to yellowish-green, any excess of sodium bisulphite being advantageous. The cotton material is padded in this liquid, and then allowed to drain for two hours, after which it may be either dried or steamed, the latter being preferable, as the dried material is not easily wetted again. The mordanted goods may be rinsed to remove any loose particles of chromium hydrate without risk of removing the actual mordant. The material can be oiled before mordanting, an advantage possessed by this process over Koechlin's alkaline method of chroming, which removes the oil. In calico printing the mordant may be discharged

with oxalic or tartaric acid, or the chromium bisulphite mordant may be thickened with starch and printed on, with subsequent steaming for fixation. Bisulphite compounds of the alizarin colours can be mixed with the chromium bisulphite solution, printed at once on the calico, and fixed by steaming, as previously proposed by Mullerns. Alumina, iron, and other metallic oxides may be applied as mordants to vegetable tissues, in a similar way.—G. H. B.

*Steam Aniline Black.* A. Kortész. Chem. Zeit. 14, 179—180.

AN essential condition for the production of the "ungreenable steam aniline black" is the freedom of the colour-mixture from excess of mineral acid. Higher oxidation of the ordinary black is not, as is generally supposed, alone sufficient. This is shown by the following considerations:—A fent padded in a colour-mixture, intended for developing aniline black by ageing, dried, and steamed, is coloured a "greenable" black; an addition to such a mixture, keeping constant the proportion of aniline, &c. present, of a certain amount of ammonium ferrocyanide renders the black obtained ungreenable; if now, to the latter mixture, excess of hydrochloric acid be added, the black produced will be again greenable. The experiment can be varied in the following way:—A fent is padded with an ordinary steam-black mixture, dried, spotted here and there with dilute hydrochloric acid, suitably thickened, steamed, and chromed; the spots appear of a bronzy-black hue on the black ground, but by treatment with sodium bisulphite they are turned green, the rest of the cloth remaining black. Whether the ferrocyanide in the steam-colour neutralises the mineral acid in becoming itself converted into aniline ferrocyanide, or whether its chief use is in forming a basis of Prussian blue, is more of theoretical interest.

Regarding the behaviour under steaming of the ordinary black mixture intended for ageing, experimenting with slop-padded fents, it was found that the black developed regularly and properly by hanging in an ageing room, yielding a colour which greened considerably under the influence of sodium bisulphite, but which did not affect the strength of the fibre, whilst, when developed by a passage through a Mather and Platt's "steam aniline-ager," the black did not green so badly, but the fibre was slightly affected, and by steaming without pressure for half an hour the fibre was completely tendered, and the black, when tested, still showed, though in less degree, signs of greening.

When the various ingredients in the colour-mixture were increased in quantity by 10 per cent., the tendering action on the cotton was increased, the blacks were more intense, but were still greenable. With a colour-mixture weaker in the same proportion, the greening was more apparent, but the fibre naturally was less injured. The standard mixture, with which these experiments were made, consisted of 2,600 grms. of aniline, 2,900 grms. of hydrochloric acid of 19½° B., 1,500 grms. of sodium chlorate, further—300 grms. of aniline, 60 cc. of vanadium solution, and sufficient water to make 31.5 litres. It was next ascertained how basic such a colour-mixture could be made without the development of the black being prevented. The limit of basicity was found to be reached with 2,500 grms. of hydrochloric acid instead of 2,900 grms. in the preceding mixture. The blacks produced with the basic mixture were greenable, but were formed with less injury to the fibre. Acetic acid added to a basic mixture appeared to be without action, the blacks developing exactly the same as though it had been absent.

Very similar results were obtained when the sodium chlorate in the standard mixture was replaced by other chlorates. Ammonium chlorate, recommended by Rosenstiehl, and aniline chlorate, proposed by Paraf, as yielding ungreenable blacks, were found no better for this purpose than sodium chlorate. Chromium and aluminium chlorates yielded less greenable blacks, but the fibre was more injured. Of the ferrocyanides the best results were obtained with the ammonium salt. Almost as good results were obtained with pure aniline ferrocyanide, but the crude compound, mixed as it is with potassium chloride, gives inferior results.

The ammonium ferrocyanide used was prepared by mixing the solutions of 18 kilos. of potassium ferrocyanide in 32 litres of water and 9 kilos. of ammonium sulphate in 13 litres of water, and allowing the potassium sulphate formed to crystallise out; 1 litre of this solution contains an amount of ferrocyanide equivalent to 340 grms. of the potassium salt. The standard steam-colour was made by replacing the vanadium salt in the recipe given for the aged-black by 2,590 grms. of potassium ferrocyanide or a corresponding amount of another ferrocyanide. This proportion of ferrocyanide must not be exceeded or else a bronzy-black is produced. In certain cases, for example, with superior cloth, it is preferable to slightly reduce the amount; thus, for goods which are to be steamed 2,000 grms. may be used, and for goods which are merely passed through an aniline-ager 1,800 grms. of potassium ferrocyanide will suffice instead of the quantity mentioned above. With less than these quantities tendering of the fibre was invariably found to take place.

The quantity of hydrochloric acid in the steam-colour can be reduced from 2,900 grms. to 2,500 grms., the amount of ferrocyanide being at the same time reduced in the same proportion. Equivalent quantities of ferricyanides, used instead of ferrocyanides, gave fuller shades, but the same results were obtained by slightly increasing the amounts of chlorates in the ordinary mixtures.

With ammonium ferrocyanide chroming of the steamed black is unnecessary, this operation being best replaced, in this case, by a rinsing in slightly acidulated water.

It must be pointed out that all the experiments here described were made by sloop-padding the cloth, so that for machine-work slight modifications in the proportions of the several ingredients would have to be made.

Some further experiments were made to ascertain the effects of the admixture of the acetates of certain metals with the ordinary aged-colour. The acetates of copper, iron, and chromium, when not present in excess, were found to have a beneficial action: the black developed more quickly, was deeper in shade and less greenable, but the fibre was injured by steaming. Copper acetate gave the best result. When a metallic acetate is added to a solution of aniline hydrochloride, it may be assumed that aniline acetate and a metallic chloride are formed, and it is found that when excess of metallic acetate is added to a colour-mixture, so that theoretically no hydrochloride is left, the development of the black takes place imperfectly, if at all.—E. B.

#### *Indamine Blue.* E. Ullrich. Chem. Zeit. **14**, 375—376.

This dyestuff, which is sold in the form of a paste, is the hydrochloride of a base of the induline series. Like other members of the same group, it is used for dyeing shades of blue, resembling indigo, on the various fibres. On wool it may be applied either in a neutral or slightly acid bath. It gives very even shades, but the colours possess the disadvantage of rubbing off. Cotton can be dyed in several ways. The brightest shades are obtained by mordanting with tannic acid, fixing with a salt of antimony, dyeing in a bath of the dyestuff, and then immediately, without rinsing, working the dyed material in a bath containing either 15 per cent. of the weight of the cotton of potassium bichromate or 2.5 per cent. of potassium bichromate with 0.8 per cent. of sulphuric acid, at a temperature of 50°—60° for 10 minutes. For dyeing a full shade, the cotton must be mordanted with at least 10 per cent. of its weight of tannic acid.

Another plan consists in applying the dyestuff on unmordanted cotton in a bath containing sodium acetate; 10—20 per cent. of the dyestuff used remains in the dye-bath. The cotton is, without rinsing, chromed with 15 per cent. of its weight of bichromate of potash, or, better still, with a mixture of 2 per cent. of this salt and 3.5 per cent. of copper sulphate, these being the proportions required to form copper chromate. As a certain amount of metallic oxide fixes on the cotton, in combination with the insoluble colour formed, the goods can be topped with logwood, &c. without further mordanting. It is, however, preferable, when such colouring matters are to be subsequently

applied, to increase the quantity of copper chromate in the oxidising bath to, say, 6 per cent. of potassium bichromate and 10 per cent. of copper sulphate, and to boil the goods in such a solution for an hour.

For calico printing, the paste only requires mixing with a thickening containing a little acetic acid, and steaming, in order to give fairly fast colours, whose fastness is further increased by chroming. The acetate and tartrate of the colour-base give the best results. The chief reactions of an aqueous solution of Indamine blue R. are as follow: sodium hydrate precipitates the base of a dark red-violet colour; tannic acid completely precipitates; concentrated sulphuric acid dissolves the dyestuff with a blue colour; stannous chloride produces a slight precipitate; zinc dust and ammonia decolourise.—E. B.

#### PATENTS.

*Improvements in Scouring or Cleansing and Bleaching Textile Fabrics, Warps, and Yarns formed of Cotton or other Vegetable Fibres, and in Apparatus to be used for the Purpose.* E. Bentz, Manchester, and C. and A. Edmeston and E. Grether, Salford. Eng. Pat. 5590, April 2, 1889. 8d.

THE improvements relate to a method of scouring, preferably with a solution of caustic soda, fabrics, &c. composed of cotton or other vegetable fibres. A steam-tight vessel is fitted with a system of rollers and steam-pipes, and its exit and entrance are closed against the admission of air or escape of steam by a seal of the caustic liquor. The material to be scoured passes continuously through the machine, being alternately subjected to the action of the boiling caustic solution and steamed. For mechanical details, the drawings illustrating the specification must be consulted.—E. B.

*Improvements in the Preparation of Bleaching Agents.* D. G. FitzGerald, Brixton. Eng. Pat. 5995, April 8, 1889. 6d.

See under XI., page 397.

*Improvements in Method and Apparatus for Extracting the Liquid or Soluble Constituents from Disintegrated Vegetable Materials.* J. E. Searles, jun., Brooklyn, U.S.A. Eng. Pat. 19,002, November 26, 1889. 11d.

See under I., page 379.

*Improvements in and relating to Printing in Colours, and Apparatus therefor.* P. A. Godchaux, Paris, France. Eng. Pat. 522, January 11, 1890. 1s. 1d.

THIS patent relates to the printing in colours of paper. The printing-machine by which this is effected consists of a number of rotary printing appliances, adjustably mounted in a common frame, each appliance being provided with pressure rolls and inking devices. Arrangements are introduced for drying the ink printed upon the paper by one appliance before it passes to the next appliance. For details of the arrangements and mechanism, reference must be made to the drawings accompanying the original specification.—E. B.

*Apparatus for use in Treating and Dyeing Cotton Slivers, Roving, or Cops, and other Textile Materials.* R. Haddan, London. From J. Bertrand-Leplat, Turcoing, France. Eng. Pat. 703, January 14, 1890. 8d.

TEXTILE materials more or less advanced in manufacture, such as sliver, roving, cops, or bobbins, are placed in perforated compartments forming the periphery of a rotary



drum. Dye and other liquors are circulated by a pump through the material, which, excepting the rotary motion, is kept at rest and so preserved from derangement. Two sheets of drawings are given; it is impossible to properly explain the mechanical arrangements apart from these.

—E. B.

## VII.—ACIDS, ALKALIS, AND SALTS.

*Nitric Acid Chlorine Processes.* G. E. Davis. Chem. Trade J. 1890, 254—256.

In 1838 Watt and Tebbutt (Eng. Pat. 7531) obtained letters patent for "improvements in the manufacture of oxides of lead, and also of carbonate of lead." Chloride of lead is treated with nitric acid, when chlorine passes off, and is passed into an apparatus such as is used in making chlorides of lime, soda, &c. This is, the author believes, the first patent in which the use of nitric acid is described for the production of chlorine. On January 11th, 1838, these same gentlemen filed another specification (Eng. Pat. 7538) in which they say that chloride of sodium is put into a firebrick oven and a certain quantity of nitric or nitrous acid added, gentle heat applied, and the chlorine liberated is passed into apparatus commonly used in forming the chlorides.

It is quite certain, adds the author, that neither of these processes could meet the requirements of the chlorine manufacture of the present day.

Nine years afterwards (March 16th, 1847) the well-known Dunlop process (Eng. Pat. 11,624) was patented. Dunlop described his invention as follows:—"My invention consists of an improved method of producing chlorine fit for manufacturing purposes, by effecting mutual decompositions between the following substances: Muriate of soda, or any other muriate, nitrate of soda, or any other nitrate, muriatic acid, nitric acid. The assistance of sulphuric acid is in some cases required. I generally prefer employing it, in order to obtain for residuum, sulphate of soda, suitable for the manufacture of soda, &c. The above substances may be employed either altogether, or only two of them; as, for instance, nitrate with a muriate (in which case, of course, sulphuric acid must be employed), or a muriate with nitric acid, or a nitrate with muriatic acid, or muriatic and nitric acids may be used together. For the latter cases sulphuric acid is employed, according to the results wished to be obtained. On the application of heat, chlorine, an oxide of azote, and muriatic acid are evolved. These gases are then passed through a suitable condenser charged with sulphuric acid of a strength sufficient to absorb the oxide of azote. The chlorine and muriatic acid are then separated by means of water." In dealing with the foregoing nitrosulphuric acid, Dunlop says, "The process I usually follow is to introduce the nitrous sulphuric acid into a suitable vessel, when by the addition of water and heat, the oxide of azote is disengaged. This latter is caused to traverse a suitable condenser along with a sufficient quantity of air and steam or water, and is by this means all transformed into nitric acid, which can be used again in the manufacture of chlorine, and again recovered in the manner described, and so on."

The year following, November 21st, 1848, McDougall and Rawson (No. 12,333, November 21st) filed a provisional specification in which the collection and oxidation of nitrous fumes from any source is described. They pass the lower oxides of nitrogen alternately into water, or an aqueous solution of nitric acid, with air containing oxygen, and this patent was actually allowed in spite of the one granted the year before to Dunlop.

Swinfells and Nicholson in 1852 (Eng. Pat. 390), patented a process for the production of chlorine, by adding nitric acid to hydrochloric acid, and heating the same so as to produce chlorine and nitrochloric acid, and this being brought into contact with oxygen gas and with the vapour of water, nitric acid is produced, and the chlorine conveyed where required.

Higgin, in 1854 (Eng. Pat. 766), in describing his invention for a mode of separating metals from each other, states that one of the liquids he prefers to employ is a mixture of muriatic acid and nitrate of soda, which yields chlorine in solution; but it is not even hinted at as being a process for the industrial manufacture of chlorine gas.

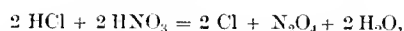
Gatty, in 1857 (Eng. Pat. 2230), proposed to employ nitrates in the manufacture of chlorine, but only as an oxidiser for the manganese, but he patented the application of the nitrous fumes for use in the manufacture of sulphuric acid, either "at once," or after they had passed through condensers.

In the year 1858 Messrs. Roberts and Dale patented a process (Eng. Pat. 2242) similar to that previously protected in 1847 by Dunlop. These inventors pursued absolutely the same methods as Dunlop for the production of their chlorine; but when the nitrogen oxides were driven off from the nitrosulphuric acid, instead of oxidising them for employment again, Messrs. Roberts and Dale conducted them into a solution of alkali.

In 1864 Messrs. Baggs and Simpson obtained provisional protection (Eng. Pat. 2313) for a process similar to that of Dunlop. It was subsequently abandoned. There is, however, one point of interest, in that the inventors, operating upon nitric acid with hydrochloric acid in a separate vessel, thought it necessary to specify that a gentle heat should be applied. Chloronitric and chloronitrous gas with free chlorine are evolved. Although Messrs. Baggs and Simpson abandoned the foregoing specification, yet it is doubtful whether it left their minds altogether, as Isham and Baggs, in 1866 (Eng. Pat. 3296), obtained letters patent for improvements in the manufacture and treatment of hydrochloric and nitric acids, which seem to bear somewhat upon the processes at which they had previously been working. These improvements were the treatment of liquid nitric or muriatic acid with sulphuric acid in order to secure the absorption of the water by the latter.

Up to this time no method essentially differing from the process of Dunlop (1847) seems to have been proposed; it is true that Baggs and Simpson preferred to bring the hydrochloric acid in contact with the nitric acid in a separate vessel, while Dunlop, as also Roberts and Dale, preferred to mix the nitrate with the salt, and to decompose both with sulphuric acid.

A period of 20 years now elapsed during which attention seems to have been riveted upon the problem of manganese recovery from the older chlorine process. On the 1st of October 1884 John Taylor (this Journal, 1885, 594), applied for provisional protection for a new process for the preparation of chlorine gas, and on the 12th of June 1885 he filed his complete specification. This specification bears a nearer resemblance to that of Baggs and Simpson than to that of Dunlop, but in the author's opinion is not identical with either. Taylor says:—"In carrying out my invention gaseous hydrochloric acid is passed through a tower or tube, or other suitable apparatus containing fragments of pumice stone, coke, or other suitable inert material, through which cold strong nitric acid is allowed to flow. The action of the nitric acid causes the formation of chlorine, nitrosyl chloride, water, and small quantities of nitrogen tri- and tetraoxides, about two-thirds of the chlorine of the hydrochloric acid being set free at once." Taylor does not give the details of the reaction, probably owing to the complications introduced by the presence of air, which always exists in very large quantity in furnace gases; but the main reaction is probably as follows:—

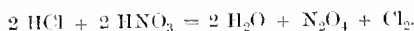


and the reason that two-thirds of the chlorine of the hydrochloric acid is liberated in the free state is probably because the oxygen of the furnace gases, in the presence of water, converts some of the  $\text{N}_2\text{O}_4$  into  $\text{HNO}_3$ , and this acts again before it has been removed from the sphere of action. The awkward features in this reaction are the by-products. The hydrochloric acid acts as a reducing agent to both tetra- and trioxide of nitrogen, and these, when formed, pass on with the chlorine and oxides of nitrogen. The excess of water formed during the reaction, and also that, I presume, coming from the vitriol used for decomposing the salt, is stated to

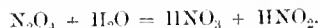
be periodically removed from the bottom of the nitric acid tower. Taylor then states that the mixed gases, chlorine, nitrosyl chloride, and nitrogen tri- and tetroxides are passed into strong sulphuric acid, which absorbs the nitrous fumes, permits the chlorine to pass on unchanged, and decomposes nitrosyl chloride with the formation of hydrochloric with which it is mixed, is sublimated a second time to nitric acid, and afterwards to sulphuric acid. The resulting gases are put through the processes a third time if necessary. The two alternations will produce a chlorine containing 90 per cent. of the hydrochloric acid as free chlorine, while three alternations produce about 96 per cent. In actual practice this chlorine is further washed with water to dissolve out the last traces of HCl.

In order to regenerate the nitric acid employed, Taylor places the nitrosulphuric acid in a suitable vessel, dilutes it by the gradual addition of water, while a rapid current of air is passed through. The nitrous vapours evolved are passed through water in a series of Woulf's bottles, whereby nitric acid of sufficient strength for use over again is obtained. (See also J. Taylor, Eng. Pat. 4201 of 1887; this Journal, 1888, 210.)

In the year 1887, William Donald obtained letters patent for an invention "to obtain chlorine from hydrochloric acid, and to recover the by-products formed during the operation" (Eng. Pat. 62; this Journal, 1887, 822; see Figs. 1 and 2). Hydrochloric acid gas is first dried by passing it through a suitable receptacle charged with sulphuric acid. The dried gas is then passed into a vessel charged with a mixture of strong nitric and sulphuric acids, where the hydrochloric acid gas undergoes decomposition. Donald represents the reaction that takes place as follows:—



The gaseous mixture resulting from the foregoing operation is now passed into a vessel containing cold dilute nitric acid, whereby nearly the whole of the oxides of nitrogen become fixed or absorbed, the reaction being expressed as follows:—



The chlorine, still containing small quantities of nitrogen compound, is now passed into a second absorbing vessel containing cold nitric acid, and finally through a scrubber containing sulphuric acid. This completes the elimination of the nitrogen compounds, and leaves the chlorine to pass on for use.

The nitric acid is regenerated by warming the nitrous liquors from the absorbers, and blowing a steady current of air through.

*Note on Concentrated Sulphuric Acid.* H. Osterberger and E. Capelle. Bull. Ind. Soc. Rouen, 1889, 307—313.

The strength of sulphuric acid of a density of 66° B. at 15° C. is given in the tables of Bineau, Bineau-Otto, and Kolb, as 100 per cent. From this it would appear that acid of 66° B. is a definite monohydrated compound. On the other hand, Winkler, Parkes, and Marignac state that the acid of such a density contains only 95 to 96 per cent. of  $\text{H}_2\text{SO}_4$ . It is, at all events, certain that the so-called acid of 66° B., as sold commercially, which tests 65.2° or 65.5° with a delicate instrument, contains some 6' or 7' more water than corresponds to the monohydrate. It may perhaps be best represented as a sesquihydrate, of the formula  $2 \text{H}_2\text{SO}_4 + \text{H}_2\text{O}$ , which consists of 91.59 per cent. of the monohydrate.

As is well known, sulphuric acid of 66° B. is obtained by concentrating acid of 60° B. in either platinum or glass stills; the latter can only be used in districts where they are obtainable at a low price, the former require a large initial outlay. Until recently, manufacturers have been content to produce with such apparatus two qualities of acid, namely, ordinary acid of 66° B. and a better quality, stated to be of the same strength, but actually marking 65.8° B. and containing 93.5 to 94 per cent. of  $\text{H}_2\text{SO}_4$ . Some manufacturers, by replacing the leaden evaporating pans by pans made of platinum and by making some other

improvements, have succeeded in producing an acid of a strength of 97 to 97.5 per cent. as determined by analysis. But the manufacture is hampered with difficulties, the wear and tear of the plant is considerable; for example, 5 to 7 grms. of platinum are lost per ton of the 97 per cent. acid made as against 1 or 2 grms. per ton in the concentration of the ordinary acid. Recently, however, Lunge has patented another process for the manufacture of the monohydrated acid in which the drawbacks connected with the older manufacturing process are not met with. This process depends upon an observation made by Marignac, namely, that on freezing ordinary concentrated sulphuric acid, the monohydrated acid separates in crystals. As actually carried out at Petit-Quevilly, at the establishments of the Malétra Company, who have obtained the monopoly for France, the process consists in first concentrating the acid as far as possible in the ordinary way. An acid containing from 93 to 98 per cent. of  $\text{H}_2\text{SO}_4$  is thus obtained. It is, however, preferable to operate upon acid containing, say, 97 per cent. of  $\text{H}_2\text{SO}_4$ , as the mother-liquors then contain still 94 or 95 per cent. of acid, and may be sold as such or be concentrated again. The concentrated acid is collected in a reservoir fixed above an iron vessel used for filling the crystallising cells. A pipe, provided with a tap, connects the reservoir with the filling-machine. The filling-machine is closed by an air tight cover, and divided by vertical partitions into the same number of compartments as there are crystallising cells in a set. Each compartment is connected with a corresponding cell by a pipe, whose end almost touches the sloping bottom of the compartment, and which passes through the cover vertically upwards to a height slightly above the level of the acid in the reservoir, then bends round and finally ends above the crystallising cell. When the cells are not being filled, the exposed end of the pipe is closed by a dripping vessel. Compressed air is forced into the filling-vessel in order to drive the acid into the crystallising cells. The capacity of the filling-machine is such that a set of the crystallising cells is exactly filled each time the machine is emptied; for instance, if there be 13 cells in a set, each capable of holding 12.5 kilos. of acid, the machine will contain exactly 162.5 kilos. The cells are placed in a solution of calcium chloride, cooled by a refrigerating machine to -20° C.

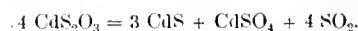
The blocks of acid so obtained are next loosened from the sides of the cells by dipping the latter for an instant in hot water. The blocks are collected in a trough, broken up by means of an archimedean screw, drained in a hydro-extractor, melted, and packed for transport, 100 per cent.  $\text{H}_2\text{SO}_4$  being so obtained.—E. B.

*On Thiosulphates.* A. Foek and K. Klüss. Ber. 23, 534—541.

In this paper the crystallographical measurements of the salts described are fully given.

When prepared by the authors, manganese thiosulphate did not crystallise (cf. Vortmann and Padberg, Ber. 22, 2641), the oily, rose-coloured liquid decomposing, both in the mother-liquor and *in vacuo* over sulphuric acid, into manganese sulphite and sulphur.

Cadmium thiosulphate,  $\text{CdS}_2\text{O}_3 \cdot 2 \text{H}_2\text{O}$  (*loc. cit.*) forms small, colourless, concentrically grouped, monosymmetric crystals, which do not lose water *in vacuo* over  $\text{H}_2\text{SO}_4$ , but gradually become yellow when kept from formation of cadmium sulphide. When heated in a closed tube it decomposes according to the equation—



*Potassium ammonium thiosulphate*,  $\text{K}(\text{NH}_4)\text{S}_2\text{O}_3$ , is obtained by fractionally crystallising a mixture of equal mols. of the two components from water; it forms small lustrous monosymmetric crystals.

*Potassium ethyl thiosulphate*,  $\text{SO}_2 \cdot \text{SC}_2\text{H}_5 \cdot \text{OK}$ , is formed when the preceding salt and ethyl bromide (equal mols.) are heated together in a reflux apparatus; it crystallises in slender, colourless, monosymmetric needles.

*Potassium magnesium thiosulphate*,  $\text{K}_2\text{S}_2\text{O}_3 \cdot \text{MgS}_2\text{O}_3 \cdot 6 \text{H}_2\text{O}$  (cf. Rammelsberg, Chem. Abhandl. 1888), separates from a

solution of its components in water when concentrated by a gentle heat, in large transparent monosymmetric crystals; it does not deliquesce in air.

Ammonium magnesium thiosulphate—



(Kessler, Pogg. Ann. **74**, 283) forms long monosymmetric prisms stable in air and *in vacuo* over sulphuric acid.

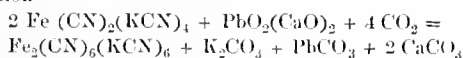
—A. G. B.

*The Valuation of Fuming Sulphuric Acid.* B. Setlik. Chem. Zeit. **13**, 1670.

See under XXIII., page 417.

*A New Process for Manufacturing Potassium Ferrieyanide.* G. Kassner. Chem. Zeit. **13**, 1701—1702.

Of the several methods that are available for converting ferrocyanide into ferrieyanide on the large scale, that involving the use of chlorine is usually employed on account of its cheapness, but there are objections to its use. Especial precautions have to be taken to avoid the introduction of any excess of chlorine over that required to complete the reaction, otherwise the ferrieyanide produced will suffer decrease both in quantity and quality. Another drawback to the use of chlorine is the occurrence of spontaneous explosions in the vessels containing the materials during the oxidation, due, as is commonly explained, to the formation of chloride of nitrogen. These inconveniences could be avoided by the use of peroxide of lead, but the cheapest means of producing that substance has been still too expensive hitherto for competing with chlorine as the oxidising agent. But a recent invention of the author's has made the use of lead peroxide practicable in the form of calcium plumbate, which is easily obtained by roasting oxide or carbonate of lead with calcium carbonate at a low red heat. In the conversion of potassium ferrocyanide into ferrieyanide by means of lead peroxide a quantity of potassium hydrate is set free which must be neutralised in some way before the reaction can go on to completion, and carbonic acid suffices for the neutralisation. The author's process consists in adding calcium plumbate to a solution of potassium ferrocyanide and passing a stream of carbonic acid gas when the reaction proceeds according to the equation—



The carbonates of lead and calcium separate as an insoluble precipitate which can be filtered off and regenerated by a simple roasting, whilst the solution contains potassium ferrieyanide in a pure form and potassium carbonate as a valuable by-product. The author considers that the intrinsic merits of potassium ferrieyanide as an oxidising agent in alkaline solution will occasion an extensive demand for it when its production has become sufficiently cheap. (See this Journal, 1890, 187.)—G. H. B.

#### PATENTS.

*Improvements in the Production of Chrome Preparations.* H. A. Seegall, Berlin, Germany. Eng. Pat. 4602, March 16, 1889. 6d.

THE chrome mineral is heated with an excess of an alkaline bisulphate in closed vessels furnished with mechanical stirrers, the sulphurous and sulphuric anhydrides evolved being passed first through condensers and then through milk of lime. It has been found that when some substance, such as barium sulphate, is present in the mixture, the amount of the acid vapours is very much reduced or practically disappears. The patentee explains this by supposing that when a mixture of the ore and bisulphate alone is taken the ore is apt to sink to the bottom and to remain unacted upon, whilst the bisulphate is decomposed at the temperature employed, about 600° C., with the evolution of sulphuric anhydride. When the barium sulphate is added the mass is much more pasty than before, and as the powdered ore is no longer able to sink to the bottom the sulphuric anhydride evolved from the bisulphate comes into more intimate contact with the ore, and is retained. The

chromium sulphate formed is dissolved out with water, and the chromium can be precipitated by the addition of caustic alkalis, &c. The precipitate so thrown down can be readily dissolved in various acids forming the required salts.

—S. G. R.

*A Method of Treating Waste or Spent Sulphuric Acid Pickle produced in the Manufacture of Tin, Terne, and Black Plate.* S. Daw, Llanelli, Eng. Pat. 4701, March 18, 1889. 4d.

See under X., page 395.

*Improvements in or relating to obtaining Chlorine.* R. Dormer, Garston. Eng. Pat. 5225, March 26, 1889. 6d.

IN preparing chlorine by treating manganese dioxide with hydrochloric acid, half the available amount of chlorine is lost as manganese chloride. In order to avoid this loss the patentee adds sulphuric acid to the mixture of the peroxide and hydrochloric acid, whereby the whole of the chlorine is obtained in the free state, the manganese being converted into the sulphate. As this salt is not applicable for the regeneration of the manganese, there is added to a solution of it chloride of calcium liquor, when calcium sulphate is precipitated and manganese chloride formed. This can be treated in the usual way by the Weldon process. It is pointed out that in order to recover the whole of the chlorine the only extra expense is that of the sulphuric acid used.

—S. G. R.

*Process of Manufacture of Potassic and Sodid Hydrates and of By-products.* G. A. H. Gabet, Paris, France. Eng. Pat. 5341, March 28, 1889. 6d.

A dilute solution, about 15 per cent., of the acid phosphate of calcium, is treated with sulphate of sodium or potassium, the calcium being thus precipitated as sulphate. To the solution milk of lime is added with the separation of calcium phosphate, and the formation of the hydrates of potassium and sodium. The double salts of potassium, sodium, and magnesium may also be used with advantage. The acid phosphate of magnesium being soluble it can, when required, be either precipitated mixed with calcium phosphate by the addition of milk of lime, or it may be obtained pure by adding potash solution, and then, after its precipitation, the remaining phosphoric acid in combination with the potash may be precipitated as before by adding milk of lime. Very pure caustic soda may be prepared by precipitating a part of the phosphate with milk of lime, and then crystallising the disodium phosphate before its final treatment with milk of lime.—S. G. R.

*Improvements relating to the Treatment and Utilisation for Agricultural and other Purposes of Bisulphate of Soda or "Nitre Cake."* H. J. Kirkman, Swansea. Eng. Pat. 5703, April 3, 1889. 4d.

IT is proposed to use the acid sulphate of soda, produced in the manufacture of sulphuric and nitric acids, as an absorbent for the ammonia from gas liquor instead of the sulphuric acid customarily employed. Or the salt may be dissolved in the gas liquor and the resulting solution crystallised to obtain sulphate of soda and sulphate of ammonia.—S. G. R.

*A Process for Producing Cupr-Ammonia.* C. F. Hime, London, and J. H. Noad, East Ham. Eng. Pat. 7716, May 8, 1889. 4d.

SCRAP copper is placed in a vessel containing ammonia solution. The copper is in connexion with the one pole of a battery, whilst attached to the other are electrodes placed in porous vessels which are plunged into the alkaline liquid. On passing the electric current the copper dissolves in the ammonia forming "cupr-ammonia."—S. G. R.

*Improvements relating to the Production of White Lead.* H. H. Lake, London. From J. B. Tibbitts, Hoosac, U.S.A. Eng. Pat. 18,076, November 12, 1889. 6d.

See under XIII., page 398.

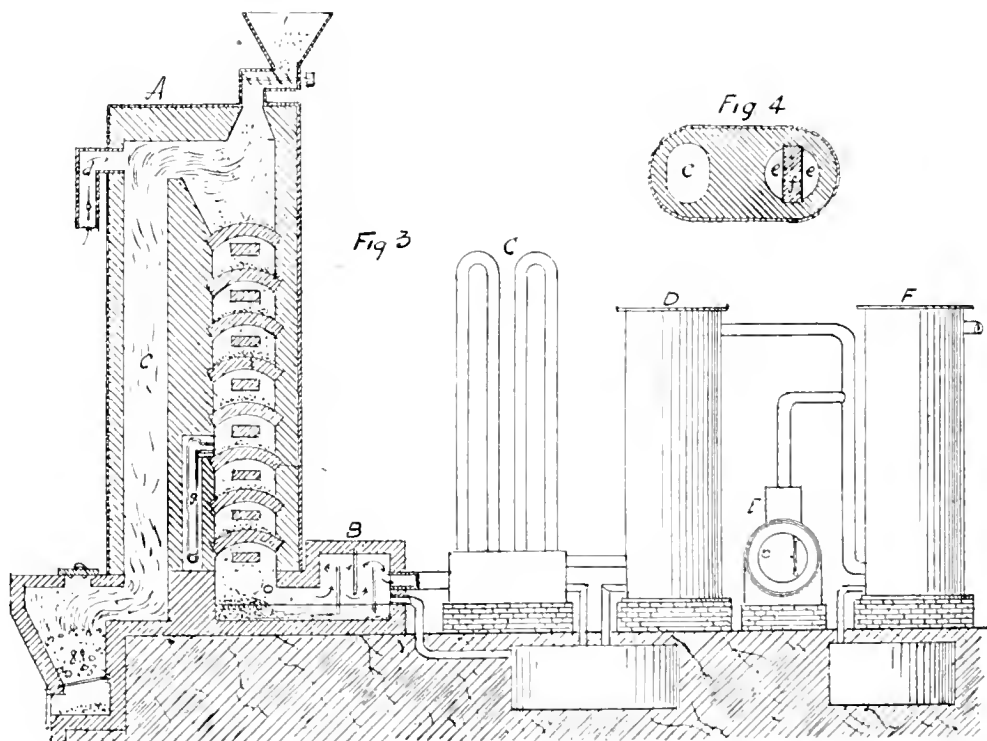
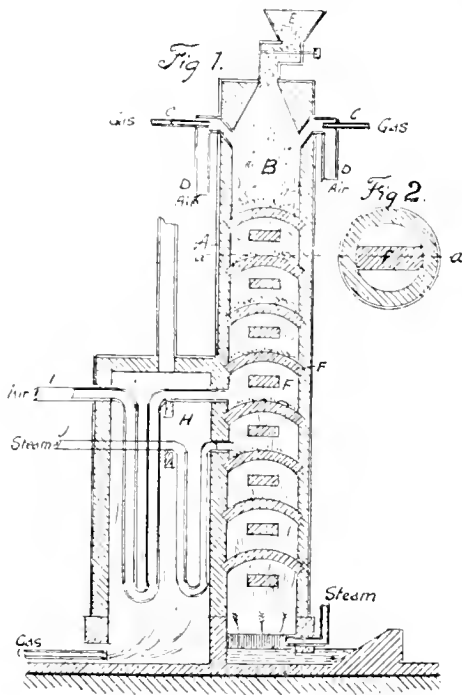
*A Method of and Apparatus for obtaining Ammonia from Atmospheric Nitrogen and the Hydrogen of Decomposed Steam.* J. C. Fell, London. From the American Ammonia Manufacturing Co., Alexandria, U.S.A. Eng. Pat. 20,722, December 24, 1889. *8d.*

By strongly heating together an alkali and carbon in the presence of nitrogen an alkaline cyanide is produced which

when brought into contact with air is oxidised with the formation of a cyanate. On treatment with steam the cyanate is decomposed with the production of ammonia, oxides of carbon, hydrogen, and various alkaline compounds. From this mixture ammonia is readily separated by passage through suitable scrubbers.

The operations are carried out in two different forms of apparatus. In one case (Figs. 1 and 2), the ammonia-generating furnace A is heated by the introduction of gas and air by the pipes C and D into the combustion chamber B. Through the hopper E is fed in the mixture of carbon and alkali which becomes intensely heated as it falls upon the baffle plates F in the furnace, and in combination with the nitrogen present forms cyanides. H is a superheater by means of which the oxidising current of air entering at I and the decomposing current of steam J are raised to a high temperature, their action being as already mentioned above. The air pipe is placed above the steam pipe so that time may be allowed for complete oxidation.

In the second form of apparatus, Fig. 3, A is the furnace in which the fuel is burned, furnished with an air inlet d, and in connexion with the ammonia-generating vessel e there is no special air supply tube, but steam is injected through the superheater g. B serves as a separator for any solid matter escaping from A, whilst in C the gases are cooled before the separation of the ammonia. D is a scrubber to remove the alkaline salts present with the ammonia. The condensing liquors used retain a little ammonia which is given off when boiled in the vessel E, whilst F is the scrubber or series of scrubbers in which the ammonia is intercepted.—S. G. R.



## VIII.—GLASS, POTTERY, AND EARTHENWARE.

*Enamelled Lava as a New Ceramic Material.* Gillet. Deut. Töpfl. u. Ziegl. Zeit. **20**, 783.

ENAMEL-PAINTING on lava, discovered by Mostelique, has lately been extended by the author to building ornaments, vessels for chemical works, and wall painting. The natural product, which was formerly used in its crude state, is now powdered and mixed with some binding material so as to form a malleable paste which can be modelled and burnt without cracking, as when dried it shrinks only slightly. It is very hard. The enamel is applied as usual with ceramic objects.—N. H. J. M.

*A New Coral Red.* Deut. Töpfl. u. Ziegl. Zeit. **20**, 796.

THIS is a colour for porcelain or stone-ware prepared as follows:—Lead chromate (20·40 parts), minium (67·83 parts) and quartz (12·27 parts) are melted together until clear. The dark red substance is powdered and used with the hard colourless flux I, or with the soft flux II.

Flux II. Minium (80 parts), quartz (20 parts); flux I. minium (80 parts), quartz (100 parts), crystallised boric acid (20 parts).

The fluxes are melted clear, powdered, and rubbed on the pallet. A good coral-red is obtained by rubbing the colour (75 parts) and one of the fluxes (25 parts) on the pallet, and laying it on to the object with turpentine and thick oil.—N. H. J. M.

*The Tarnishing of Glass during Manufacture.* Moritz. Sprechsaal, **22**, 943.

DURING the flattening-out of window-glass on gas-furnaces the surface of the glass is corroded or tarnished by the action of the flame; and it is usual to remove this tarnish by bathing the separate sheets in some fluid, such as dilute hydrofluoric acid. This operation is a very tedious one, and according to the author may be avoided by supplying the gas-furnace with cold air only, instead of heated air, as is usually done. By this means a more or less sooty flame is produced which does not injuriously affect the glass.

—H. T. P.

### PATENTS.

*Improvements in the Manufacture of Blocks of Terra-cotta and like Materials, and in Machinery for this purpose.*

H. L. Doulton and S. H. Leech, Lambeth. Eng. Pat. 5205, March 26, 1889. *8d.*

OWING to the great difficulty of drying large blocks of terra-cotta it is necessary to make them hollow, and hitherto they have had the form of hollow casings or shells which have to be filled when used for building purposes in order to possess the necessary strength and solidity. According to the present invention the blocks are formed with a number of internal cells or cavities similar to the well-known perforated bricks, but that the cavities are only open at one end, the closed end forming part of the face of the block. The cellular structure allows of complete drying of the clay whilst the block remains practically solid and requires no filling. Such blocks can be made in a variety of ways, and the machine shown in the drawings serves to illustrate the principle involved which especially provides for the admission of air into each cavity at the moment of formation, so that each cavity is prevented from collapsing by the enclosed plug of air. The machines may be constructed to deliver the clay in a continuous block to be cut to required lengths and faced by hand, or the block may take its final form in the machine.—G. H. B.

*The Use of Sugar in the Manufacture of Fire-clay Goods.* G. Palin, Kildgrove. Eng. Pat. 7387, May 3, 1889. *4d.*

ABOUT 3 per cent. of unrefined or raw sugar from beet root or sugar cane is added to the gannister sand or fire-clay used in the manufacture of fire-bricks, furnace linings, seggars, tiles, &c. The result is said to be that the materials, after baking, possess particularly good fire-resisting qualities.

—E. G. C.

*Improved Process and Furnace for the Preparation of Crude Glass, according to Leuffgen's Method, by the combined use of Water-Gas, Coal-Gas, Wood-Gas, Oil-Gas, or Natural Gases, and Generator-Gas.* J. Quaglio, Berlin, Germany. Eng. Pat. 7659, May 7, 1889. *8d.*

THE arrangement consists of a tank-furnace heated by gaseous fuel, and on the roof of the furnace are melting pots with a lateral opening at the bottom, the contents of which are slowly but continuously melted by the action of a blow-pipe flame impinging on the edge of the materials. The fused crude glass, called calcin, runs down a gutter to a shallow receptacle, from which it overflows into the main tank to be drawn off as required. The refining of the crude glass is accomplished by the action of the flame playing on the stream of glass on its way through the furnace.

—G. H. B.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

*Valuation of Roofing Slates.* H. Brunner. Schweiz. Wochenschr. f. Pharm. 1889.

THE specific gravity and the colour are no criteria of the value of a slate: the true markings or lines which are most easily seen by holding the slate in a slanting position close to the eye, should run parallel to the long axis, otherwise the slate will break easily. A good slate must be hard, should not be easily scratched with the finger nail, should not break into laminae, and should give out a clear note when struck.

The presence of calcium carbonate, pyrites, and marcasite can be easily detected by examining a thin fragment with a polarisation microscope.

For examining the absorptive qualities, a piece of slate 12 centimetres long and 6 centimetres broad is placed in a beaker containing water 1 centimetre in depth, the beaker is covered with a glass plate and left for 24 hours; at the end of this time, if the slate is a good one, only a few millimetres above the level of the water will be damp.

In examining slates chemically the calcium carbonate (magnesium carbonate) and the sulphide of iron are estimated in the usual way, and the weathering qualities, which are of the greatest importance, are examined by R. Fresenius' method (Zeits. Anal. Chem. **7**, 72).

The action of heat and cold should also be tried: for this purpose the slate is saturated with water and then placed for 24 hours in a freezing mixture. Another sample is heated at 250°—300° for five to six hours and then placed in water.

For an approximate determination of the commercial applicability of a slate it is treated with hydrochloric acid; a brisk effervescence indicates a bad, calcareous slate. Another portion is heated in a glass tube; a yellow sublimate of sulphur and evolution of sulphur dioxide indicates a bad slate.—F. S. K.

*The Examination of Roofing Slates.* F. Reverdin and C. de la Harpe. *Chem. Zeit.* **14**, 61—65, 94—95, and 126—127.

H. BRUNNER (preceding abstract) has described methods for judging the quality of roofing slates, and the authors criticise his conclusions and advance their own. Of the physical properties of a slate, such as its specific gravity, tenacity, elasticity and porosity, Brunner considers the last of greatest importance, and determines it thus: A test-piece is placed in a beaker and water poured in to the depth of 1 cm. After 24 hours the height to which the water has risen on the test-piece on account of its capillarity is read; the better kinds of slate give a smaller rise than those inferior to them. The authors doubt whether this is of much value on Brunner's own figures, as a sample reported "bad" by him gave a rise of 3 mm., and one stated to be "very good" a rise of 4.5 mm.

The chemical constitution and behaviour of slates are however the data to which Brunner attaches most weight, and the chief tests he uses are the determination of the quantity of calcium carbonate and pyrites in the sample and its power of resisting the action of sulphurous acid. The authors dissent from this view on the several grounds that slates of known excellence, such as that from Morzine, may contain as much as 49 per cent. of calcium carbonate, while other specimens (*e.g.*, from Servoz) free from that substance may be of poor quality; that the same observation applies in the case of pyrites, and that further pyrites, when present, appears to suffer no change on exposure of a slate to weather; that the use of sulphurous acid to imitate the disintegrating effect of exposure though convenient lacks precision, and is only of limited usefulness.

The methods and conclusions adopted by the authors are as follows:—The agencies by which the deterioration of slates is effected are—

(1.) Chemical action of the atmosphere (weathering).

This is comparatively of small importance.

(2.) Chemical action of gases arising from the wood work beneath the slates.

(3.) Physical action of changes of temperature favoured by the dissimilar conductivity of heat of slate in the direction of and at right angles to its plane of cleavage.

This is considered the most weighty of the three.

The samples examined included slates of various origins, *e.g.*, Sembrancher, Salvan (grey), Salvan (black), Outre-Rhône, Mex, Morzine, Servin, Angers, Ardennes, both old and new specimens; Orny, Sion, Saxon, Isérable, Pfeffers, La Chambre, Le Plet, new only.

The methods used were:—

(1.) *Determination of Porosity after the Action of Acids.*

—Test-pieces 6 cm.  $\times$  2 cm.  $\times$  ? were treated with 10 per cent. acetic acid in the cold, the flask containing them being rendered vacuums from time to time, until the evolution of carbon dioxide ceased—a result sometimes taking weeks to attain. The extracted pieces were then washed, dried, weighed, and immersed in diphenylamine (m.p. 54° C. b.p. 310° C.) contained in a thick-walled tube 12 cm.  $\times$  3.5 cm. The tube was exhausted, heated for two hours in an oil-bath at 170° C., the air pressure restored, and the heating continued for 4—5 hours at 150° C., after which the test-pieces were removed, the diphenylamine clinging to their surface wiped off (the wiping being supplemented by rapid washing with ether), and the increase in weight taken. Some figures thus obtained are: Salvan, 0.05; Outre-Rhône, 0.08; Sembrancher, 0.15; Orny, 0.22; Pfeffers, 5.8; La Chambre, 9.2 parts per cent. respectively.

(2.) *Determination of Soluble Ferrous Iron.*—The finely powdered samples were digested with acids, such as acetic and hydrochloric, and the ferrous salt extracted determined colorimetrically. Old and new specimens of the same slate gave similar quantities of iron in the ferrous state, showing that oxidation by exposure to weather was trifling.

(3.) *Determination of the Porosity of Old and New Slates.*—The method used was similar to that described

under (1) save that the test-pieces were not extracted with acetic acid. Some of the results are given below:—

Name.	New.	Old.	
	Increase in Weight.	Age.	Increase in Weight.
	Per Cent.		Per Cent.
Salvan (grey).....	0.03	45	0.19
Outre-Rhône.....	0.02	10	0.06
Ardennes.....	0.03	30	0.15
Sembrancher.....	0.04	10	0.28
Angers.....	0.06	30	1.11
Morzine.....	0.02	30	1.18
Mex.....	0.04	30	0.31
Salvan (black).....	0.06	..	..
Orny.....	0.09	..	..
Sion.....	1.15	..	..
Servin.....	..	20—25	1.16
Unknown (much disintegrated).	..	..	2.58

From this it appears that new slates are considerably less porous than old, and that the rate of increase in porosity is much higher in some specimens than in others.

(4.) *Resistance to the Effect of Rapid Alterations of Temperature.*—Test-pieces 6 cm.  $\times$  2 cm.  $\times$  ? were heated in a wrought-iron tube for half an hour to 300° C., and the tube then suddenly cooled by a stream of water for half an hour; the process was repeated 24 times, after which the test-pieces were impregnated with diphenylamine as described above, with the following results:—

Name.	Increase in Weight after Treatment.	Increase in Weight of Untreated Specimens.	Difference.
Outre-Rhône.....	0.08	0.02	0.06
Salvan (black).....	0.13	0.06	0.07
Sembrancher.....	0.12	0.04	0.08
Mex.....	0.15	0.04	0.11
Orny.....	0.27	0.09	0.18
Morzine.....	0.24	0.02	0.22
La Chambre.....	0.74	0.60	0.14
Sion.....	2.35	1.15	1.20

In the light of these investigations, the authors conclude:—That the valuation of a slate can be best arrived at by determining its porosity when new and after having been subjected to alterations of temperature. A high degree of porosity when new, or a considerable increase after repeated heating and cooling, condemns a slate. Though not laid down as absolute limits, the authors consider that the porosity of a good slate when new should be below 0.1 per cent., and after treatment less than 0.2 per cent.—B. B.

#### PATENTS.

*An Improved Non-corrosive Material, suitable as a Cement Packing or Covering to Resist Acids.* B. Rhodes, London, Eng. Pat. 5363, March 28, 1889. 4d.

The composition is made by mixing specified quantities of mica, talc, "flower of sulphur," and asbestos fibre with india-rubber, asphaltum, and "albo-carbon" dissolved in

mineral spirit, the whole forming a dough which may be applied to the purpose required. In some cases the solvent may be omitted and vulcanisation effected by heating.

—G. H. B.

*Improved Combination of Ingredients for Use as Mortar, Plaster, and the like for Building Purposes.* T. K. Ferguson, Forest Hill. Eng. Pat. 5412, March 29, 1889. 4d.

SPECIFIED proportions of clay, sand, plaster of Paris, and quicklime are mixed together along with water and a small quantity of vitriol to form the mortar.—G. H. B.

*Improvements in the Manufacture and Composition of Refractory or Fire-resisting Cement or Cements.* W. S. Lea and H. M. Lynam, Hanley. Eng. Pat. 5919, April 6, 1889. 6d.

THE materials composing the two cements are given as follows:—

	No. 1.	No. 2.
	Parts.	Per Cent.
Disintegrated granite.....	20	50—65
Blast furnace or basic steel slag.....	50	5—15
Steatite.....	32	15
Silica, siliceous ochre.....	32	5
Kaolin.....	60	..
Phosphate of lime.....	40	5
Loam.....	24	..
Argil.....	32	..
Sodium chloride.....	8	2
Calcium carbonate.....	8	2
Caustic potash.....	4	1

The powdered mixture is made into a plaster with water to be used as a lining for furnaces. It should be applied in several coats to a thickness of half an inch. It withstands rapid changes of temperature without disintegration. The disintegrated granite is prepared by exposing the intensely heated rock to the action of gaseous nitric and hydrochloric acid, with subsequent reduction to powder.—G. H. B.

*Improvements in Machines for the Manufacture of Bricks and Briquettes.* N. Procter, A. Middleton, C. E. Fraser, and H. M. Carter, Leeds. Eng. Pat. 8845, May 28, 1889. 6d.

THE invention relates to the automatic measurement of the materials entering any brick-making machine. For this purpose the charging hopper is placed above a revolving drum having a diametrical channel for the passage of the materials, its function resembling that of the plug in an ordinary tap. But the fall of the materials is checked by a metal piston sliding freely within the channel. At each revolution a portion of material enclosed in the channel is carried round, and when the piston is uppermost it presses the charge downwards into the machine, giving room for a fresh charge from the hopper. The quantity of material to be delivered may be adjusted by altering the size of piston.

—G. H. B.

*An Improved Composition or Plaster for the Treatment of Walls and the like.* J. B. King, West Brighton, Rd., U.S.A. Eng. Pat. 10,997, July 9, 1889. 4d.

THIS compound consists essentially of a restraining material (preferably glue), a ground stone or stone-like material (marble, chalk, plaster, slaked lime, &c.), and a fibrous material (preferably asbestos).—E. G. C.

## X.—METALLURGY.

*Chemical Society of Hamburg.* Chem. Zeit. 13, 1706.

A BRIEF notice of a report presented by Dr. Gilbert of his journey to the Rio Tinto mines. After some remarks of historical interest there followed a description of the disposition of the strata and the methods of working the ores. In the year 1888 one and a half million tons of ore were worked yielding 20,000 tons of copper. The ores contain on the average 49 per cent. of S., 43 per cent. of Fe, 3 per cent. of Cu, 1 per cent. of PbO, "6 per cent. of As," and 2 per cent. of SiO<sub>2</sub>, with small quantities of gold, silver, bismuth, tin, antimony, nickel, selenium, and thallium. The burnt ores from the vitriol works are worked up for copper, gold, silver, and recently also for bismuth, the residual iron oxide going to ironworks.—G. H. B.

## PATENTS.

*Improvements in the Manufacture of Metallic Alloys.* G. A. Dick, London. Eng. Pat. 2484, May 25, 1882. Amended December 4, 1889. 6d.

THE patentee now restricts his claims to the manufacture of alloys "by introducing into copper zinc and copper zinc and tin alloys definite quantities of iron by previously causing the zinc uniformly to dissolve or absorb a known proportion of iron." The three other claims in the original specification relating to the reduction of oxides by means of "manganese copper," and to the production of alloys containing, in addition to the above metals manganese or lead, being withdrawn.—O. H.

*A New or Improved Method of Coating Plates and Sheets of Iron with Copper or Brass, and Apparatus to be Employed in effecting the said Coating.* W. E. Everitt, Birmingham. Eng. Pat. 4335, March 12, 1889. 6d.

THE sheet of iron is cleaned by "pickling" or otherwise, and an even layer consisting of a mixture of about equal parts of granulated solder and powdered borax sieved over the surface. Upon this the sheet of copper or brass is laid and the plates placed in a heating furnace when the solder and borax are melted. The plates are then passed between rolls.—J. B. C.

*A Method of Treating Waste or Spent Sulphuric Acid Pickle produced in the Manufacture of Tin, Terne, and Black Plate.* S. Daw, Llanelly. Eng. Pat. 4701, March 18, 1889. 4d.

IN order to clean the iron plates they are immersed in dilute sulphuric acid, a solution of sulphate of iron being thus produced. To this is added magnesium oxide and the whole warmed, when iron oxide is precipitated with the formation of magnesium sulphate. The liquors may be then concentrated and crystallised. Instead of magnesium oxide, dolomite in the proper proportion may be used.—S. G. R.

*Improvements in the Manufacture of Artificial Cryolite.* M. Netto, Newcastle-on-Tyne. Eng. Pat. 6668, April 18, 1889. 4d.

THE residual slags left after the extraction of the aluminium consist in the main of sodium fluoride and chloride. To a strong solution of these there is added a hot concentrated solution of hydrated aluminium sulphate, when aluminium fluoride is precipitated. This is removed and mixed with more of the slag, either in a solid condition or else in the form of a strong solution. On fusion an artificial cryolite is obtained.—S. G. R.



*Improvements in the Manufacture of Steel.* W. F. Jackson, Litton, and W. Gallraith, Stoke-on-Trent. Eng. Pat. 7047, April 27, 1889. 8d.

THE object of the invention is to regulate the quality of the steel manufactured from phosphoric irons by the "basic" Bessemer, Siemens, or open-hearth process. The molten charge when sufficiently decarbonised and dephosphorised is teemed into the ladle either with or without a previous partial addition of hematite iron, spiegeleisen, ferromanganese or other alloy. The charge is run from this ladle into a second ladle, simultaneously with a charge of molten spiegeleisen, ferromanganese, or other alloy, and a suitable quantity of molten silicious slag, and teemed into moulds from the second ladle.—J. B. C.

*Improvements in Means for effecting the Separation of Particles of different Specific Gravities, more especially intended for the Separation of Gold and other Metals from Crushed or Divided Ores.* M. Crawford, Liverpool. Eng. Pat. 8789, May 27, 1889. 8d.

THE invention consists of an apparatus or machine, which holds a number of troughs arranged in tiers. The bottom of each trough is inclined, the inclination being opposed to the flow of a stream of water, which is allowed to flow over the bottom. At the same time the troughs are attached to spring hangers and are moved by an eccentric, which imparts a sideways movement. The troughs can be readily removed for cleaning.—J. B. C.

*Improvements in Machinery or Apparatus for Extracting Precious Metals from their Ores.* D. Dumes and T. K. Rose, London. Eng. Pat. 19,622, December 6, 1889. 8d.

THE finely divided ore is injected by air into a muffle where it meets with a current of water-gas, and the ore thereby roasted. From the muffle the roasted ore is fed into a chlorinating apparatus consisting of a drum or cylinder lined with lead and revolving on hollow trunnions. Through the hollow trunnions the chlorine gas is driven out by compressed air, when the extraction is complete. The contents of the drum are discharged into a conical leaching vat, the bottom or filter bed of which is movable, and by compressed air the liquid is forced through the filter bed. The filter bed may then be lowered and the contents of the vat discharged into a hopper and conveyed away. The solution of the precious metal is forced through a filter press containing an insoluble sulphide (either of iron or copper), which precipitates the precious metal.—J. B. C.

*Improvements in Calcining and Refining Copper Ore, Copper Regulus and Copper Precipitate, and in the Construction of Furnaces therefor.* F. W. Dähne, Swansea. Eng. Pat. 1641, January 30, 1890. 6d.

THE improvement consists in calcining "white metal" containing about 75 per cent. of copper in a suitable furnace by its own heat of oxidation. The calcining furnace, which is included in the patent, has several horizontal circular beds arranged in tiers and enclosed in rectangular brickwork. Each bed is provided with a mechanical rake, all the rakes being worked by a common shaft. Air may be admitted through the central core of the shaft to each bed of the furnace as well as by suitable openings in the walls. Heat is maintained by combustion of the sulphur. The calcined ore is mixed with 6–8 per cent. of carbon and also with suitable fluxes, and refined in the ordinary way. The "blister copper" process is avoided and arsenic and other impurities are expelled.—J. B. C.

*Improvements in Apparatus or Machinery for Coating Metal Sheets with Metals or Alloys.* R. Heathfield, Darlaston. Eng. Pat. 1682, January 31, 1890. 8d.

THE patent has special reference to the feed and exit rolls in connexion with a galvanising pot. The rolls are boxed in so as to keep them as much as possible out of contact with the surface scum. The rolls may be fixed above or below the surface of the molten metal, and the feed and exit rolls may be inclined at any angle required or readily removed from the pot.—J. B. C.

## XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

### PATENTS.

*An Improved Apparatus for Economically Effecting the Double Decomposition of certain Substances by the Aid of Electricity.* W. H. Dowland and W. Mills, London. Eng. Pat. 2331, February 9, 1889. 6d.

TO obtain the whole of the products of the decomposition of haloids, the chief of which is common salt, a special apparatus is employed consisting of a rectangular vessel divided by porous partitions into three compartments, the two outer of which can be covered with gas-tight covers of convenient form with pipes let into them to allow of any gases being carried away. The central compartment and one of the outside ones are filled with a saturated solution of common salt, and in this outside compartment is also placed the positive electrode of, say, carbon, while the other compartment contains the negative electrode in a solution of caustic soda. On the passage of an electric current through the bath, hydrogen is given off at the negative electrode and chlorine at the positive.—B. T.

*Improvements in the Form and Manufacture of Secondary Battery Frames and Plates.* A. Douglass and F. Smith, London. Eng. Pat. 2544, February 13, 1889. 6d.

THESE frames are cast with V, U, or half-round inside edges in order to retain the active material, spun-glass being mixed with this latter to assist its adhesion; lead wire can also be laced backwards and forwards in a sort of trellis-work for the same purpose.—B. T.

*Improvements in the Extraction of Copper and Zinc from their Ores, as also of the Precious Metals contained therein by Electrolysis, and Apparatus therefor.* C. D. Abel, London. From Siemens and Halske, Berlin, Germany. Eng. Pat. 3533, February 27, 1889. 8d.

THE pulverised ore, together with a solution such as ferrie sulphate, is introduced into long narrow channels arranged in a zigzag fashion, each channel containing two cylinders of suitable material carrying vanes or paddles on their surfaces and caused to rotate by mechanical means, adjacent cylinders rotating in opposite directions, so that the ore, although of higher specific gravity than the liquid, is held in suspension. The temperature of the solution is maintained at any desired point by means of steam pipes passing along the channels. If the amalgamation process be used the steam pipe is replaced by a rotating copper cylinder, the lower part of which dips into mercury in order to keep it amalgamated, and the paddles cause the ore to impinge upon the cylindrical surface, or a salt such as potassium cyanide is added to the liquid and an electric current is sent through the bath to precipitate the precious metal, the copper cylinder serving as the cathode. To regenerate the sulphate of copper solution and to remove the copper it is passed into a cell containing rotating

cylinders as cathodes, on which the copper is deposited, while the solution gradually sinks down through a filtering arrangement past the horizontal anode and is converted into ferric sulphate and is again ready for use.—B. T.

*Improvements in the Preparation of Bleaching Agents.* D. G. FitzGerald, Brixton. Eng. Pat. 5995, April 8, 1889. 6d.

THE patentee has previously described (this Journal, 1887, 515) a process for the regeneration of chromic acid from spent solutions. He now adds to the solution thus obtained sulphuric and hydrochloric acids. Chlorine is given off and may be passed into water or into an aqueous solution containing some base in solution or in suspension within it. The chromium sulphate formed may be electrolysed, chromic acid being produced, but on a further continuance of the electrolytic action, oxygen is evolved which may, together with the chlorine, be used for bleaching purposes.—S. G. R.

*Improvements in the Accumulation of Electrical Energy and Apparatus therefor.* F. Marx, Berlin, Germany. Eng. Pat. 9496, June 7, 1889. 8d.

INSTEAD of using electrodes which become chemically changed during charging and discharging, a solution is employed which alone undergoes alteration, while the carbon electrodes are unaffected. This fluid is termed "electrolin" and may be made up of 450 grms. protochloride of iron, 900 grms. of water, 500 grms. of hydrochloric acid. During the passage of the electric current the protochloride is converted into perchloride of iron. For the discharge, one electrode consists of a metal such as iron and the other of carbon. Salts of copper may also be similarly utilised.—B. T.

*Improvements in Secondary Batteries.* C. H. Logan, Detroit, U.S.A. Eng. Pat. 19,861, December 10, 1889. 8d.

VARIOUS methods of connecting up the plates are described in which wedges are used to produce the required pressure between the contact pieces, or the connecting pieces dovetail into one another. A frame of non-conducting material notched to hold the plates and provided with india-rubber cushions is also shown.—B. T.

*Improvements in Plates for Secondary Voltaic Batteries.* C. H. Logan, Detroit, U.S.A. Eng. Pat. 19,861A. December 10, 1889. 6d.

To present large active surface with contrivances for holding the active material, the lead frames are cast with vertical tubular slots in them closed at the bottom, or with curved or diamond-shaped pockets.—B. T.

*An Improved Method of and Means for the Production of the Grids of Electric Accumulators in which the Bars narrow from each side inwards.* E. Correns, Berlin, Germany. Eng. Pat. 20,235, December 16, 1889. 6d.

THESE plates are formed of a lead framework with square or suitably shaped apertures, across which apertures are cast cross-shaped pieces of triangular section with their apices pointing inwards so as to better retain the active material.—B. T.

## XII.—FATS, OILS, AND SOAP MANUFACTURE.

*Electrical Phenomenon in a Ceresin Manufactory.* B. Laeh. Chem. Zeit. 13, 1671.

TOWARDS evening, in a manufactory of stearin and ceresin in Italy, four tanks holding about half a ton each of white ceresin, were being stirred to assist the cooling. Suddenly the electric lighting accidentally failed, leaving the workroom in darkness, when the workmen were astonished to see pale flashes given off on stirring the solidifying ceresin, whilst sparks of 4 cm. long could be drawn by approaching the hand to the surface of the ceresin. The ceresin was quite free from resin. Such a phenomenon has not been previously reported.—G. H. B.

### PATENTS.

*An Improved Anti-Corrosive Lubricant and Lubricating Vapour for Carbon Disulphide and other Vapour Motors.* D. S. Hawke, Cernees. From E. L. Brady, New York, U.S.A. Eng. Pat. 5974, April 8, 1889. 4d.

FIVE quarts of a weak solution of saponin (e.g., 1 oz. to 5 quarts of water) is mixed with 13 quarts of "paraffin or other light mineral oil," and the product injected into the boiler of any engine using the vapour of such a liquid as carbon bisulphide as its motive power. On condensing the exhaust vapour the lubricant is recovered and may be returned to the boiler. The lubricant is said to accompany the vapour through the engine and protect the working parts.—B. B.

*An Improved Manufacture of a Compound of Vinegar and Oil Applicable for Various Purposes.* H. Noerdlinger, Stuttgart, Germany. Eng. Pat. 9564, June 8, 1889. 4d.

A CLEAR and homogeneous mixture of oil and vinegar may be made by adding to an emulsion of the two a soluble salt, such as common salt, which removes the water, leaving a mixture of acetic acid and oil. If acetic acid be used instead of vinegar, no salt is required. An incidental advantage claimed is the purification of the oil. The product is designed for medicinal or culinary purposes.—B. B.

*Improvements in the Manufacture of Lubricating Grease and in the Apparatus Employed therefor.* P. Plisson, Paris, France. Eng. Pat. 1659, January 30, 1890. 8d.

A MIXTURE composed of 2—10 per cent. of some alkali or alkaline earth, 5—60 per cent. of a vegetable oil, and 25—90 per cent. of a mineral oil, is made by placing the saponifiable oil in a steam-jacketed vat, adding the alkaline substance, and incorporating by the aid of a stirring arrangement consisting of a vertical shaft carrying a frame, one of the vertical members of which is free to rotate and bears blades for stirring the mass, while to another are attached scrapers which fit closely to the wall and bottom of the vat, and clear them from adhering material. The whole of this contrivance can be lifted bodily from the vat for cleaning or the removal of its contents. The manufactured product is designed as a lubricant where heavy pressures and high temperatures exist.—B. B.

### XIII.—PAINTS, PIGMENTS, VARNISHES, AND RESINS.

#### PATENTS.

*Improvements in and in the Manufacture of a Preparation or Substance applicable as a Substitute for India-rubber, and for Waterproofing or Dressing Cloths, Sheetings, and other Articles; also for other Purposes.* S. Kenyon, Warrington. Eng. Pat. 4179, March 9, 1889. 4d.

Oxidised linseed oil is incorporated, by the aid of heat, with the distillate or "foots" produced in the manufacture of varnish from gums or gum resins, or with pine oil, resin oil, or resin; and the mixture is soluble in oil, turpentine, bisulphide of carbon, mineral spirit, or any of the ordinary solvents for india-rubber.—E. G. C.

*An Improvement in the Manufacture of Coloured Sealing Wax.* J. Fremont, London. Eng. Pat. 5349, March 28, 1889. 4d.

INSTEAD of using vermilion or ultramarine, the inventor dyes the barytes or other pulverulent body added to the wax, with aniline colours.—E. G. C.

*An Improved Material for Making Whitewash, Colour-wash, and Water-Colour Paints.* J. J. Carr, Scarborough. Eng. Pat. 13,792, September 2, 1889. 4d.

A COMPOSITION of whiting and other suitable white substances, colours, and binding or glutinous materials, dried and ground to powder.—E. G. C.

*Improvements in or relating to the Manufacture of Vulcanised Plastic Compounds.* A. J. Boulton, London. From W. Kiel, Butler, New Jersey, U.S.A. Eng. Pat. 15,826, October 8, 1889. 6d.

THIS specification, in which there are 13 claims, refers to the production of a hard substance (suitable for use in place of hard rubber, whalebone, ivory, and the like), by vulcanising wood, sulphur, oil, and crude rubber, the mixture thus obtained being reduced to powder and combined with sulphur, oil, beeswax, and crude rubber. (See this Journal, 1889, 204.)—E. G. C.

*An Improved Adhesive Solution.* J. Saunders, E. Kerry, and E. C. Kerry, Highgate. Eng. Pat. 16,673, October 22, 1889. 4d.

A MIXTURE of gutta-percha tissue ( $1\frac{1}{2}$  part), carbon bisulphide ( $1\frac{1}{2}$  part), and methylated ether (1 part).—E. G. C.

*Improvements relating to the Manufacture of White Lead.* H. H. Lake, London. From J. B. Tibbits, Hoosae, U.S.A. Eng. Pat. 18,076, November 12, 1889. 6d.

ACCORDING to this invention, a lead electrode is electrically dissolved in an electrolyte containing free or nascent carbon dioxide, the lead compound formed by electrolytic action being precipitated to form hydrated carbonate of lead, or pure white lead, which is removed, washed, and dried.

—E. G. C.

*Process and Apparatus for the Manufacture of Linoleum Cloth.* D. N. Melvin, Richmond, U.S.A. Eng. Pat. 18,749, November 22, 1889. 8d.

SHAPES of differently coloured linoleum composition are cut from sheets and placed on wooden blocks according to the desired pattern. The shapes are held in position by a number of pins or thin metal strips. The blocks with the shapes in position are inverted and placed on a canvas

backing laying on a heated table. Pressure is applied to the block, causing the shapes to adhere to the canvas. The block is then removed and the shapes are made to form a continuous sheet by further pressure, a hot plate being placed on the surface. A drawing of the blocks and press used accompanies the specification.—E. J. B.

*Improved Blacking Mixture for Use on Heels, Shanks, and Edges of Boots and Shoes.* A. J. Boulton, London. From J. G. Drew, Lynn, Mass., U.S.A. Eng. Pat. 20,754, December 24, 1889. 4d.

A MIXTURE of shellac, water, borax, oil of sassafras, ammonia, and colouring matter (aniline black).—E. G. C.

### XIV.—TANNING, LEATHER, GLUE, AND SIZE.

*Chemistry of Tannic Acids.* C. Etli. Monatsh. 10, 647—664, and 805.

THE results of previous investigations have shown that the tannic acid  $C_{17}H_{16}O_9$  obtained from *Quercus Robur L.*, and that obtained from *Quercus pubescens W.* occur in the oak-bark not only in the form of the tannic acid but also as anhydride; it was also found that the tannic acids which are almost insoluble in water do not occur in combination with a sugar, and are not therefore glucosides; their basis is not tannin but a substance isomeric therewith, namely, a ketone acid (gallylgallie acid) of the constitution—



formed from two molecules of gallic acid with elimination of one molecule of water.

In this paper the author gives an account of experiments, which show that the tannic acid  $C_{16}H_{14}O_9$  is a derivative of the ketone acid referred to above; as this tannic acid closely resembles the acids previously investigated both in physical and chemical properties, it follows that they also are derivatives of the same ketone acid.

The greater part of the sparingly soluble ketone tannic acids occurs in the plant in combination with a metallic base, probably magnesium, and as these salts are very readily soluble in water they can be easily and completely extracted. After concentrating the aqueous extract and acidifying with hydrochloric acid the tannic acid is precipitated in an insoluble condition, and can be washed free from hydrochloric acid and then purified by extracting with alcohol or ether. In this way the tannic acids can be easily obtained in a pure condition; the most suitable source of these acids are the commercial extracts which are obtained from the bark and wood of various trees and largely used in tanning.

The author has isolated the following tannic acids by the method described above. An acid of the composition  $C_{16}H_{14}O_9$  from the extract obtained by the "Oak-Extract Company" in Slavonia from the wood of the stalk-oak. An acid of the composition  $C_{18}H_{18}O_9$  from the oak-bark used in a tannery at Pesth. An acid  $C_{20}H_{20}O_9$  obtained from the bark of the copper-beech in the neighbourhood of Salzburg. An acid of the composition  $C_{22}H_{22}O_9$  from the hop cones obtained from Saaz.

All these tannic acids have a different colour, varying from brownish-red to bright red; those obtained from the stalk-oak and holm-oak give in dilute alcoholic solution a deep blue, the others, however, a green colouration with ferric chloride. It seems that all the tannic acids obtained from the oak are simply different derivatives of the same ketone tannic acid.

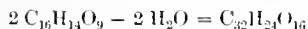
The tannic acid of the composition  $C_{16}H_{14}O_9$ , which occurs in the Slavonian stalk-oak (see above) is best isolated as follows:—The commercial extract, which contains, besides other substances, principally ellagic acid and the tannic acid, is treated with water, the insoluble matter allowed to

settle, and the filtered solution gradually mixed with concentrated hydrochloric acid as long as a red precipitate is produced (excess of acid should not be used). After standing for several days the supernatant liquid is siphoned off, the precipitate collected on a filter, washed with water till free from hydrochloric acid and dried. The dry substance is then treated with 95 per cent. alcohol, the filtered solution mixed with  $1\frac{1}{2}$  volumes of water, again filtered from resinous substances and evaporated to dryness; if the residue thus obtained is completely soluble in 40–45 per cent. (by vol.) alcohol it may be considered to be pure, if not it is redissolved in alcohol of this strength, and the filtered solution evaporated to dryness.

The liquid siphoned from the crude tannic acid contains the whole of the ellagic acid and larger or smaller quantities of tannic acid. It is first evaporated to a small volume, whereon the ellagic acid is converted into insoluble ellagic acid. After adding water the black precipitate is filtered off, washed with water, dried, and extracted with 45 per cent. alcohol. The filtered extract leaves on evaporation the tannic acid,  $C_{16}H_{14}O_9$ , mixed with a small quantity of another tannic acid; the latter can, however, be dissolved out by washing with a large quantity of water.

The tannic acid,  $C_{16}H_{14}O_9$ , is a brownish-red amorphous compound; under the microscope it is seen to consist of small spherical nodules, which are all so much alike that the presence of impurities can be easily detected. It is almost insoluble in water and ether, but very readily soluble in alcohol and acetone. Its solution in dilute alcohol has an acid reaction, gives a precipitate with lead acetate, and a dark blue colouration with ferric chloride. The formation of the phenylhydrazine-derivative,  $C_{22}H_{20}N_2O_8$ , and the oxime,  $C_{16}H_{13}NO_9$ , proves that the acid contains a ketone group.

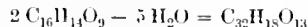
When the acid is heated with dilute sulphuric acid at  $120^\circ$ – $130^\circ$ , it gives gallic acid (m.p.  $238^\circ$ – $240^\circ$ ), and a large quantity of insoluble anhydro-derivatives. It forms a soluble neutral salt, and several very sparingly soluble basic salts with magnesia. When heated alone at  $130^\circ$ – $135^\circ$ , or at  $100^\circ$  with water, in sealed tubes, or when boiled with hydrochloric or dilute sulphuric acid, it yields various very stable anhydrides, a property which is especially characteristic of the ketone tannic acids, and serves to distinguish them from tannin. When the acid is boiled with dilute (1:10) sulphuric acid, for example, as long as a red precipitate is produced it is converted into an acid anhydride,  $C_{32}H_{24}O_{16}$ , according to the equation—



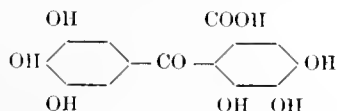
When heated at  $120^\circ$ – $130^\circ$ , under the same conditions, it gives two anhydrides. The one is soluble in 96 per cent. alcohol, and is formed according to the equation—



The other is insoluble in 96 per cent. alcohol, and no longer contains a carboxyl group, its formation being expressed thus:—



These experiments show that the tannic acid contains four hydroxyl groups; its behaviour with hydriodic acid and with hydrochloric acid shows that it contains two methoxy groups. The acid,  $C_{16}H_{14}O_9$ , is therefore a dimethyl derivative of a ketone acid of the constitution—



but the position of the two methoxy groups has not yet been determined.—P. S. K.

*The Testing of Glue.* R. Kissling. Chem Zeit. 13, 1667.

THE author describes a simple apparatus for measuring the tensile strength of glue. It consists of two solid cylinders of nickel-plated iron, with one end of each made smooth whilst the other end has a hole to receive a hook for support. One part of glue with two parts of water are warmed together on a water-bath, in which the metal cylinders are also heated. The solution is then placed between the smooth ends of the cylinders, which are squeezed together and left overnight in an upright position between suitable wooden supports, and surmounted with a 5-kilo. weight to keep them pressed together. The glued cylinders are then hooked on to a lever carrying a scale pan, to which weights are added, until the cylinders part at the glued joint. The process and apparatus resembles that employed in testing the strength of cement. The results of a number of tests are given, from which it is seen that the price of the glue bears no relation to its tensile strength.—G. H. B.

## XV.—AGRICULTURE, MANURES, Etc.

*Bulletins of the Kentucky Agricultural Experiment Station.* Lexington, Kentucky, U.S.A. Nos. 15, 16, 17, 18, 20, and 21.

No. 15. *Wheat Experiments* (1888).—Of 24 varieties sown in October, nine were killed by the winter, viz., Golden Drop, Squarehead, Hunter's White, Browick Red, four Algerian varieties, and Genoese. Diehl Mediterranean, Hicks, Californian, Red Sea, Fulcaster, Velvet Chaff, and Extra Early Oakley gave good results. The latter has yielded on an average of three years 27.9 bushels per acre. Velvet Chaff gave 30 bushels and was the hardiest.

Experiments with wider drills (14 in. apart) to test the effect of twice cultivating between the drills, showed no improvement over the uncultivated plots. Various manurial experiments showed the land to be in too good condition to benefit by dressings.

No. 16. *Potato Experiments*.—The season was unfavourable for potatoes, and the results of comparison of different varieties are vitiated by this circumstance. But experiments on the best sets for planting confirmed unmistakably the conclusions of a previous year, and those drawn from similar experiments at the Maryland Agricultural Station. These are that large potatoes planted whole produce the largest crops, and that next follow in order average whole potatoes, small whole potatoes, large potatoes cut to two eyes, small potatoes cut to two eyes, single eye on good sized piece, single eye on small piece. If the season and soil be bad, it may not pay to use large potatoes for seed, but with a good soil and fair average season it certainly does. For example, in 1886, large whole potatoes gave value of crop less cost of seed 73.80 dollars per acre; cut to two eyes, value of crop less cost of seed 54.30 dollars per acre.

Potash manures alone gave greatly increased crops on the potato field and were by far the most profitable. This result, of course, applies to the particular field—a blue-grass soil on crystalline limestone of Lower Silurian age, cold and backward, with clay subsoil, cropped since (and perhaps before) 1860 chiefly with cereal and a few hemp crops.

"London Purple" sprayed twice over the field at intervals of a week, was found very effectual against the potato beetle.

No. 17. *Maize Experiments*.—The manurial experiments (made on Ville's principle of comparing a complete manure with no manure, and with potash, nitrogen, and phosphoric acid omitted in turn) showed conclusively the value of potash salts as a manure for maize on this soil, the addition of 60 lb. of potassium sulphate per acre to the hillocks only, raising the crop from 31.6 bushels to 63.7 bushels per acre, and of fodder from 3,450 lb. to 6,420 lb. 100 lb. of potassium sulphate gave still better results, not improved by additions of superphosphate or ammonium salts. The improvement was as great in quality as in

quantity. The results of similar experiments on hemp, tobacco and potatoes, and indications obtained with wheat, point to the advantage of using potash generally on similar well-cropped soils of this region. 60 lb. of copperas per acre mixed with earth or with a complete fertiliser produced no increase, or a diminution of crop, though the leaves were for a time of a darker colour.

Tobacco stems, a waste product, gave very promising results as a manure for maize, probably by reason of the contained potash.

No. 18. *Hemp Experiments*.—Potash salts and ammonium sulphate gave the best results with this crop on the well worn land of the station, which was very unfit for hemp cultivation without manure. *Apple orchard*.—Spraying when the apples are about the size of peas with London Purple (1 oz. to 10 gallons water) has been found most effectual against the ravages of the codlin moth.

No. 20 and No. 14. *Commercial Fertilisers*.—Contain an explanation of the principles of manuring, tables giving analyses and valuations of many current samples of bone manures and mixed manures, and reprints of the Kentucky State Acts regulating the sale of fertilisers.

No. 21. *Wheat Experiments* (1889).—Canadian Finley and Hunter's White varieties gave the best looking grain; German Emperor and Egyptian the largest yields, 30 bushels per acre. Diehl Mediterranean, Hicks, and Extra Early Oakley did not do so well as in the preceding year. The manuring experiments showed that little manuring is needed for wheat on this soil, although, taking three years' results, some effect from the fertilisers is shown, especially from those containing potash. Much damage was caused in 1889 by the grain louse (*Siphonophora avenae*), a species of "green fly," the life history of which is detailed in the bulletin.

The following notice, printed with each bulletin, is worth quoting: "It is desired that every farmer in the State shall receive these bulletins, and become interested in the work of the station. Persons already receiving them would confer a favour by sending in the names of any others they know who are likely to be interested in them. The bulletins of the State will be mailed to any citizen in Kentucky who sends his name and address for that purpose."—J. M. H. M.

*Superphosphate Gypsum*. W. Cohn. Chem. Zeit. **13**, 1671.

OBJECTION has been raised to a statement made by the author in an article on the manufacture of artificial manure (this Journal, 1890, 201), apparently to the effect that superphosphate gypsum is not of greatly more value to the farmer than ordinary gypsum as a means for preventing the escape of ammonia generated in farmyard manure. The author justifies his remarks by reference to the careful experiments of König on this subject, and explains the position he has taken up by showing that the production of superphosphate gypsum is confined to a very few factories, and any advantage derived locally by its use becomes quickly neutralised by cost of transport, whilst the general distribution of gypsum factories causes that substance to be available at a cheap rate in all places.—G. H. B.

*Contribution to the Question of Manuring on the Basis of Soil Analysis*. R. W. Bauer. Chem. Zeit. **13**, 1705.

IN relation to a discussion on the subject of manures, the author draws attention to the advisability of ascertaining by analysis in what respect a soil may be deficient before manuring it with substances in which it may be rich. Thus he found a soil rich in nitrogen but very poor in phosphoric acid, magnesia, and sulphuric acid, and he shows by a tabular statement of the mineral matters present in various cereals that the requisite mineral substances, as well as nitrogen, must all be present in appreciable quantities and available to the plant from the soil.—G. H. B.

*Constitution of Bone Meal*. J. Stocklasa. Chem. Zeit. **14**, 1—2, 21 and 32—33.

See under XXIII., page 420.

*Superphosphate Gypsum and Gypsum Phosphate*. T. Meyer. Chem. Zeit. **14**, 2.

SUPERPHOSPHATE gypsum and gypsum phosphate are both by-products of the manufacture of phosphoric acid from natural phosphates. The former is the substance obtained by drying the residue from the phosphoric acid, after this has been liberated by sulphuric acid. It is valuable from the considerable amount of soluble phosphoric acid it contains. Gypsum phosphate, on the other hand, is the waste product obtained when the precipitate is washed out before being dried: it contains about 3·5 to 4 per cent. of phosphoric acid ( $P_2O_5$ ), most of which is insoluble. Being produced by the action of sulphuric acid on very finely powdered phosphate suspended in water, it is obtained in an extremely finely-divided state and is, therefore, well adapted for the absorption of gases. It will, moreover, absorb about 15 per cent. of moisture (beyond the amount required for the formation of hydrate) without forming lumps. It forms an excellent nitrogen preservative for farmyard manure.—N. H. J. M.

#### PATENT.

*Improved Machine for Disintegrating and Pulverising Hay, Straw, and the like*. C. I. Olsen and H. P. Kjoller, Copenhagen, Denmark. Eng. Pat. 19,689, December 7, 1889. 6d.

INSTEAD of cutting hay in the ordinary way the inventors tear it into a fibrous mass, thereby loosening the outer silicious coating and rendering the nutritive portion more easy of digestion. Knives of a peculiar construction are used.

—E. J. B.

## XVI.—SUGAR, STARCH, GUM, Etc.

*Arabinon, the Saccharon of Arabinose*. C. O'Sullivan. J. Chem. Soc. **57**, 59—63.

THIS body was noticed under the name of  $\alpha$ -arabinose in a previous communication of the author (J. Chem. Soc. **45**, 55). It was prepared from the sugars obtained by the limited action of sulphuric acid on geddie acid (a body to be described in a future communication). The sugar syrups thus obtained were fractionally precipitated by ether until the optical activity of the most insoluble fractions rose to nearly  $[\alpha]_D^{20} = 190$ , and further fractionation did not yield precipitates materially differing from one another.

The purified syrup has an optical activity  $[\alpha]_D^{20} = 202$ , a reducing power of  $K = 58$ , the specific gravity of a solution containing 10 grms. in 100 cc. is 1·0395, and it has a sweet taste.

It appears to be arabinon, the saccharon of arabinose; this is borne out by the fact that it yields arabinose on hydrolysis, 104·9 parts arabinose being obtained from 100 parts of the new body. The equation—



requires a yield of 106·3 parts arabinose. The molecular weight as determined by Raoult's method was found to be 239·2, that of  $C_{10}H_{18}O_9$  being 282, and its analysis gave 42·46 per cent. of carbon and 6·55 per cent. of hydrogen, 42·58 per cent. of carbon and 6·38 per cent. of hydrogen being the theoretical numbers.—A. L. S.

*The Application of the Electrical Conductivity for the Determination of the Ash in Raw Sugar.* A. Fock. Zeits. d. Vereins f. Rübenzucker Industrie, 1889, 710.

This is a critical examination of Reichert's work on the subject, together with a description of the apparatus employed and of the reliability of the constants needed for the determination according to Reichert's method (Zeits. Anal. Chem. 28, 1, and this Journal, 1889, 994). Three sources of error in the determination of the electrical conductivity are pointed out, viz.: (1), the method of preparation of the solution; (2), the measurement of the temperature; (3), the inefficiency of the measuring apparatus. These items introduce an error of 0.4 to 0.5 per cent., but are not sufficiently great to interfere with the accuracy required of the method. The main point to settle, and one which is not supported by enough experimental data in Reichert's work, is whether his constant *b*, the consistency of a pure sugar solution, is a constant quantity, i.e., whether the electrical conductivity of such a solution is really equal to nil, and if not, what its value is. And, further, whether the electrical conductivity is independent of the degree of dilution of the solution. Experiments by the author decide the first point in the affirmative. As regards the second, it is shown that the values for *b* vary considerably with the degree of concentration of the solution, and that the most constant and maximum values are obtained with 1/10 and 1/20 normal solutions (a normal solution contains 26.048 grms. of sugar in 100 cc. of water). The value of *b* also increases with the percentage of ash in the sugar. Since the reliability of the method depends upon the constancy of this value *b*, it appears that the extended applicability of the method pointed out by Reichert is out of the question, but for sugar containing a small percentage of ash the error does not exceed 0.1 per cent., and this is sufficiently accurate. The objections to the method for practical use are pointed out as consisting in the careful manipulation required, the constant attention necessary, and the great difficulty of keeping the solution tested at a constant temperature, without which the results are of little value, since the electrical conductivity varies greatly with the temperature.—C. A. K.

#### PATENTS.

*Improvements in Preparations of Cane and Beet Sugar.* A. G. Salamon, London. Eng. Pat. 3526, February 27, 1889. 6d.

THE invention consists in the production of mixtures of saccharin (benzyl sulphonic imide) with cane or beet sugars, the resulting products being much sweeter, weight for weight, than either cane or beet sugar alone. By the addition of  $7\frac{1}{4}$  lb. of pure saccharin to 1 ton of sugar, the sweetening power of the sugar is doubled weight for weight. A claim is also made for decreased tendency to fermentation in the saccharinated sugar.—A. J. K.

*Improvements in and connected with Apparatus for Evaporating and Concentrating Saccharin or other Solutions.* J. G. Chapman, London. Eng. Pat. 8596, May 23, 1889. 8d.

IN order to facilitate the discharge of the "masse euite," the size of the discharge valve is greatly increased. To effect this the bottom of the pan is made moveable, so that the area of the discharge outlet is equal to the section of the vacuum pan at its base.—A. J. K.

*Improved Process for obtaining the Entire Quantity of Crystallisable Sugar contained in Sugar Mass.* C. Steffen, Vienna, Austria. Eng. Pat. 20,515, December 20, 1889. 4d.

THE methods hitherto known and applied for setting free sugar masses from thin syrup are limited to the separation of the syrup from the crystals. The syrup is afterwards

allowed to cool down until the crystallisable sugar which it still contains separates out. By this method the sugar of the syrup, instead of going to increase the size of the crystals of the "masse euite," is obtained separately in a finely crystalline mass of low commercial value. According to this invention, the whole of the pure "masse euite" is run off into suitable vessels, mixed with molasses of the refinery to a thin homogeneous crystalline pulp. The whole mixture is now submitted to a cooling and stirring process for from 12 to 24 hours.

The entire crystallisable sugar contained in the mother-liquor of the "masse euite" is in this manner won in a marketable form, together with the sugar crystals previously formed in the vacuum pan.—A. J. K.

*Improved Treatment of Sugar Crystals or Grains for Facilitating the Purifying or Washing thereof.* C. D. Abel, London. From Pfeifer and Langen, Cologne, Germany. Eng. Pat. 1282, January 24, 1890. 4d.

IN order to facilitate the washing of the grains of "masse euite" or other granular sugar in cases where the grains are very small and the "masse" is dense, the sugar to be washed is previously treated as follows:—(1.) The "masse" is first heated and then allowed to cool slowly; (2.) The "masse" is subjected to a drying process.

—A. J. K.

#### ERRATUM.

This Journal, 1890, 312, left-hand col., lines 13 and 21 from bottom, also right-hand col., lines 1, 5, 26, and 29, for "Mannosic" read "Mannonic." Right-hand col., line 1, for "Arabinosic" read "Arabinonic."

### XVII.—BREWING, WINES, SPIRITS, Etc.

*The Decomposition of Gelatin by Anaerobic Bacteria.* L. Sclitrenny. Monatsh. 10, 908—917.

THE author dissolved 800 grms. gelatin in 16 litres of water, which was sterilised, and then inoculated with *Bacterium liquefaciens magnus*. The air was then expelled from the vessels holding the solutions by a current of carbon dioxide.

The solution was investigated, one portion after a period of 22, and another after 32 days.

The liquid was distilled from a retort after the addition of oxalic acid. A small quantity of methyl mercaptan was collected, but indol, scatol, and phenol were not detected. The chief portion of the distillate consisted of phenyl propionic acid. The residue in the retort, after precipitation of the oxalic acid, was found to be mainly glycecol, which was separated in the form of the copper salt.

A second experiment was conducted similarly, but in presence of a small quantity of air. In the second experiment, the distillate contained phenyl acetic acid as well as phenyl propionic acid, the former being the larger amount. In both cases a considerable quantity of gelatin peptone was formed.

The author supposes that by the complete decomposition of gelatin in this way, one-half is converted into peptone, and the other into crystalloid substances, of which 2—3 per cent. is phenylamidopropionic acid.

The fermentation products of gelatin differ from those of ordinary albumen, in that no *p*-oxyphenylpropionic acid nor scatol acetic acid are formed, and also by the presence of large quantities of amido acids of the fatty series in the case of the gelatin fermentation.—J. B. C.

*Substances occurring in Raw Spirit.* H. Bornträger. *Zeits. Anal. Chem.* **28**, 670. (Compare this Journal, 1889, 64.)

In continuation of the author's investigations on the reactions of substances occurring in raw spirit, the principal characteristics of normal propyl alcohol and fermentation butyl alcohol are given.

Normal propyl alcohol has a pleasant fruity smell, and boils at 97°; with water, at 87°. Its specific gravity is 0.813, and it is readily soluble in water. It is not extracted from a 33 per cent. alcoholic solution by shaking with chloroform.

Fermentation butyl alcohol has an unpleasant smell, recalling that of fusel oil, and boils at 108°–109°. Its specific gravity is 0.805, and it is only sparingly (1:10) soluble in water. It is extracted from its solution in 33 per cent. alcohol by shaking with water. When dissolved in alcohol and the solution treated with three drops of concentrated hydrochloric acid and 10 drops of aniline, a red colouration is produced, but only in strong solutions.

If raw spirit is diluted to 33 per cent., and then shaken with chloroform, the extract will contain amyl alcohol, acetal, aldehyde, and fermentation butyl alcohol, whilst ethyl alcohol, acetic acid, and tertiary butyl alcohol will remain in the dilute alcoholic solution.—F. S. K.

*The Development and Prospects of the Spirit Manufacture in Hungary.* A. von Asbóth. *Chem. Zeit.* **14**, 65–66 and 127–128.

HUNGARY being a wine-producing country, little attention was paid to the manufacture of spirits until the beginning of this century, when the trade being found profitable grew largely, and soon supplied the home market and began to produce a surplus for export. To meet these altered conditions fiscal changes were made which considerably hampered the industry. In 1862 the number of distilleries and their output declined considerably, and though their burden of taxation was somewhat reduced in 1865, yet the many small distilleries attached to estates and not worked as separate enterprises were unable to compete successfully with the large manufactories. For the relief of the former measures were adopted which resulted in a further rapid growth, until production was again checked by the bounties granted by neighbouring countries, especially Russia and Roumania. But the regulations then in force were successful neither financially nor industrially, as may be gathered from the facts that the tax yielded only 66 kr. per head, while in other European States the return varies from 2.25 fl. to 5 fl. per head, and that the favoured small distilleries adopted wasteful methods of working. The upshot of these difficulties was the imposition in 1888 of an uniform tax on the output of spirit, which was made legal in both Austria and Hungary and had the following provisions: 35 kr. were levied on each litre of alcohol supplied for home consumption, which was reckoned at 1,878,000 hectolitres, and divided among the distilleries in due proportion. If a distillery made more than its allotted share, the surplus was taxed at the rate of 45 kr. per litre. This tax was not levied on spirit exported in other forms, such as liqueurs, nor was it required from spirit for industrial, medicinal, or scientific purposes, a duty of 1½ kr. only being charged. Spirit for export was also made duty-free. For the benefit of the smaller distilleries, a bounty of 3 fl. per hectolitre for those producing 4–7 hectolitres per day; of 4 fl. for those producing 2–4 hectolitres per day, and of 5 fl. for those producing less than 2 hectolitres per day was granted. To encourage export, a bounty of 17½ kr. per hectolitre-degree on untaxed denatured spirit, and 5 kr. on duty-paid spirit was instituted, with the proviso that the total sum thus paid should not exceed one million gulden, to ensure which only half the bounty was paid at the time of export, and the rest at the end of the season. Duty-free spirit is denatured with wood-spirit and pyridine bases; in order to ensure its detection if added to potable liquids, phenolphthalein is also added. An opinion on the effect of these regulations cannot be given until the returns are available.

In conclusion, the author remarks that the Bohm-Fredersdorf and Ulger stills are chiefly used in the larger Hungarian distilleries; that the raw materials employed include maize, potatoes, wheat, rye, sugar-beet, molasses, different kinds of fruit, and refuse from the wine and sugar manufactures. An idea of the size of the industry may be gathered from the fact that there were, in 1887, 65 distilleries which paid a tax of more than 20,000 fl. apiece.—B. B.

*The Quality of Commercial Alcohol.* J. Szilágyi. *Chem. Zeit.* **14**, 66.

SPRIT made on the estates of various Hungarian land-owners (see preceding abstract) was examined for fusel oil by the Röse-Herzfeld method (this Journal, 1888, 871) with the following results:—

Source of the Crude Spirit.	Alcohol.	Fusel Oil.
	Per Cent. by Weight.	Per 100 Parts abs. Alc.
Potatoes .....	89.04	0.3279
	89.71	0.2566
Turnips .....	80.50	0.793
	78.10	0.8178
Maize .....	81.95	0.2068
Maize and potatoes .....	80.74	0.3629
Molasses .....	81.35	0.3745

The percentage of fusel oil increases after long uninterrupted use of any form of continuous still; on cleaning the still used for the production of maize spirit (see table) it fell to 0.1671 per cent. The process of purification commonly adopted consists in diluting the crude spirit to the extent of about 50 per cent. with water, filtering through linden-wood charcoal and rectifying; the portion collected after the first runnings (which are not marketable) is known as "second quality" ("secunda sprit"), the next as "good" or ordinary quality, the next as "fine quality," and the middle fraction as "finest spirits of wine," after which fractions similarly named in reverse order come over, and finally last-runnings and fusel oil. The character of these products may be gathered from the following figures, which were obtained by the examination of those manufactured by a single large firm:—

—	Alcohol.	Fusel Oil.	Remarks.
	Per Cent. by Weight.	Per 100 Parts abs. Alc.	
Second quality	93.4	0.0206	Perceptible aldehyde reaction.
Ordinary do.	93.4	0.0	Trace of aldehyde.
Fine do.	94.6	0.0	.. ..
Spirits of wine	94.6	0.0	.. ..

—B. B.

*The Action of Diastase on Ungelatinised Starch.* C. J. Lintner. *Brauer and Mälzerkalender*, **13**, 83.

THE author has made some experiments to determine the action of diastase at various temperatures on unaltered starch. In each experiment 2 grms. of air-dried starch were treated for four hours with 50 cc. of malt extract. The solution was then diluted to 100 cc. and filtered from unaltered starch. The filtrate was inverted with hydrochloric acid, the glucose formed being determined by Fehling's solution. The following table shows the



percentage of dry starch converted in each case by the diastase :—

Nature of Starch.	Temperature.			
	50° C.	55° C.	60° C.	65° C.
Potato starch.....	0·13	5·03	52·68	90·34
Rice starch .....	6·58	9·68	19·68	31·14
Barley starch .....	12·13	53·30	92·81	96·24
Green malt starch .....	29·70	58·65	92·13	96·26
Cured malt starch .....	13·07	56·02	91·70	93·62
Wheaten starch.....	..	62·23	91·08	94·58

—H. T. P.

*A New System of Brewing.* R. Wahl and M. Henius. Der Braumeister, Chicago, 3, 88.

THE authors describe a new system of brewing, designed by L. Frisch, for the purpose of more completely utilising the starch of raw grain and malt. The raw grain is mixed with water and heated to a temperature of about 115° R. (143·3° C.) in a closed iron boiler. The mixture is then cooled to 55° R. (68·3° C.), ground malt is added, and the whole is stirred up until the conversion into maltose and dextrin is complete. Frisch employs for these operations Hollefreund's apparatus, as used in distilleries. Further, it is possible to produce by means of this apparatus a pale all malt beer. In this case the mash is boiled in vacuo at 65° R. (81·4° C.). A trial mash was made in the experimental brewing station at Chicago, using maize and malt. The ratio of sugars to non-sugars in the raw grain wort was 100:51·7; in the finished wort before pitching, 100:44. The finished beer gave the following results on analysis: Saccharometer reading, 4·15 per cent.; alcohol, 3·48 per cent.; extract, 5·81 per cent.; sugar, 2·17 per cent.; albuminoids, 0·48 per cent.; lactic acid, 0·86 per cent.; ash, 0·17 per cent. The beer and worts were, therefore, of normal composition.—H. T. P.

#### PATENTS.

*Improvements in the Preparation of Malt.* G. Reininghaus, Mainz, Germany. Eng. Pat. 5981, April 8, 1889. 6d.

IN the process of soaking or steeping of the grain (barley) in breweries and brandy distilleries, the grain is usually conveyed to the steeping troughs by means of elevators, carts, spirals, &c. By the present invention the grain is carried through pipes by a current of water.—A. J. K.

*An Improved Process and Apparatus for Testing the Presence of Colouring Matter in Wines.* L. Mathieu and J. Morfaux, Constantine, France. Eng. Pat. 6083, April 9, 1889. 8d.

WHITE ungummed silk is immersed for 12 hours in a bath of one part of nitric acid (63° B.) and nine parts of water; it is subsequently well washed with water. A bunch of silk so treated is then suspended, by means of a ring, from the metal cap of a test tube, filled with the wine to be tested. The immersion in the wine lasts 10 minutes, after which the silk is pressed between the fingers and plunged into a tube containing a solution made up as follows: 450 grms. of neutral acetate of lead in crystals are dissolved in 1,000 grms. of distilled water containing 20 cc. of glacial acetic acid; dilute this solution with 10 times its volume of water. If the wine be unadulterated with colouring matter the silk as above treated turns green, greyish, or bluish, according to the character of the vintage. If any red tint remains it denotes the presence of some of the red coal-tar dyes, or of cochineal, carmine, or orseille.—T. L. B.

*Improvements in the Manufacture of German Yeast.* C. R. Boune, London. Eng. Pat. 6918, April 25, 1889. 4d.

THE patentee considers that the worts obtained in the usual manner do not contain sufficient albuminoid for the proper

growth of the yeast, and he extracts a further quantity from the spent grains remaining after the usual mashing.

The spent grains are placed in a high pressure converter with a little water and a small quantity of acid to prevent "caramelisation," and heated for 3—4 hours under a pressure of about four atmospheres. By this treatment most of the albumen will have become soluble. This may be filtered from the residue and added to the original wort and the yeast grown in the mixture.—A. L. S.

*Improvements in Apparatus for Sterilising Fermented Alcoholic Liquors.* L. A. Einzinger, Worms, Hesse. Eng. Pat. 8343, May 20, 1889. 8d.

THE inventor passes the liquor to be sterilised through three series of tubes, each series being arranged in a water tank. The water in the first tank is kept boiling by injected steam, while the water in the second tank is kept cold by a continuous current of water, the third tank being filled with ice. The alcoholic liquor passing through the tubes is thus first of all heated to the boiling point, which completely sterilises it, then cooled as much as possible by cold water, and lastly, reduced to nearly the freezing point by ice. If beer, or any other liquor containing dissolved carbonic acid, be treated by the apparatus, the tanks should be arranged one above another, so that the gas disengaged by the heating may rise into the cool liquor and be re-absorbed. Drawings of the apparatus accompany the specification.

—A. L. S.

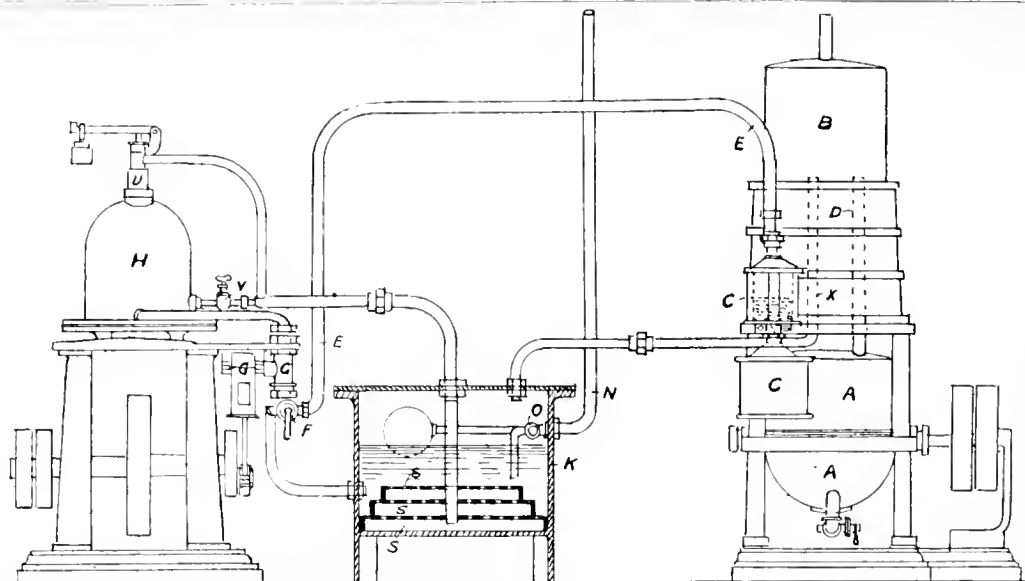
*Improvements in and relating to Distillatory Apparatus.* A. de Kunwald, Paris, France. Eng. Pat. 16,478, October 18, 1889. 8d.

THERE are two claims, the first for an improved method for heating the still by steam, and the second for a valve for regulating the amount of wash passing into the still, according to the amount of spirit distilling off. The steam heater consists of a boiler and a steam jacket to the still, the steam from the boiler being conducted to the jacket by one pipe and the condensed water run back to the boiler by another. The apparatus may be simplified by making the boiler also serve as the steam jacket, the still being mounted over the boiler. The regulating valve consists of a pressure gauge attached to the still, which, by means of levers, controls a valve on the pipe conducting the wash to the still. As the opening by which the vapours escape from the still is of constant size, so, the greater the amount of evaporation, the greater will be the pressure inside the still, and the regulating valve will admit more wash, and *vice versa*. Drawings of the apparatus accompany the specification.—A. L. S.

*Improvements in or relating to Apparatus for Manufacturing Aërated Water Beverages.* J. McEwen, Manchester. Eng. Pat. 20,490, December 20, 1889. 6d.

THE apparatus described herein belongs to that class in which aërated waters can be manufactured "continuously." A is a generator in which the carbonic acid gas is liberated, and conducted by pipe D into the gas-holder B. C is a washing apparatus through which the gas passes on its way to the condensing pump through pipe E. K is a closed cistern, supplied with water through pipe N and valve O. The condensing pump G draws a combined charge of gas and water through the three-way cock F, and delivers it into the condensing vessel H, from whence it is drawn off to the bottling machine. The escape valve V is adjusted so that the excess of water, charged with gas thrown by the pump, over that taken away by the bottling machines, is returned to the tank K, through the contents of which it is distributed by the perforated inverted trays S S S. The weighted relief valve U serves for the automatic return of the charged water to the tank K when the valve V does not take it away from the condensing vessel at a sufficient rate. The apparatus as shown is capable of delivering a "continuous" supply of aërated water.—C. C. H.

(For drawing see top of next page.)



*Improvements in Apparatus for Syringing Aerated Water Beverages.* J. McEwen, Manchester. Eng. Pat. 20,845, December 28, 1889. 6d.

THE improved syringing pump will be understood from the annexed illustration. K is a glass cylinder enclosed in a metallic casing J, in this is a pump plunger X with hollow piston rod. H is the inlet valve of the pump admitting syrup from pipe F. Q, on the outer end of the rod, is the outlet valve. The adjustable screwed tube V, provided with lock-nut W, regulates the stroke of the pump, and hence the quantity of syrup delivered. The bottle is placed as shown, and driven upwards by the treadle B.—C. C. H.

(For drawing see next column.)

*Improvements in or Connected with Sterilising Apparatus.* J. F. H. Gronwald and E. H. C. Oehlmann, Berlin, Germany. Eng. Pat. 206, January 6, 1890. 6d.

See under XVIII. (A.), page 405.

## XVIII.—CHEMISTRY OF FOODS, SANITARY CHEMISTRY, AND DISINFECTANTS.

### (A.)—CHEMISTRY OF FOOD.

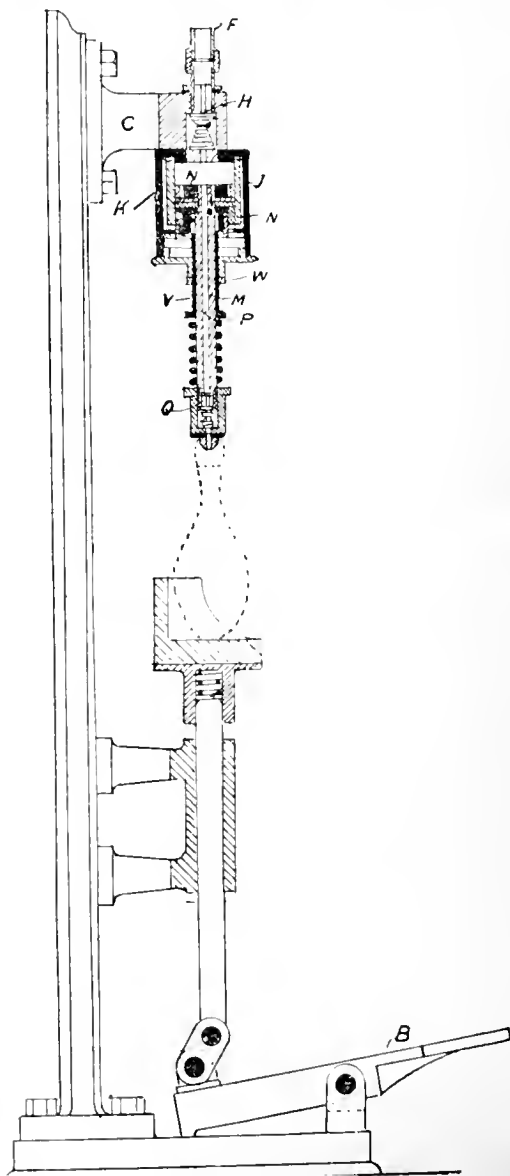
*Sulphurous Acid in Preserved Foods.* J. Brand Bayer. Ind. and Gewerbebl. N. F. 1889, 21, 703.

THE limits (in Germany) to the amount of sulphurous acid allowed, are in beer 10 mgr. per litre, and in wine, 20 mgr. per litre. Direct addition of sulphurous acid or its salts to foods for preservative purposes is not allowed. The author has detected in the liquid from a bottle of preserved asparagus, 0.157 per cent. sulphurous acid and 0.195 per cent. sulphuric acid, part of which was probably produced by oxidation of sulphurous acid.—A. L. S.

### PATENTS.

*Further Improvements in the Preparation and Combination of Animal Substances for use as Food.* J. L. Johnston, London. Eng. Pat. 19,009, December 29, 1888. 6d.

THE first portion of this specification deals with the preparation of sterilised pulverised meat. The meat operated upon is chopped or suitably minced and heated in a closed



vessel at a temperature of from 240° to 300° F. from half an hour to two hours. In order to facilitate the reduction of moisture the interior of the vessel is connected with a suitable exhausting apparatus. It is sometimes desirable to admit superheated steam to the interior of the vessel so as to effectually destroy all spores which would lead to subsequent putrefaction, or the interior surfaces may be cleansed by the use of an antiseptic, e.g. bisulphite of lime. The meat is then removed to a drying room, where it is spread on steam-heated shelves and kept at a temperature of 100° to 180° F., after which it is sufficiently dry to be comminuted or pulverised. The second portion of the specification deals with the preparation of non-coagulable albumen. This substance if heated becomes first coagulated; at a temperature of 265° F. it is converted into a deep brown jelly, and if heated up to 330° F. it becomes finally liquid and does not again coagulate. Albumen so prepared, if heated with an equal bulk of meat peptone, solidifies to a jelly. The various substances described above may be mixed in varying proportions in order to produce a suitable food. The third portion of the specification describes the substitution in such foods of the vegetable product "Japanese isinglass" for animal gelatin.—C. C. H.

*Improvements in the Manufacture of Butter.* J. Boyd, Elmhurst, Illinois, U.S.A. Eng. Pat. 10,693, July 2, 1889. 8d.

THE Danish method of securing a good yield of butter from cream by the addition of a small quantity of skim milk which has been soured by heating and allowing to cool is not always certain in its action. The inventor finds this is due to fluctuations of temperature during the cooling, and rectifies the defect by placing the milk and also the cream used in vessels enclosed in a non-conducting casing. The operation is then as follows:—The "starter" of skim milk is prepared by heating to 90° F. and cooling in a vessel as described above for 24 hours; this is rubbed through a hair sieve and added to the cream at a temperature of 65° F. enclosed in a non-conducting coated vessel in the proportion of one pint of "starter" to 100 quarts of cream, allowed to stand 24 hours and then churned. The yield of butter is then found to be good in quality and quantity and uniform.—C. C. H.

*An Improved Infusing Apparatus, chiefly Designed for Use in obtaining Infusions of Coffee or Tea.* E. Ringaud, Paris, France. Eng. Pat. 13,224, August 22, 1889. 8d.

A METALLIC boiler is connected by means of a siphon with an upper receptacle of glass containing the substance to be infused. The boiler is provided with a screw valve, having a side channel for the admission of air. The water being heated is caused to rise up the siphon into the receptacle, producing a vacuum in the boiler. On discontinuing the heating of the boiler, the atmospheric pressure on the liquid in the receptacle causes it to descend, but the valve being now opened equalises the pressure and prevents the liquid saturated with coffee, &c., from descending into the boiler during the time of the infusion. The infusion having been made, the valve is closed and heat again applied to the boiler for a few seconds. The small quantity of water remaining in the boiler is vaporised and carries the air with it. A vacuum is re-established and the liquid is forced out through the siphon, leaving the residue of coffee, &c., the whole aromatic principle having been expressed. The infusion is thus prevented coming in contact with the hot metal and is drawn off by a tap near the bottom. The principal advantage consists in obtaining the infusion without heating anything but the water.—E. S.

*Improvements in the Preparation of Rennet Powder for Use in Cheesemaking.* R. Dendy, Bristol. Eng. Pat. 17,380, November 2, 1889. 4d.

THE vells are finely divided and then macerated in a solution of common salt containing from 12½ to 18 per cent. of salt. After separation of the mucus, &c., a clean extract is

obtained by filtration and a further quantity of salt not exceeding 5 per cent. is added. The rennet precipitated is dried at a temperature under 35° C. and additional salt added according to the desired degree of concentration. The extract from which the ferment has been precipitated may be used as a weak solution of rennet or as a menstruum for the preparation of liquid extract of rennet. The patentee claims that the ferment as prepared above yields a superior cheese to that prepared in the usual way by the aid of acids or certain preservatives.—C. C. H.

*An Improved Method of and Apparatus for Determining the Quantity of Fat in Milk.* L. K. Lindström, Akerby, Sweden. Eng. Pat. 20,859, December 28, 1889. 6d.

THE fat is obtained in the solid state and in this respect the method is an improvement on the older methods, where the fat is obtained in a liquid or semi-liquid state. 10 cc. of milk are measured into a test tube and a similar quantity of hydrochloric acid, either alone or mixed with acetic acid or sulphuric, is added. The mixture having been well shaken is introduced into a narrow cylinder and subjected to a boiling temperature for 15 minutes. The cylinder is then placed horizontally in a centrifugal machine and the machine rotated at a speed of 2,000 to 3,000 revolutions per minute for five minutes, the temperature being kept at 55°–60° C. by a stream of water running through the machine; revolution is continued for another five minutes at a temperature of 10° C. The cylinder is then removed and the fat may be read off by means of a scale and vernier, as it will have deposited itself in a semi-transparent layer with distinct edges. A large number of tests may of course be made at one and the same time. The results agree very well with results obtained by analysis. Drawings of the apparatus are given in the specification.—T. L. B.

*Improvements in or connected with Sterilising Apparatus.* J. F. H. Gronwald and E. H. C. Oehlmann, Berlin, Germany. Eng. Pat. 206, January 6, 1890. 6d.

THE invention relates to apparatus for sterilising milk and other liquids in bottles, the liquid contained in which can be sterilised under steam pressure, thereby preventing the boiling over of the liquid, and the bottle, immediately after the operation, can be closed inside the apparatus, without coming in contact with the outer atmosphere. A heating vessel contains a stand, with receivers for the bottles. Through the bottom of the vessel and bottle stand a rod, guided in a stuffing-box, passes, and carries at the top a press-plate, while at the bottom it is connected to a lever. The bottles, provided preferably with Fritznor's stoppers placed loosely upon the necks, are placed upon the receivers in such a manner that the press-plate in descending closes all the bottles simultaneously. The vessel being filled with water, the cover is bolted on, and heated by steam. The milk is gradually raised to boiling point; the steam meanwhile filling the interior of the apparatus, not only sterilises the air, but, by its pressure, prevents the rising and boiling over of the milk, and allows the bottles to be almost completely filled, instead of partially, as in the ordinary method. Sterilisation having been completed, the pressure-plate is drawn down, and all the bottles closed simultaneously. "As a mixture of air and steam was contained in the apparatus during boiling, the refrigeration of the milk in the bottles will not produce any appreciable vacuum to affect the contents injuriously."—E. S.

#### (B.)—SANITARY CHEMISTRY.

*Experiments on the Movements of Sewer Air at Wimbledon.* 1888. W. S. Crimp. Proceedings of the Institution of Civil Engineers, 97. Part 3.

THIS paper gives an account of a long series of experiments made by the author to determine exactly the causes which influence the movements of air in a sewer. It has generally been supposed that the direction of such movements

depended upon differences of level or differences of temperature. These suppositions are, however, shown to be quite erroneous, and the author conclusively establishes that in many instances the movements are in a direction contrary to that in which it should be, according to the conditions of one or other of these circumstances. Further, the air currents may be downward or upward, and entirely depends upon the direction and effects of the wind. His conclusions are, "that the wind is the only agent affecting sewer ventilation which need be recognised, and its proper utilisation is a simple and inexpensive matter."—C. C. H.

#### PATENTS.

*Improvements in the Treatment of Sewage.* T. Donnithorne, Mill Hill. Eng. Pat. 11,663, August 13, 1888. 11d.

This specification will not admit of proper abstraction. It deals with the transportation of the sewage and a combination of processes for the treatment of the latter, for details of which the original must be consulted.—C. C. H.

*Improvements relating to the Treatment of Sewage, and to Apparatus therefor.* W. Clark and W. A. Clark, Charlton, and K. Ginnam and W. Ginnam, Plumstead. Eng. Pat. 14,108, October 1, 1888. 1s. 1d.

The sewage is diverted into channels from the main sewer, and each of such channels communicates with a closed filter. The filter is cylindrical; the walls of the cylinder are formed of sheets of gauze or other fabric containing between them a bed of filtering material. The interior of the cylinder is provided with a revolving scraper or "flyer," like a centrifugal pump, and this serves the double purpose of imparting centrifugal force to the water, and hence an urged filtration, and also scrapes the interior of the filter clean, forcing the solid matter through a door or valve in the bottom of the filter. The liquid from the filter is conducted away by channels beneath each row of filters, and the solid matter from the filters is conveyed away by an endless band.

—C. C. H.

*Improvements connected with Apparatus employed for Softening or for Clarifying Water.* A. K. Brown, London. Eng. Pat. 14,761, October 13, 1888. 8d.

This invention describes modifications in the arrangements of the settling plates in the well-known "subsidence" form of apparatus used in the process of water softening. The first modification described and illustrated shows the corrugated plates employed with the line of corrugations placed alternately vertically and horizontally. (2.) Plain flat sheets inclined may be set in a tank to fulfil similar functions. (3.) Plain sheets may be disposed in a tank so that one of their ends can be moved or shaken in order to loosen the deposit which settles upon them.—C. C. H.

*Improvements relating to the Filtration of Water and to Apparatus therefor.* G. D. Gerson, Hamburg, Germany. Eng. Pat. 15,330, October 24, 1888. 8d.

The filtering apparatus herein described is applicable to the filtration of water for technical purposes merely by the removal of the suspended matter and, in addition, its further purification for drinking purposes. Cylindrical vessels are suspended in pairs on trunnions and suitably connected; these are packed with sponge compressed between perforated plates operated by a screw external to the cylinder. The upper portion of the filter is packed with pumice stone, and both this and the compressed sponge may be saturated with an antiseptic insoluble salt, such as tannate of iron. If the water is to be used for domestic purposes after treatment in the filters above described, it is passed through a similar pan packed with treated pumice stone.—C. C. H.

*Improved Apparatus for Screening and Extracting the Suspended Matter from Sewage or other Liquid.* C. F. Gower, Ipswich. Eng. Pat. 1144, January 22, 1889. 8d.

THE improved screening apparatus is set in a chamber through which the sewage passes. It consists of a continuous web of wirework passing over two rollers, one set in the screening chamber and below the level of its bottom, the other some little distance above the top of the walls of the chamber. On the exterior surface of the web of wirework strips of iron forming shelves or pockets are secured. Rotation of the upper drum secures the movement of the continuous screen, bringing with it the matter separated. This drops off into a suitable receptacle, or is removed by revolving brushes to which an endwise motion is also given.

—C. C. H.

*An Improved Method for Softening and Clarifying Water and Apparatus therefor.* L. Archbutt and R. M. Deeley, Derby. Eng. Pat. 1791, February 1, 1889. 8d.

WHEN water is softened by Clark's process, the precipitate, formed takes several hours to settle. If, however, when the reagents have been added, the settled precipitate from a former operation be thoroughly mixed with the water under treatment, the settlement is much more rapid, indeed half an hour suffices to obtain water sufficiently free from solid matter for most purposes. The apparatus described consists of a tank large enough for the precipitation, a reagent vessel from which the precipitating agent is added, and a well inside the precipitating tank provided with a trajectory so as to secure a thorough circulation and consequent mixture of the water and the reagents used. A series of perforated pipes along the bottom of the tank are provided, so that by means of steam or air the mud from a previous operation can be mixed with the freshly treated water.—C. C. H.

*Improvements relating to the Treatment of Feculent Matter, and to Apparatus therefor.* H. H. Lake, London. From J. Hirschfeld, Philadelphia, U.S.A. Eng. Pat. 6528, April 16, 1889. 8d.

IN specification of Eng. Pat. 17,793 of 1888, the present patentee described a form of closet or receiver for feculent matter similar to Moule's earth closet. The present specification describes various improvements effected in the arrangements of the parts of the apparatus referred to, such improvements being covered by 18 claims.—C. C. H.

*Improvements in and relating to Means for Purifying Water.* J. Davis, Allegheny, Pennsylvania, U.S.A. Eng. Pat. 7630, May 7, 1889. 8d.

THE patentee describes at considerable length an improved apparatus for the treatment and purification of water. Generally described this consists of an oblong closed vessel with convex sides and a dished top and bottom containing filtering material resting on a perforated false bottom. Filtering materials similar to those usually employed rest on this perforated bottom. Prior to entering the filter the incoming water drives a turbine which works air pumps for the purpose of aerating the water, and also actuates a series of vertical agitating shafts provided with blades which effectually stir up the filtering beds so as to secure "a free passage of the water through it." The incoming water is also mixed in proper proportions with a suitable reagent, e.g., alum, so as to "coagulate the animalcular and other impurities."—C. C. H.

#### (C)—DISINFECTANTS.

#### PATENTS.

*Self-acting Apparatus for the Periodic Dispersion of Disinfectants, Antiseptics, or other Substances of a like Nature in the Form of Jets and Sprays.* A. R. Upward, London. Eng. Pat. 2248, February 8, 1889. 8d.

IN this apparatus a jet of water is utilised to produce an air blast which actuates the sprays or jets. A jet of water of

high pressure issuing from a nozzle of small aperture flows into a passage somewhat larger than the nozzle and immediately opposite it, and in so doing draws air in with it, and both enter an air-tight vessel in which is a float actuating a waste valve so adjusted as to provide an air-tight space in the upper part of the vessel for the compressed air. A pipe communicating with the air space supplies the spray with the necessary blast. To obtain an intermittent action of the sprays, the above vessel is combined with a tap controlling the supply of water, and consists of a counterbalanced lever and bucket fitted to the tap, the bucket lever periodically opening and closing the valve by the variation of its weight by being first filled from a tipping cup, supplied by an independent minute and accurately adjusted drip from the main, and then afterwards emptied by a minute aperture at the bottom of the bucket. "Where the spray is only occasionally required, the valve connecting the nozzle with the water supply is so connected with the door of the place in which the spray jet is placed, that it is opened by the opening of the door. The valve is fitted with a dash-pot, which only allows the valve to close slowly."—E. S.

*A New Disinfectant and Deodoriser.* A. J. Shitton, Reading. Eng. Pat. 2537, February 13, 1889. 4d.

THE new disinfectant consists of iodine dissolved in an alkaline iodide, *e.g.*, 1 oz. of iodine, 2 oz. of potassium iodide, 1 gallon of water.—C. C. H.

## XIX.—PAPER, PASTEBOARD, Etc.

*Tobacco Juice as a Reagent for Lignin.* A. Hbl. Chem. Zeit. 14, 67.

See under XXIII., page 418.

*On the Physical Changes of Paper when Stored.* W. Herzberg. Mittheil. der Kön. techn. Versuchsanstalten, 1890, 50—52.

THE author has examined at different times three kinds of paper which were stored away for the last three years. These papers had the following composition:—

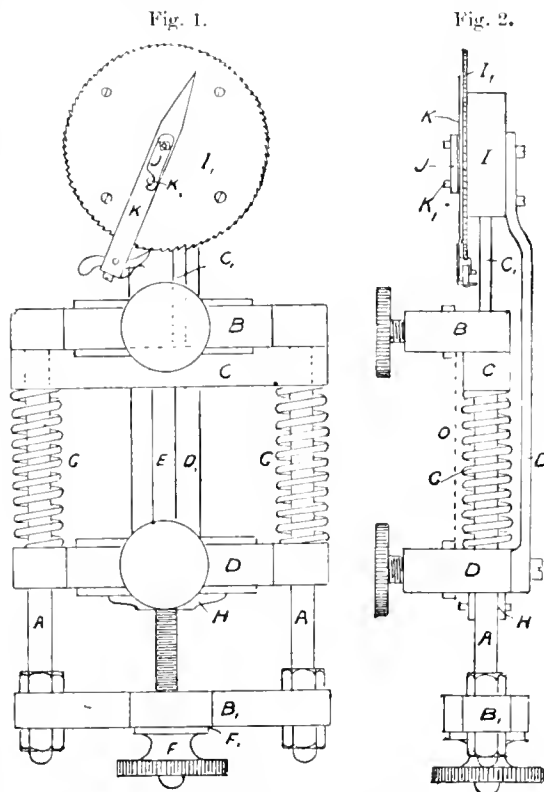
- I. Made of pure rags.
- II. Made of 75 per cent. rags and 25 per cent. sulphite-cellulose.
- III. Made of 40 per cent. rags and 60 per cent. sulphite-cellulose.

The object of the examinations was to find out whether the papers had changed as regards tearing length and elongation at fracture. The result in all cases was that the papers had not changed during the time mentioned. The author points out that, even in the most carefully manufactured papers, no sheet is of an entirely uniform firmness. Therefore slight differences in the tearing length and elongation at fracture are by no means sufficient for conclusions to be drawn on variations in these respects. It has been stated (compare this Journal, 1888, 689) that the firmness of paper sized with resin increases by storage, and that the tensile strength of paper prepared with the addition of sulphite-cellulose is diminished by storage, whilst paper made from rags does not suffer in this respect. The author does not deny the possible correctness of these statements, but thinks they are not proved conclusively and no prejudice based upon the few existing experiments should be raised against a certain class of raw materials. Moreover, the very important influence which moisture has upon the firmness of paper has in many instances been overlooked, and the author feels sure that no comparative tests for firmness are conclusive, unless made under equal or similar conditions of atmospheric moisture.—H. S.

## PATENTS.

*An Improved Portable Machine for Testing the Strength of Paper.* W. Sinclair, Glasgow. Eng. Pat. 3161 February 22, 1889. 8d.

THIS machine is shown in front and side elevation in Figs. 1 and 2. It consists of two bars A, to the ends of which are



fixed two cross-pieces B and B<sub>1</sub>, forming a rectangular frame. Sliding freely on the rods A are two other cross-pieces C and D. C is rigidly connected with the bolt E which passes loosely through the cross-piece D. C and D are kept apart by the spiral springs G surrounding the rods A, and D is kept from B<sub>1</sub> by the stop H fixed on the bolt E. The paper to be tested is clamped to B and D as shown by the dotted line O in Fig. 2. By moving the thumb-screw F the cross-piece C is brought towards B<sub>1</sub>. At the same time pressure is communicated through the springs G to the cross-piece D and therefore to the strip of paper, until the latter breaks. The pressure required to break the paper is recorded by the pointer K on the dial I. The dial is fixed to a box I which is itself attached to the cross-piece D by the piece of metal D<sub>1</sub>. The rod C<sub>1</sub> is fastened to the cross-piece C. As the latter descends, the rod C<sub>1</sub>, which is furnished with a rack, actuates a small pinion connected with the pointer K and causes the latter to move over the dial.—E. J. B.

*Improvements in the Preparation of Tissue Transfer Paper and of Printed Transfers for Transferring Designs on to Pottery Ware.* T. Phoenix, Newcastle-under-Lyme, and G. Kirk, Uttoxeter. Eng. Pat. 4264, March 11, 1889. 8d.

IN many cases it is desirable to print the designs on transfer paper immediately after it has been sized. Hitherto this has not been possible in the case of rolls of paper owing to the thinness of the paper. According to this invention the paper is first sized by leading it over the upper part of a roller, the lower half of which dips into a trough of size,

then passing it over a roller covered with india-rubber, which presses the paper against the engraved printing roller.—E. J. B.

*Improvement in Medicating, Odorising, and Disinfecting Paper.* W. W. Cosley, London. Eng. Pat. 4558, March 15, 1889. 4d.

TEREBENE is incorporated with the paper either in the course of manufacture or subsequently.—E. J. B.

*Improvements in the Treatment of the Recovered Ash of Paper Mills.* J. W. Kynaston, Liverpool, and J. Sutherland, Ballyclare. Eng. Pat. 5779, April 4, 1889. 6d.

IN the manufacture of paper the raw material is heated under pressure with caustic soda solutions. This becomes largely contaminated with silica and alumina from the fibre to the extent of about 15 per cent. of the whole soda present, as well as with various resinous products. The soda solution is thus both reduced in strength, and the impurities introduced are harmful in its further utilisation. The inventors add sodium bicarbonate to the solution converting any caustic soda present into the monocarbonate, the bicarbonate being simultaneously reduced. Under these circumstances the silica and alumina combine and are precipitated, especially on warming. The precipitate may be at once filtered off or removed with the calcium carbonate formed, when the liquors are causticised.—S. G. R.

*A Process for Producing Cupr-ammonia.* C. F. Hime, London, and J. H. Noad, East Ham. Eng. Pat. 7716, May 8, 1889. 4d.

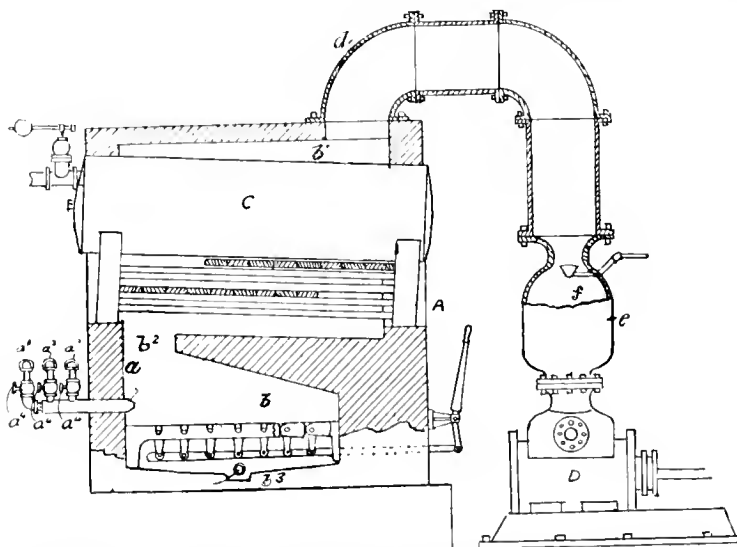
See under VII., page 391.

*Improvements in Machinery for Refining and Finishing Half-Stuff in Paper Making, and in Hydrostatic Balances for Changing the Contents of the Pulp Reservoirs.* F. Faviers fils, Gromelle, France. Eng. Pat. 8509, May 22, 1889. 11d.

UNDERNEATH a beater of the ordinary form, but with the knives somewhat closer together, are placed two tanks with semi-circular bottoms, and provided with agitators. These tanks are connected with each other and with the overhead beater by means of pumps. One of the tanks being filled with half-stuff, the pumps are started and the "half-stuff" is pumped into the beater. It passes under the roll and then into the other chest. If necessary the pulp can be passed again through the beater.—E. J. B.

*Improved Method of and Apparatus for Recovering Chemicals from Solutions, or when mixed with Substances which are Volatilisable or Destructible by Heat, especially adapted for Recovering Soda from Waste Liquors of Pulp Digesters.* M. B. Mason, S. D. Warren, and F. Warren, Boston, U.S.A. Eng. Pat. 20,908, December 31, 1889. 8d.

THE principle of the process is very simple and can best be gathered by a description of the apparatus employed. The liquors to be treated are first evaporated to about 35° B., and then fed into the combustion chamber *b* of the furnace *A* by the pipe *a*<sup>2</sup>, which is in connexion with the pipe *a*. Attached to this latter tube also are pipes *a*<sup>1</sup> and *a*<sup>2</sup> regulated by cocks *a*<sup>1</sup>, by which steam and oil can be blown in respectively. The tube *a* acts as an "atomiser" or pulveriser. The oil is used to heat the chamber in the beginning of the operation; subsequently it can be shut off, and the temperature maintained by the combustion of the carbonaceous matter present in the liquors. The products of combustion pass through *b*<sup>2</sup> into the chamber *b*<sup>1</sup>, in which



is situated the boiler *C*, heated by ordinary tubes. The gases then pass to the condenser *c*, where any particles of soda, mechanically carried over, are washed out by the water-spray *f*, and the wash liquors are removed by the pump *D*. The fused soda in the chamber *b* flows out by the duct *b*<sup>3</sup> to the exterior, and is received in suitable vessels.

—S. G. R.

## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

*Report on New Drugs and Fine Chemicals.* Merck's Bull. 1890 [2], 9.

*Aluminium Aceto-tartrate.*—This salt is prepared in transparent, very faintly yellowish, crystalline granules, soluble in water, but insoluble in alcohol, ether and glycerol. Its value as a therapeutic agent is due to its caustic astringent properties, with which it combines further value as an energetic disinfectant. These properties were fully tested and ascertained in 1885 by Schäffer, of Bremen, and Lange, of Copenhagen. Mixed with boric acid or some indifferent

substance it is very effective in the destruction of nasal polyp (Berliner Klin. Wochenschr. 1889 [30]). A 50 per cent. solution is recommended by Michaelis as a wash for chilblains.

**Cocaine Phenate.**—This is a slightly coloured substance of the consistency of honey, readily melting when heated, and containing about 75 per cent. of the cocaine alkaloid. It is completely soluble in 50 per cent. alcohol, the solution possessing a faint odour of phenol. *Viau* (Nouveau Remède, 1887, 192) has found it, administered hypodermically, a very valuable local anæsthetic in dental operations. Experiments in 86 cases showed that complete topical anæsthesia is produced without any subsequent derangement. *Viau* uses an alcoholic solution of the salt of 0.08 per cent. strength (1 in 1,250). *Von Oefele* has employed cocaine phenate with success in nasal, gastric, conjunctival and other forms of catarrh. He finds also that painting or rubbing with a 1 per cent. solution of the phenate in 30 per cent. alcohol is effective for the treatment of all local pains.

**Salol.**—Löwenthal finds that Koch's "Comma Bacillus," which flourishes on a nutritive basis containing pancreatic juice, is speedily killed in the presence of salol, for the pancreatic ferment brings about the resolution of the salol into salicylic acid and phenol, which completely sterilise such a culture. He proposes large doses as a prophylactic, and larger still as a remedy for Asiatic cholera.

**Sodium Dithiosalicylate**, "No. 11."—(H. Baum, Ber. 22, Part 3 (Patents), 175). Dithiosalicylic acid is a compound of two molecules of salicylic acid connected by a two-atom molecular bond of sulphur—



The substance in question is the sodium salt of one of several isomeric acids. The discoverer distinguishes this particular isomeric acid as "No. 11," and its sodium salt, that under consideration, forms a greyish white and very hygroscopic powder, completely and easily soluble in water. From its solution the dithiosalicylic acid is precipitated in yellow viscid drops on the addition of stronger acids. *Lindenborn* (Frankfort-on-the-Maine) concludes as the result of a series of experiments, that sodium dithiosalicylate No. 11 is preferable to sodium salicylate in articular rheumatism, &c. (Berliner Klin. Wochenschr. 1889, 568). The preference is based on smaller dose required, prompt and reliable action and entire freedom from ill accessory effects. The antiseptic powers of the dithiosalicylate were found by *Hueppe* to be largely superior to those of the single salicylate.

**Zinc Permanganate.**—This salt forms crystalline granules of a violet-brown colour. Like the potassium salt it is very soluble in water. *Berkeley Hill* (Lancet, 1889, June 29th) has used it long and successfully in all forms of *Urethritis*. A special characteristic of the action of the drug was the total absence of any irritation. However, only very dilute solutions are to be used, such as one part of the salt in 4,000 of water. Like the potassium salt, this salt should never be compounded with alcohol, glycerol, sugar, or dry or fluid vegetable extracts, or there may be danger of explosions.

**Oxidation Products of Quinoidine.** H. Strache. Monatsh. 10, 642—646.

**CINCHOMERONIC** acid, which is at the present time obtained by the oxidation of quinine (Weidel and Schmidt's method), can be prepared at a smaller cost from the resinous by-products known commercially as quinoïdine, which are obtained in the preparation of quinine.

For this purpose the quinoïdine, reduced to pieces the size of hazel-nuts, is treated in quantities of 200 grms. with 3 kilos. of commercial concentrated nitric acid, and the mixture boiled for 5 to 6 days until ammonia produces no precipitate in the diluted solution, the acid which evaporates during the process being continually renewed; about 26 kilos. of acid are required for the oxidation of 800 grms. of quinoïdine. After evaporating the acid on the water-bath and repeatedly evaporating with water, a small quantity

(7.3 grms.) of the copper salt of  $\alpha$ -pyridine-tricarboxylic acid separates from the thick syrup in crystals. This salt is separated by filtration, the filtrate diluted with cold water, the solution filtered from the brown resinous precipitate, and then treated with excess of copper acetate. A green precipitate (A) is thus produced, and on standing for several days its quantity increases considerably. The acid filtrate is then evaporated, whereon most of the cinchomeronic acid separates in crystals; on standing for some time two further crops (B) of crystals are obtained, and finally crystals of cinchomeronic acid nitrate separate from the solution. On diluting the mother-liquors with a large quantity of cold water a resinous precipitate is formed at first, then a yellow substance (C) separates, and the filtrate from the latter yields, on addition of sodium acetate, a considerable quantity of a yellowish-green copper salt, which was not further investigated. The filtrate from this salt, after removing the copper with hydrogen sulphide and evaporating, yields a further crop of crystals of the same nature as (B).

The precipitate (A) contains the copper salt of  $\alpha$ -pyridine-tricarboxylic acid, free cinchomeronic acid, and a considerable quantity of a dark yellow amorphous compound. It is first repeatedly extracted with boiling water; the copper salt which remains, in an almost pure condition, is decomposed with hydrogen sulphide and the  $\alpha$ -pyridine-tricarboxylic acid recrystallised from hot water. The aqueous extract is evaporated, the residual cinchomeronic acid crystallised once from water and then dissolved in hot hydrochloric acid; on cooling, the hydrochloride separates in crystals.

The crystals (B) consist essentially of cinchonic acid which can be purified by dissolving in boiling water, precipitating with copper acetate, decomposing the copper salt with hydrogen sulphide, and recrystallising the acid from hot water with addition of animal charcoal.

The precipitate (C) contains a small quantity of cinchomeronic acid, which can be extracted by treating with dilute hydrochloric acid; the residue dissolves readily in hot concentrated hydrochloric acid, and on cooling the hydrochloride of nitroquinolinemonocarboxylic acid separates in crystals. This salt is decomposed on warming with water, yielding the free acid, and when sublimed it gives *La Coste's ana-nitroquinoline* in colourless plates melting at 153°—154°.

The quantities of the various products obtained by the oxidation of 800 grms. of quinoïdine with 26 kilos. of nitric acid, as described above, are as follows:—

73 grms. of  $\alpha$ -pyridinecarboxylic acid = 9.1 per cent., corresponding to 6.4 per cent. of cinchomeronic acid.

48 grms. of cinchomeronic acid hydrochloride = 6 per cent., corresponding to 4.9 per cent. of cinchomeronic acid.

34 grms. of cinchonic acid = 4.3 per cent., corresponding to 4.0 per cent. of cinchomeronic acid.

Total, 15.3 per cent. of cinchomeronic acid.

The quantity of cinchomeronic acid obtained by Weidel and Schmidt's method the author finds to be 44.2 per cent. of the alkaloids employed.

The cost of the materials (quinine, or quinoïdine and nitric acid) for the preparation of 100 grms. of cinchomeronic acid is about 22 M. when quinine is employed, and only about 11 M. when quinoïdine is used, but in the latter case the preparation is more troublesome.—F. S. K.

**Formation of Alkyl Derivatives of Orthohydroxyquinoline.**

E. Leppmann and F. Fleissner. Monatsh. 10, 665—672.

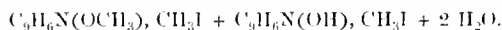
The hydriodide of methoxyquinolinehydroxyquinoline methyl iodide,—



is formed when hydroxyquinoline (1 mol.) is heated under pressure for some hours with methyl iodide (1 mol.) and methyl alcohol. The crystalline mass which is obtained is washed with a little water and then recrystallised from hot water. It forms lemon-yellow needles and melts at 143°. When treated with ammonia or sodium carbonate it is converted into methoxyquinolinehydroxyquinoline methyl iodide,  $C_9H_6N(OCH_3) + C_9H_6N(OH), CH_3I$ . This

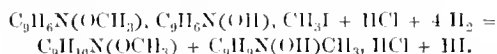


base crystallises from alcohol in orange-red needles and is readily soluble in hot, but only sparingly in cold water, and insoluble in ether. It combines with methyl iodide, when heated therewith under pressure yielding the *methyl iodide* of methoxyquinolinehydroxyquinoline methiodide—



This compound crystallises in yellow needles with two molecules of water, and when treated with silver oxide in aqueous solution it is converted into *methoxyquinolinehydroxyquinoline ammoniummethyl hydrate*,  $C_{18}H_{16}N_2O_3$ .

Kairine is obtained, together with tetrahydromethoxyquinoline, when methoxyquinolinehydroxyquinoline methyl iodide (see above) is dissolved in hydrochloric acid and the calculated quantity of tin added to the solution in the course of about six hours. The reaction which takes place is expressed by the equation—



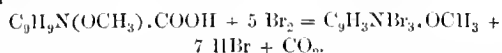
In order to isolate the kairine the tin double salt is separated by filtration, decomposed with hydrogen sulphide, the filtered and concentrated solution neutralised with sodium carbonate and the kairine extracted with ether; the crude reddish-brown product obtained in this way is mixed with resinous substances and changes on exposure to the air. By careful sublimation at  $100^\circ$  in an atmosphere of hydrogen it can be obtained in colourless needles, melting at  $114^\circ$ . The compound prepared in this way is identical with the kairine described by O. Fischer.

The tetrahydromethoxyquinoline formed in the above reaction cannot be isolated.—F. S. K.

#### *Hydroquinoline Derivatives.* O. Sreck. Monatsh. **10**, 701—720.

WHEN quininic acid is reduced with tin and hydrochloric acid it is converted into a tetrahydroquininic acid,  $C_{10}H_{12}NO_3$ , in which all four added hydrogen atoms are probably situated in the pyridine nucleus.

Tetrahydroquininic acid, when treated with bromine, yields tribromoquinanisol,  $C_{10}H_6Br_3NO$  (m.p.  $233^\circ$ ), identical with the compound obtained by Skraup from thalline; the formation of this compound may be represented by the equation—



Tribromoquinanisol is decomposed by hydrochloric acid yielding tribromohydroxyquinoline; both these compounds are formed when thalline is brominated in hydrochloric acid solution.

Tribromoquinanisol and tribromohydroxyquinoline both contain one bromine atom in the pyridine nucleus in the  $\gamma$ -position, and two bromine atoms in the benzene nucleus, because, on oxidation with nitric acid, tribromoquinanisol gives bromonicotinic acid (monobromopyridinecarboxylic acid), whilst tribromohydroxyquinoline yields bromoquinolinic acid (monobromopyridinecarboxylic acid) when it is treated with potassium permanganate.

Bromonicotinic acid, when fused with alkalis, gives two products, neither of which contains bromine; the one is an acid, the other is most probably  $\gamma$ -hydroxypyridine. It follows, therefore, that the bromine atom, both in bromonicotinic acid and bromoquinolinic acid, is in the  $\gamma$ -position. Tetrahydrobromohydroxyquinoline is obtained by the reduction of tribromohydroxyquinoline with tin and hydrochloric acid.—F. S. K.

#### *Phloroglucinol.* Z. H. Skraup. Monatsh. **10**, 721—725.

WHEN commercial phloroglucinol is treated with benzoic chloride and soda, diresorcinoltetrazobenzoate and phloroglucinoltribenzoate are obtained in about equal quantities; they can be separated, but only with difficulty, by repeatedly recrystallising from benzene.

*Diresorcinoltetrazobenzoate*,  $C_{22}H_{12}(C_7H_5O_2)_4$ , crystallises in prisms, melts at  $199^\circ$ , and is readily soluble in hot benzene,

but only sparingly in the cold, although, on cooling, it separates very slowly. In the partial "benzoylation" of commercial phloroglucinol almost the whole of the diresorcinol is contained in the first portions of the precipitate.

*Phloroglucinoltribenzoate*,  $C_6H_3(C_7H_5O_2)_3$ , crystallises in plates and melts at  $173^\circ$ — $174^\circ$ ; it shows the same behaviour as diresorcinoltetrazobenzoate with benzene, and like the latter, is very sparingly soluble in alcohol. It can also be obtained by boiling phloroglucinol with benzoic chloride.

The purity of phloroglucinol can be easily and quickly ascertained, even with only small quantities, by the following method: 0.1 gm. of the sample is treated in a test tube with 5 cc. of 10 per cent. soda, 0.3 cc. of benzoic chloride added, and the mixture shaken until the smell of the chloride is no longer perceptible. The solution is then filtered, the residue dissolved in a little hot benzene, and the solution boiled; if the crystals, which separate on cooling, melt at the right temperature ( $173^\circ$ — $174^\circ$ ) the sample is free from diresorcinol.

The complete purification of commercial phloroglucinol by the methods hitherto employed is not only a very troublesome, but also an uncertain process. When phloroglucinol is warmed with potassium carbonate it is almost immediately converted into the carboxylic acid at  $60^\circ$ — $70^\circ$ , whereas diresorcinol, under the same conditions, undergoes no change and is only slowly acted on even on boiling. When, therefore, Herzig-Zeisel's method of purification is employed, the diresorcinol passes into the ethereal extract, together with phloroglucinol and a little diresorcinolcarboxylic acid, and the smaller the quantity of substance employed, the larger the quantity of diresorcinol dissolved. By making use of Will's suggestion and extracting with ether before acidifying, this error can be eliminated, but the process becomes more troublesome.

The author finds that the purification of commercial phloroglucinol is best carried out as follows: One part of the finely divided sample is dissolved in one part of boiling water, the solution cooled quickly to  $60^\circ$ — $70^\circ$  in a water-bath, and then two parts of finely divided potassium bicarbonate added, the first half in small quantities at a time because of the effervescence, the second half all at once. The mixture is kept in the water-bath until it ceases to crystallise, then mixed with 6—8 parts of warm alcohol, shaken well and allowed to cool completely; it is advisable to keep the flask filled with coal-gas while it cools. The portion insoluble in alcohol consists of a mixture of potassium carbonate and the potassium salt of phloroglucinolcarboxylic acid, which is very sparingly soluble in alcohol or in potassium carbonate solution; the diresorcinol remains unchanged and dissolves readily in the alcohol. The solution is therefore filtered, the residue washed first with alcohol, then with alcoholic ether, and after drying for a short time in the air, triturated with a well-cooled mixture of two parts of concentrated hydrochloric acid and two parts of water until the evolution of carbonic anhydride is at an end. The solid phloroglucinolcarboxylic acid is then filtered off, washed with a little cold water and boiled with  $1\frac{1}{2}$  parts of water until the whole has dissolved, and the evolution of carbonic anhydride ceases. The pure crystalline product, which is deposited on cooling, is almost colourless and amounts to 50—60 per cent. of the substance employed. A further quantity can be obtained by evaporating the mother-liquors, and from the aqueous filtrate from the carboxylic acid.

In this way phloroglucinol can be easily prepared in a pure condition in the course of two hours; the method is especially convenient for the purification of small quantities, but it can doubtless be employed for the treatment of large quantities as the fact that ether is not used in the process compensates for the slight amount of loss.—F. S. K.

#### *Kynurine.* Z. H. Skraup. Monatsh. **10**, 726—731.

It has been previously shown that kynurine (hydroxyquinoline) is formed in the oxidation of cinchonine and cinchoninic acid; it is also produced in relatively large quantities in the oxidation of cinchonidine, whilst quinine yields neither kynurine nor any analogous compound.

The preparation of kynurine from cinchoninic acid is described below. The process is rather troublesome, but as the yield is about 10 per cent. of the weight of the acid employed, it is more convenient than that by which kynurine was first obtained, namely, from the urine of the dog.

A solution of 50 grms. of anhydrous cinchoninic acid, 20 grms. of chromic acid, and 30 grms. of concentrated sulphuric acid, in about 200 cc. of water, is evaporated on the water-bath until it becomes deep brown, and the heating is then continued until the colour changes to green, the water lost by evaporation being continually renewed. This operation occupies about 20 hours. The green solution is then mixed with a large volume of water, and allowed to cool completely, whereon the unchanged cinchoninic acid is deposited. After further concentration, a second crop of this acid is obtained. The solution is then poured into excess of cold potash, the organic salts separated from the chromium hydroxide and inorganic salts in the usual manner, and the aqueous solution freed from potassium sulphate by mixing with alcohol. Copper sulphate is then added as long as copper cinchoninate is precipitated, and the filtered solution slightly acidified with hydrochloric acid. After standing for some time, the kynurine separates as a basic salt in long needles; the weight of this product is about 4 grms. The mother-liquors, which still contain cinchoninic acid and kynurine, are evaporated, the inorganic compounds separated, and, after precipitating the cinchoninic acid with copper sulphate, the kynurine precipitated with soda.

The kynurine obtained by the oxidation of cinchoninic acid is identical with the compound obtained from cinchonine and from kynurinic acid.

When kynurine is boiled with potash and ethyl iodide it yields a quaternary iodide and two kynurine ethyl ethers; when treated with phosphorus oxychloride and phosphorus pentachloride at 100°—110° it is converted into a chloroquinoline which melts at 34°, and is not identical with  $\alpha$ -chloroquinoline.—F. S. K.

**Codeine Methylolide.** Z. H. Skraup and D. Wiegmann. *Monatsh.* **10**, 732—733.

WHEN codeine methylolide is heated with alcoholic potash it yields ethyldimethylamine and a small quantity of trimethylamine, but not dimethylamine, as stated by Knorr. This fact is in accordance with the authors' view, that the nitrogen atom in morphine is directly combined both with a methyl- and an ethyl- group. The two bases obtained in this way can be easily separated by fractional crystallisation of their platinichlorides, the double salt of ethyldimethylamine separating in compact octohedra, which melt at 193°, and are very readily soluble in water, but only sparingly in alcohol.

Numerous experiments on the behaviour of various samples of morphine and codeine methylolide with alcoholic potash show that the two compounds exhibit no noteworthy difference in behaviour, and give no grounds for doubting that morphine is a distinct chemical compound.

—F. S. K.

**Methysticine.** C. Pomeranz. *Monatsh.* **10**, 783—793.

CUZENT, in 1861, obtained a crystalline neutral substance, which he named "Kawain," from the roots of *Macropiper methysticum*, one of the piperaceæ indigenous to the Polynesia, and termed Kawa-Kawa or Ava-Ava by the natives, by whom it is employed for the preparation of an intoxicating beverage. At about the same time Goble and Rorke obtained from the kawa-root, in the same way, a substance which, though resembling kawain in its properties, they named methysticine.

Methysticine,  $C_{15}H_{11}O_5$ , was prepared by the author from kawa-root according to the method described by Cuzent (*Compt. Rend.* 1861, 205) and Goble (*Jour. Pharm. Chim.* 1860, 598). The powdered root is extracted with 80 per cent. alcohol, the alcoholic solution concentrated, and then kept for some time in a cool place; the crystals which are deposited are separated, the mother-liquors concentrated again in order to obtain a further small quantity of crystals, and the product is then purified by repeated recrystallisation

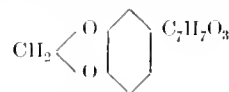
from 70 per cent. alcohol with the addition of animal charcoal. It is thus obtained in colourless, tasteless, silky needles, which melt at 137°. It is insoluble in cold water, and only sparingly soluble in hot water, light petroleum, and ether, but more readily in alcohol, benzene, and chloroform, and most readily in boiling alcohol. It decomposes when heated, giving off aromatic vapours; when treated with concentrated sulphuric acid it turns purple-violet. It is insoluble in cold potash or soda, but on boiling it gradually dissolves, and crystals of a salt of methysticine acid separate from the solution on cooling.

**Methysticine Acid**,  $C_{14}H_9O_5$ , is best prepared by boiling 10 grms. of methysticine for 20 minutes with 300 cc. of 6 per cent. potash and acidifying the cold solution with acetic acid; the precipitate is washed with water and crystallised twice from boiling 90 per cent. alcohol with the addition of animal charcoal. Five grms. of the acid are obtained in this way. It forms yellowish, silky needles, very like the crystals of piperinic acid in appearance, and melts at 180° with evolution of carbonic anhydride. It is much more sparingly soluble in hot alcohol than methysticine, and it is only sparingly soluble in chloroform, ether, and benzene, but it dissolves freely in alkalis and alkaline carbonates; its alcoholic solution gives a red colouration with ferric chloride.

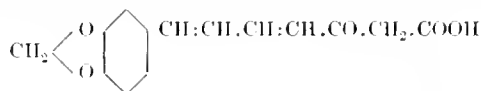
If methysticine is boiled with alkalis for more than about 20 minutes the solution becomes turbid, owing to the decomposition of the acid, one of the products being piperonal. When methysticine acid (4 grms.) is dissolved in 200 cc. of 3 per cent. potash, the solution heated to boiling, and then 200 cc. of a 3 per cent. solution of potassium permanganate gradually added, the acid is rapidly oxidised and a strong odour of piperonal is observed. When the filtered solution is distilled a small quantity of a crystalline substance passes over; this compound melts at 37°, and possesses the odour of piperonal, with which it is most probably identical. On acidifying the residual solution (about 60 cc.) with hydrochloric acid, piperonylic acid is precipitated; the yield of this product is 0.7 gm. from 4 grms. of methysticine acid.

A compound of the composition  $C_{13}H_{12}O_3$ , which the author names *methysticol*, is obtained when methysticine is boiled with 40 parts of 4 per cent. hydrochloric acid for 15 minutes, or when methysticine acid is boiled with dilute mineral acids. It crystallises in flat prisms, melts at 94°, and is insoluble in alkalis; it is not acted on by acetic anhydride at 160°, but it combines with phenylhydrazine, yielding a hydrazide which melts at 143°.

The author's experiments show that methysticine is the methyl salt of methysticine acid, and that this acid has the constitution—



Judging from its chemical behaviour, which resembles closely that of a  $\beta$ -ketone acid, methysticine acid is probably a piperinyllactic acid of the constitution—



—F. S. K.

**The Compounds of the Volatile Fatty Acids with Phenols.**  
M. Nencki. *Monatsh.* **10**, 906—907.

THE author prepares the oxyketones containing a fatty acid and phenol radiæly by heating the phenol and fatty acid with zinc chloride. He has prepared in this way the acetyl phenols of resorcinol, orcinol, quinol, and pyrogallol. Formic acid and phenol under similar conditions yield leucaurin. Propionic acid, butyric acid, and valeric acid also combine with phenols in presence of zinc chloride. Propionyl phenol is obtained by heating 2 parts of zinc chloride dissolved in 1 part of propionic acid, adding 1 part

of phenol, and boiling for 5–10 minutes. On oxidation with fused potash, it yields *p*-hydroxybenzoic acid. The propionyl group occupies, therefore, the para-position to the hydroxyl group.—J. B. C.

*Frangulin.* T. E. Thorpe and H. H. Robinson. J. Chem. Soc. **57**, 38–50.

This body has been previously investigated by several authors, but the statements concerning it differ considerably. The authors prepared it from the bark of the alder buckthorn (*Rhamnus frangula*) by extracting it, first with low-boiling petroleum, which dissolves only fat and chlorophyll, and then with methylated spirit, which dissolves the frangulin and other matter. The methylated spirit extract deposited a quantity of resin on standing for two days. The tannin was removed by lead acetate, and the excess of lead by sulphuretted hydrogen. The clear solution after this treatment, on standing for five weeks, deposited some frangulin. The mother-liquor was evaporated and mixed with calcium sulphate in order to form a powder suitable for extraction with ether. This extract deposited more frangulin. Altogether 0.09 per cent. was obtained.

It is an orange-yellow crystalline powder, very slightly soluble in cold alcohol and not much more soluble in hot alcohol. Melting point 225°.

The mean of seven closely-agreeing analyses of different specimens dried at 120° gave 61.20 per cent. carbon and 5.29 per cent. hydrogen. If the formula of the body be  $C_{20}H_{22}O_9$ , it would contain 61.40 per cent. of carbon and 5.11 per cent. of hydrogen.

It was hydrolysed by boiling with hydrochloric acid in alcoholic solution for 3–4 hours. On boiling off the alcohol and diluting with water an orange-yellow precipitate was separated. This amounted to 67.6 per cent., and was found to be emadin ( $C_{15}H_{10}O_5$ ). There then remained in solution a body possessing the power of reducing Fehling's solution. A phenylhydrazine derivative was prepared by evaporating the solution to dryness with sodium acetate and phenylhydrazine hydrochloride (cf. Beythien and Tollens, Ann. **225**, 217–221; this Journal, 1890, 310). The residue, insoluble in water, was dissolved in alcohol, and precipitated by water; it had a melting point of 158°, and was not phenylglucosazone.—A. L. S.

*Norwegian Oil of Carraway.* C. Nicolaysen. Chem. Zeit. **13**, 1704.

THE carraway is one of the most widely distributed plants in Norway, growing as far north as 71° of latitude, and, in Southern Norway, up to 1,098 m. above the sea level. The author has examined the seeds of Norwegian carraway by passing steam through 5 kilos. of the bruised seeds contained in an iron still, obtaining 6.1 per cent. and 6.4 per cent. of oil in two experiments, but of this a considerable proportion was contained in the aqueous distillate, which had to be treated with ether to recover the dissolved oil. Carraway oil consists of carvene and carvol, the latter being the most valuable. Three samples gave, by fractional distillation, 48.9, 47.1, and 48.0 per cent. of carvol respectively, which is less than the average continental oil containing about 64.5 per cent. of carvol and 35.5 per cent. of carvene.—G. H. B.

*Dilution Tables for Practical Use.* F. J. Herz. Chem. Zeit. **14**, 3–4.

IN order to avoid troublesome calculations, it is proposed to have dilution tables for acids, lyes, and solutions of salts, by means of which the amount of diluent required may at once be found. As an example, spiritus dil., of the German Pharmacopœia, contains 60 to 61 per cent. (by weight) of ethyl alcohol; in order to dilute 100 grms. of 86 per cent. alcohol to the above strength, 41 to 43 cc. of distilled water has to be added. 1,000 grms. of 90 per cent. alcohol give with 475 cc. of water 61 per cent., and with 500 cc. water 60 per cent. alcohol. Tables for alcohol and for ammonia are given.—N. H. J. M.

*Iodine Cotton-Wool.* Bréaudet and Cathelineau. Rép. de Pharm. **45**, 529.

ETHEREAL and alcoholic iodine solutions, when brought into contact with the skin, irritate and even cauterise, whilst the absorption of iodine is limited. Good results are obtained by using iodine cotton-wool, from which the iodine is liberated by very gentle heat. It is prepared as follows:—Carefully cleaned cotton-wool is put for a few minutes into 2 per cent. aqueous sodium carbonate, washed and then kept for half an hour in 4 per cent. bleaching powder solution. It is washed until free from alkali and kept for a quarter of an hour in water acidified with 5 per cent. hydrochloric acid; it is again washed and dried. Finely powdered iodine (8 parts) is strewn over the sheets of cotton-wool (100 parts), which is then tightly rolled together and heated in a glass bottle until iodine vapour is given off. The bottle is then stoppered and heated for two hours in a water-bath. The iodine cotton-wool is kept in well-closed glass vessels.—N. H. J. M.

*The Poisonous Constituents of Castor-Oil Seeds.* H. Stillmark. Pharm. Central-halle, **30**, 650.

A DIASTASIC ferment called "*Ricin*" is extracted from castor-oil seeds as follows:—Fresh, shelled seeds are ground to a powder and agitated with a 10 per cent. solution of sodium chloride. The solution is filtered, saturated at the ordinary temperature with magnesium sulphate and sodium sulphate at the same time, and then allowed to stand. Large crystals of the two sulphates separate out, and in addition a white amorphous precipitate which can be readily separated from the crystals and collected. This is the substance ricin. It is purified by repeated dialysis at a low temperature and then dried in vacuum over strong sulphuric acid. Thus prepared it forms a white friable powder, free from taste and smell and very poisonous; it still contains 10–20 per cent. of sulphates. It loses its toxic properties when boiled, but keeps perfectly in the substance. A 10 per cent. solution of sodium chloride is the best solvent for it. It appears that similar poisonous albuminoid bodies exist in other species of *Recinus*, and also in the seeds of *Croton tiglium* and of *Jatropha caracas*.—C. A. K.

*A New Antipyretic.* R. Kobert. D. Medecin. Wochenschr. 1890, No. 2.

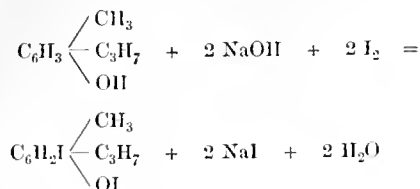
THE name *Orthin* is given by the author to orthohydrazine-para-hydroxybenzoic acid ( $\text{OH:N}_2\text{H}_3\text{:CO.OH} = 1:2:4$ ). The free base is very unstable, but the hydrochloride is a stable body which reduces the per-salts of the heavy metals and which possesses a marked antiseptic action. Experiments with animals have shown that it can be administered without injurious results, and that a single dose suffices to render the body proof against the action of reducing agents for weeks. It is probable that orthin hinders the processes of oxidation going on in the system, and that it reduces the temperature by increasing the ease with which heat is given up by the body, by extending the pores of the skin. Solutions of 1 to 10,000 of orthin had no injurious effect on the heart of a frog. Para-hydrazine salicylic acid has a similar physiological action to its isomer, orthin. The author does not consider that the latter is likely to be much employed as an antipyretic.—C. A. K.

#### PATENTS.

*New or Improved Manufacture of Periodides of Phenols and Salicylic Acid.* B. Wilcox, London. From "The Farbenfabriken vormals F. Bayer and Co.," Elberfeld, Germany. Eng. Pat. 5079, March 23, 1889. 6d.

NEW iodine substitution products, or periodides of phenol, cresol, resorcinol, thymol,  $\beta$ -naphthol, and of phenol carboxylic acids result by the treatment of the aqueous alkaline solutions of these bodies with a solution of iodine in an alkaline iodide. According to the nature of the phenol or of the phenol carboxylic acid, the amount of iodine

entering into reaction varies; but in all cases the hydrogen of the hydroxyl group is replaced by iodine, *e.g.*, in the case of thymol:—



One-half of the iodine is apparently lost in this process, but the filtrate from the iodo-thymol iodide is again treated with an alkaline solution of thymol and the iodine in the sodium iodide liberated by the addition of bleach or of other hypochlorites. The iodo-thymol iodide is a brown-red amorphous powder, which gradually loses iodine in the air, readily when heated to form mono-iodo-thymol; it melts with decomposition at 110° C. The red colour of this product is removed by treatment with sodium hyposulphite; it is insoluble in water, soluble in alcohol and ether. The other phenol iodides described possess similar properties. Those of salicylic and cresol carboxylic acids are odourless and insoluble in alkalis, that of  $\beta$ -naphthol is an amorphous green yellow precipitate, insoluble in water and readily reconverted into  $\beta$ -naphthol. Details for the preparation of the thymol and  $\beta$ -naphthol compounds are given.

—C. A. K.

*A Process for the Production of a Substance to be called Chloral Formamide or Chloralamide.* J. F. von Mering, Strasburg, Germany. Eng. Pat. 7391, May 3, 1889. *Ad.*

By warming chloral and formamide together in molecular proportions, chloral formamide ( $\text{C}_2\text{H}_3\text{Cl}_3\text{COH}\cdot\text{COH}\cdot\text{NH}_2$ ) is obtained. The recrystallised product forms colourless crystals with a mild and slightly bitter taste, and without smell; soluble in water, alcohol, ether, &c., and melting at 115°–116° C. It is more stable than chloral, but acts like the latter as an hypnotic or soporific, and as an antiseptic. It has no corroding action, does not affect the action of the heart, and does not disturb the digestion.—C. A. K.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

*"Flash Powder" Explosions.* C. L. Mitchell. Phot. News, 34, 191–192 and 225–226.

THE author calls attention to the great danger attending the use of mixtures of magnesium powder with picric acid, potassium chlorate, &c. The substances which have been employed in conjunction with magnesium are potassium ebhorate, nitrate, dichromate, and permanganate, amorphous phosphorus, picric acid, sulphur, antimony sulphide, and possibly some others. Five deaths have recently been caused in America by the explosion of one of these powders either in the process of mixing, or, in one case which caused three deaths, whilst being handled 18 months after preparation. Picric acid was one of the chief constituents of this powder, and the excessive explosibility may have been due to the presence of sodium or potassium picrate, which is frequently found in the commercial acid. In the case of the powder which had been kept for some time there was evidence that magnesium picrate had been formed by the gradual action of the acid on the metal, an action doubtless assisted by the hygroscopic nature of the powder.—C. H. B.

*Photo lithographic Ink containing Gum Elemi.* K. Kampmann. Phot. Correspondenz, 26, 564–568.

ASPHALT, 20 parts, and colophony, 100 parts, are dissolved in turpentine, 40 parts, and mixed with yellow wax, 20 parts, suet, 140 parts, gum elemi, 230 parts, Marseilles soap, 30 parts, linseed oil varnish (medium strength), 60 to 80 parts, finest lamp black, 80–100 parts. Instead of the soap, 20 parts of Venice turpentine and 40 parts of good transfer ink may be used.

The materials must be well melted together and very thoroughly mixed, an operation requiring much care and practice. The materials should be as free from water as possible, the vapours should not be allowed to burn, and towards the close of the operation the mixture should be heated in an uncovered vessel until it is completely melted.

This ink is especially valuable when working on zinc, but works equally well on stone.—C. H. B.

*Intensification of Collodion Negatives by Means of Quinol.* v. Huhli and Eder. Phot. Correspondenz, 27, 14–16.

TWO solutions are required:—(A.) Quinol, 10 parts, citric acid, 6 parts, water, 1,000 parts; and (B.) Silver nitrate, 1 part, water, 30 parts. One volume of B. is added to 3 vols. of A., and the mixture poured on the unfixed collodion negative. Not only is the existing image intensified, but development is extended, and new details become visible.

The intensifier can also be used after the negative has been fixed, provided that comparatively little intensification is required.—C. H. B.

*New Positive Printing Process.* Amer. Am. Phot. 1890, 232.

PAPER is coated with an emulsion containing ferric oxalate 7 parts, silver nitrate 7 parts, gelatin 7 parts, tartaric acid 3 parts, and water 128 parts. The images are printed out, have a pleasant brown colour, and are fixed in the usual way.—C. H. B.

*Ferric Oxalate as a Reducer.* L. Belitski. Phot. Wochenblatt, 16, 71–73.

TEN parts of ferric potassium oxalate, 8 parts of normal sodium sulphite, 2.5 to 3 parts of oxalic acid, and 50 parts of sodium thiosulphate are dissolved in this order in 200 parts of water and the solution filtered and kept in the dark. It can be kept unaltered for a long time in closed vessels, but becomes yellow and deposits sulphur on prolonged exposure to air. When brought in contact with a negative part of the silver is dissolved and ferric oxalate is reduced. The ferrous oxalate formed takes up oxygen from the air and thus the activity of the reducer continues for a long time.

—C. H. B.

## PATENT.

*Improvements in and in Connection with Photographic Printing.* W. W. J. Nicol, Birmingham. Eng. Pat. 5374, March 29, 1889. 6d.

PAPER, wood, or any other surface is sensitised with a ferric salt, with a salt of an organic acid which prevents precipitation of the iron by ammonia. It is then exposed to light under a negative, &c., until a faint image is visible, and is developed by treating it with an ammoniacal solution of silver nitrate containing a salt of an organic acid. The print is washed with water containing ammonia and an organic salt, and finally with pure water. A solution (1) containing 20 per cent. of ferric sodium citrate and 5 per cent. of potassium oxalate gives very good results. A solution (2) containing 5 per cent. of normal ferric oxalate, 5 per cent. normal ferric tartrate, and 1 per cent. of tartaric or oxalic acid also works well, but the developing solution requires modification in order to prevent precipitation of the iron.

If necessary the sensitising solution may be thickened with gelatin, starch, &c. Paper sensitised with solution (1) may be developed with one of the following solutions: (a) for cold blue-black tones, 20 per cent. of potassium oxalate, 1.5 per cent. silver nitrate; (b) neutral black

tones, 10 per cent. of potassium oxalate, 1.5 per cent. silver nitrate; (c) *sepia tones*, 7 per cent. borax, 1.5 per cent. silver nitrate. In all cases the precipitated silver salt is nearly redissolved by adding ammonia.

If the paper has been sensitised by solution (2) the following developers may be used: (a) 15 per cent. potassium citrate, 10 per cent. sodium acetate, 1.5 per cent. silver nitrate; or (b) 10 per cent. of potassium oxalate in place of sodium acetate. In both cases the precipitate is redissolved by ammonia. The first series of developers may also be used, but in this case the print after development must be immersed for a short time in a clearing solution containing 20 per cent. of an alkaline citrate or tartrate made alkaline with ammonia. The developed prints are first washed in a solution prepared by mixing a 25 per cent. solution of an alkaline citrate or tartrate with an equal volume of strong ammonia solution, and diluting 150 cc. of this mixture up to 10 litres with water. They are afterwards washed with water and dried.

Development and the other operations are conducted at the ordinary temperature. The process is applicable to fabrics as well as to glass and paper.—C. H. B.

## XXII.—EXPLOSIVES, MATCHES, Etc.

### PATENTS.

*Improvements in the Preparation of Explosive Compounds.*

A. V. Newton, London. From A. Nobel, Paris, France. Eng. Pat. 4479, March 14, 1889. 6d.

This invention consists in incorporating with nitroglycerin and nitrocellulose an acetic ether of glycerin or analogous non-volatile solvent for the purpose of producing explosive compounds of determinate degrees of explosive sensitiveness.

It has been found that explosive compounds produced according to Eng. Pat. 1471 of 1888 (this Journal, 1889, 214), alter on keeping owing to the volatilisation of the camphor. This defect is now obviated by the use of the non-volatile tri-acetin. The explosive made according to this specification when used for blasting purposes may either be fired by a spark or fuse or ordinary detonator, according as a slow or quick action is desired, and it is found advantageous to compress it into cartridges, the grains readily adhering together when slightly warm, or after having been moistened on their surface with a solvent such as acetate of amyl, and then subjected to gentle pressure.—W. M.

*Improvements in and connected with the Manufacture of Matches, and in Machinery or Apparatus therefor.*

C. R. E. Bell, Wandsworth. Eng. Pat. 5208, March 26, 1889. 11d.

This is an invention for a "continuous process of match making by means of an automatic machine, into which the veneers are fed, and by means of which the veneers are cut into splints, the splints inserted into grippers carried by an intermittently rotating drum and tipped and dried through the rotation of the drum and the matches delivered in a shoot or boxes." Numerous drawings and detailed description are given in the specification.—W. M.

*Improvements in the Manufacture of Explosives.* C. O. Lundholm and J. Sayers, Stevenston. Eng. Pat. 6399, April 15 1889 4d.

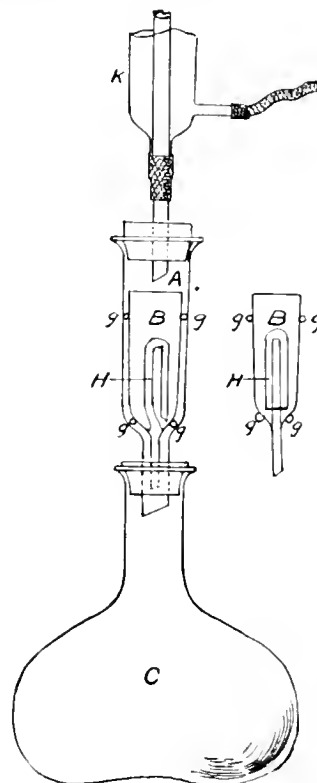
The principal feature in this invention is the employment of suitably nitrated hydro- or oxy-cellulose instead of the usual nitrated celluloses. The nitrated substance is subsequently treated with camphor or with camphor in solution for the purpose of consolidating the explosive, and the treatment is varied so as to obtain different densities of grains, and consequently different rates of combustion. The nitrated substance may also be mixed with nitroglycerin and nitrates, chlorates, picrates, or picric acid.—W. M.

## XXIII.—ANALYTICAL CHEMISTRY.

*Extraction Apparatus.* O. Knöfler. Zeit. Anal. Chem. 28, 671—672.

The author describes with the aid of a diagram a form of extraction apparatus, which has the advantages of being cheaper than, and not so fragile as the ordinary Soxhlet's extractor, whilst the solvent action takes place at the boiling point of the liquid employed.

The substance is placed in a small cylindrical glass vessel B, which is open above but closed below; through the



lower conical extremity of B passes a U-shaped syphon H. The vessel B is surrounded by a cylinder A, the two being kept a short distance apart by small glass knobs *g g* on the outside of B; the lower extremity of A is drawn out and passed through a cork into the flask C containing the solvent, the upper extremity being closed with a cork, through which projects the tube of an ordinary Liebig's condenser K.—F. S. K.

*Apparatus for Fractional Distillation under Reduced Pressure.* E. Valenta. Zeit. Anal. Chem. 28, 673—676.

The author describes with the aid of diagrams an apparatus for distilling under reduced pressure, suitable more especially for substances which are solid at the ordinary temperature.

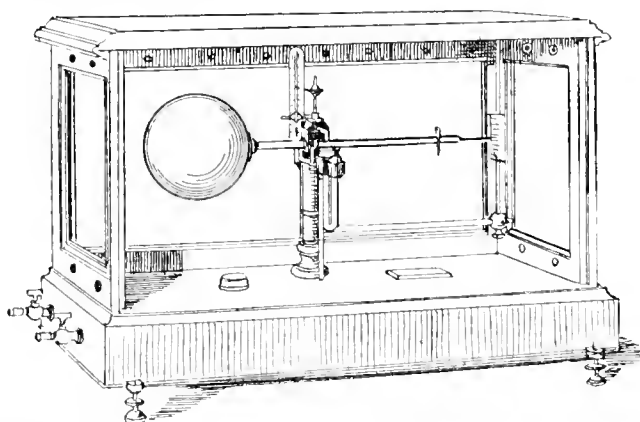
The substance is heated in a fractionating flask, provided with a short fractionating column which is connected by means of an adapter to an ordinary Liebig's condenser, kept at from 40° to 80° by a stream of warm water. The cylindrical receiver is funnel-shaped below, a three-way stop-cock being situated on the stem of the funnel, which serves to connect the receiver with a small flask or test-tube in the usual way. The cylindrical receiver is kept at a suitable temperature by means of a hot-water jacket.—F. S. K.

*A New Gas Balance.* F. Lux. *Zeits. Anal. Chem.* **29**, 13—18.

THIS is the latest and most improved form of the author's gas balance originally described in the *Zeits. Anal. Chem.* **28**, 38 (also this Journal, 1888, 95; and 1889, 668). It is known as the form C.E.

The balance is fitted into a glass case, provided with levelling screws and a hinged window in the front (not shown in the Figure). The central support is a hollow brass

cylinder with a forked top, which latter carries the agate planes of the pivot of the balance, these being fixed in suitable grooves. On either side of these grooves are carriers for the ivory cups, which are filled with mercury and act as lutes for the entrance and exit of the gas. Screws are attached to these carriers, to allow a thermometer and manometer to be fixed on when required, so that the temperature and pressure of the gas can be taken (shown in the Figure). The gas supply and exit pipes are attached just below the carriers referred to. Inside the central



column is placed the adjustment for bringing the balance to rest; the method employed is described as the "cylindrical adjustment." A mill-headed screw placed above the axis of the beam serves to free or to arrest the latter. The other essential parts of the balance are the hollow brass sphere which is filled with the gas and a six-sided beam with a divided scale and a serrated edge to accommodate a rider. At the end of this beam a pointer is attached reaching to the ivory scale shown in the Figure; the beam is divided into 10 parts, and each part again into tenths (0.1, 0.2, &c.). The scale is divided into 50 parts in a similar way, with the zero point in the centre and the signs + and - above and below the zero point respectively.

To use the balance the case is first levelled, the ivory cups of the main support filled with mercury, and then the beam placed in position and screwed firmly down so that it cannot move. The rider (which is made of nickel) is then placed on division 1 of the beam and the beam released. The pointer should rest at the zero of the ivory scale. If it does not, adjustment can be made by a suitable screw. The rider is then placed in a second position on the beam, so as to see whether each one division on the beam corresponds to one division on the scale. This done the beam is fixed in position and the gas to be tested passed in. Under a pressure of 25 mm. of water five minutes is ample to completely displace all the air in the sphere. The rider is then placed on a suitable division (0.4 for coal gas) and the beam released. The reading on the beam + or - the reading on the scale gives at once the specific gravity of the gas. The 25 divisions on the scale give a range of from 0.15 to 0.65 in specific gravity, so that without changing the position of the rider the specific gravity of the gas is got at once, except in very exceptional cases. The author considers this new form of gas balance just as convenient and considerably more accurate than the older form (form C.).—C. A. K.

*Drying of Gases.* J. D. van der Plaats. *Rec. trav. Chim.* **6**, 45.

DEHYDRATED calcium chloride absorbs water better than the salt containing two molecules of water of crystallisation, but the difference at low temperatures is slight. Calcium chloride dried at 180° always contains caustic lime, which cannot be reconverted into a calcium salt by prolonged treatment with hydrochloric acid or with carbon dioxide as long as it remains dry. Caustic lime only absorbs moisture slowly, and leaves twice as much water in the gas to be dried as the calcium chloride with two molecules of water of crystallisation does. Fused caustic potash acts more rapidly and more efficiently than calcium chloride, but less so than sulphuric acid. Phosphorus pentoxide is the best drying agent. According to Stas, it can be freed from phosphorous anhydride by distillation in a current of dry air. Sulphuric acid is the most convenient substance for drying gases, and removes the moisture completely, even from a rapid current of gas. Its efficiency is greatest at low temperatures. It should be free from sulphur trioxide, and to make quite sure on this point it is advisable to use acid containing 6—8 per cent. of water. Carbon dioxide is kept back to some extent by strong sulphuric acid, but is readily driven out again by a current of air. In using pumice soaked in sulphuric acid for drying purposes, the author recommends that the pumice should be first boiled with a mixture of strong sulphuric acid and a little nitric acid, so as to remove chlorides, fluorides and oxides likely to absorb oxygen.—C. A. K.

*A Rapid Method for the Detection and Estimation of Chlorine in Alkaline Sulphocyanides.* C. Mann. *Zeits. Anal. Chem.* **28**, 668—669.

WHEN a stream of hydrogen sulphide is passed through a solution of an alkaline sulphocyanide mixed with copper sulphate, the colourless copper sulphocyanide which is precipitated at first is only slowly converted into black copper sulphide. When, however, more copper sulphate is present than is necessary for the formation of copper sulphocyanide, and the current of gas is stopped just when the solution begins to turn brown (*i.e.*, when the formation of copper sulphide commences), after adding more copper sulphate to combine with the hydrogen sulphide in solution and to precipitate the free hydrogen sulphocyanide, and filtering, the filtrate will be free from sulphocyanides. The precipitate can be rapidly separated by filtration, and the solution shows

*Impurity in Commercial Barium Carbonate.* R. Weysscheider. *Zeits. Anal. Chem.* **29**, 20.

THE author found traces of zinc and iron in a sample of commercial barium carbonate. Fresenius found a considerable quantity of manganese in another sample.

—C. A. K.



no tendency to pass through turbid, even after repeated washing.

The presence of very small quantities of chlorine (or bromine) can then be detected by acidifying the filtrate with nitric acid and adding a few drops of silver nitrate solution.

For estimating the chlorine, 5 grms., at the most, of the sulphocyanide are dissolved in 100 cc. of water, and the solution mixed with 20 grms. of copper sulphate in 100 cc. of water; hydrogen sulphide is then passed until the solution begins to turn brown, then 8 grms. of copper sulphate dissolved in 40 cc. of water are added, and the mixture stirred well, and filtered.

Quantitative experiments with mixtures of ammonium sulphocyanide and ammonium chloride gave very good results. Commercial ammonium sulphocyanide rarely contains more than a trace of chlorine.—F. S. K.

*Estimation of Copper by Converting Copper Sulphide into the Oxide.* C. Holthof. Zeits. Anal. Chem. **28**, 680—681.

WHEN freshly-precipitated and moist copper sulphide, in quantities of 0.18—0.22 gm., is heated in a porcelain crucible, it commences to glow, and is converted into copper oxide. After heating for 7 to 10 minutes over a good Bunsen burner, the change is complete, no further decrease in weight takes place, and the residue is completely free from sulphuric acid.—F. S. K.

*Volumetric Determination of Sulphuric Acid.* A. Gawalowski. Zeits. Anal. Chem. **29**, 19.

THE author, in reply to adverse criticisms of his method for the volumetric determination of sulphuric acid (this Journal, 1888, 455), says he has reviewed his data carefully, and finds the method, as originally described, trustworthy as long as all details are carefully attended to.—C. A. K.

*Analysis of Chrome Iron Ore.* R. Fresenius and E. Hultz. Zeits. Anal. Chem. **29**, 29—35.

A SAMPLE of chrome iron ore which was finally shown to contain 61 per cent. of chromium, 28 per cent. of iron, and 9 per cent. of chemically combined carbon and graphite, and which was hardly attacked by acids, was analysed by the following method:—

1. *Determination of Metals and Gangue.*—About 5 grms. of the ore are heated in a boat in a current of dry chlorine. The boat is placed inside a combustion tube, attached to one end of which are a number of Peligot's flasks and Woulff's bottles partially filled with water, for absorbing the volatilised chlorides. After 3—4 hours' chlorination, the absorbing vessels are removed, and the chlorine in the tube displaced by dry carbon dioxide. The contents of the boat and any sublimed chromic chloride in the tube are reheated and a current of hydrogen passed through the tube, to reduce any chromic to chromous chloride and render it soluble in water; a fresh Peligot's flask is attached on passing the hydrogen. The boat is withdrawn on cooling, and its contents extracted with water; manganese chloride and any chloride of iron or of chromium is dissolved, whilst the carbon, gangue and undecomposed ore remain behind. These are filtered, washed, &c., and ignited in a current of oxygen, so as to burn off the carbon, then heated in a current of hydrogen to reduce any ferric oxide formed, and finally the residue once more subjected to the treatment with chlorine. After the second treatment the insoluble portion of the residue is weighed as gangue. The solutions obtained from these two operations together with the contents of the absorption flasks are acidified with a measured volume of hydrochloric acid of sp. gr. 1.1 and allowed to stand. Any insoluble matter is filtered off and fused with a mixture of sodium carbonate and potassium chlorate; the melt is then extracted with water and this extract added to the main solution. The residue (A) is weighed. The solution is transferred to a flask, the weight of which is approximately known, and so much sodium carbonate is added that the

resulting solution contains about 4 per cent. of hydrochloric acid of sp. gr. 1.1. In this solution the metals of the copper group are precipitated by a current of sulphuretted hydrogen and separated as usual. The filtrate is evaporated to dryness to separate the silica, which is filtered off, and weighed together with the residue A. When coloured it is refluxed with sodium carbonate and nitre and the bases removed; if it be white the silica should be determined by treatment with hydrofluoric acid and the residue tested for titanate. The filtrate from the copper group is oxidised with chlorine, nearly neutralised with sodium carbonate and precipitated with barium carbonate.

The filtrate containing nickel, manganese, cobalt and zinc together with barium carbonate, is treated as usual. The residue, containing the iron and chromium, is dissolved in hydrochloric acid and made up to 1 litre, of which 200 cc. are taken, evaporated to dryness, and fused with sodium carbonate and potassium chlorate. The fused mass is extracted with water and the residue fused again. The iron is determined in the insoluble portion of the extract and the chromium in the filtrate by the usual methods, care being taken to note the presence of alumina and of phosphoric acid, and to determine them if present.

2. *Determination of the Total Carbon, Phosphorus, and Sulphur.*—Another 5 grms. of the ore are decomposed as in (1) in a current of chlorine; the residue washed first with water and hydrochloric acid, to which a little chromous chloride is added, and then with water, and the dried residue oxidised by chromic and sulphuric acids, the evolved carbon dioxide being collected and weighed. The phosphorus and sulphur of the ore are contained in the absorption vessels, after the ignition, in the form of sulphates and phosphates. They are estimated as usual, after separating any silica present, by evaporation with sodium chloride.

3. *Determination of the Graphite.*—Ten grms. of the ore are dissolved in hydrochloric acid—weeks are needed to effect the solution—and the separated graphite filtered off on an asbestos, filtered, washed, and oxidised with chromic and sulphuric acids to carbon dioxide.—C. A. K.

*Determination of Free Sulphuric Acid in Aluminium Sulphate.* F. Beilstein and T. Grosset. Zeits. Anal. Chem. **29**, 73—78.

ERLENMEYER and Levinstein's method for the determination of the free acid in aluminium sulphate by treating with magnesium ammonium phosphate and titrating the free acid in the filtrate, is only accurate when the double phosphate is freshly prepared. When the magnesium ammonium phosphate has been kept for some time (even under water) it loses ammonia, and further, boiling is necessary to effect complete decomposition between the aluminium sulphate and the phosphate, and here a further loss of ammonia occurs. A review of other methods for the determination of the free acid in the presence of neutral or of acid salts follows. The authors consider that the methods dependent on the insolubility of sulphates in alcohol and on the direct titration with alkali employing one of the tropaeolin colours as indicator, are inaccurate, and are qualitative rather than quantitative tests. The following method is recommended as the most reliable:—Dissolve 1—2 grms. of the substance according to its richness in free acid, in 5 cc. of water, add 5 cc. of a cold saturated neutral solution of ammonium sulphate, allow to stand for a quarter of an hour with repeated shaking, and precipitate with 50 cc. of 95 per cent. alcohol. Filter, wash with 50 cc. of 95 per cent. alcohol, evaporate the filtrate on the water-bath, dissolve the residue in water, and titrate with decinormal caustic potash, using litmus as indicator. All the neutral aluminium sulphate is thus thrown down as ammonium alum, and the whole of the free acid remains in solution. Only a little ammonium sulphate is dissolved by the alcohol. For technical purposes the results are sufficiently accurate; the mean error of six test analyses amounts to about 0.15 per cent. The samples contained from 1.27 per cent. to 5.89 per cent. of free sulphuric acid.—C. A. K.



*Valuation of Roofing Slates.* H. Brunner. Schweiz. Wochenschr. f. Pharm. 1889.

See under IX., page 393.

*The Examination of Roofing Slates.* F. Reverdin and C. de la Harpe. Chem. Zeit. 14, 64—65, 94—95, and 126—127.

See under IX., page 394.

*The Valuation of Fuming Sulphuric Acid.* B. Setlik. Chem. Zeit. 13, 1670.

INSTEAD of the usual method of determining the sulphuric anhydride present by titration with normal caustic soda solution, the author proposes to titrate with water until the acid ceases to give off fumes of anhydride. 100 grms. of the fuming acid are weighed into a 200 cc. flask, and water is slowly dropped in from a burette whilst the flask is kept cool. At first a thick cloud is formed over the liquid on the addition of each drop, but as the composition of the liquid approaches that of the monohydrated acid, the fumes become less apparent, and the flask must then be shaken after each addition of water until the fumes have become completely absorbed. The point when a further drop of water causes no fuming is thus easily seen, and several results are quoted to attest the correctness of the method, which is very suitable for use in the factory.

—G. H. B.

*The Examination of Zinc Ores.* W. Minor. Chem. Zeit. 13, 1670.

THE article refers to the difficulty of discriminating between the amounts of zinc present as carbonate, silicate and sulphide. According to one method the carbonate and silicate may be extracted by boiling the finely powdered ore several times with strong ammonia, but the silicate is not satisfactorily reduced. The author recommends the following procedure. The total zinc is first determined by treatment with strong hydrochloric acid in the usual way. A second portion is carefully exhausted with boiling dilute caustic soda, which completely dissolves the carbonate and silicate of zinc, leaving the sulphide untouched. A third portion is boiled for 15 minutes with acetic acid of 50 per cent. strength, which dissolves only the carbonate of zinc. The author has proved the accuracy of this method of separation from the results of numerous concordant analyses.

—G. H. B.

*Estimation of the Cadmium in the Products of Zinc Manufacture.* W. Minor. Chem. Zeit. 14, 4.

IN order to avoid the fourfold precipitation with sulphuretted hydrogen (see this Journal, 1890, 109) the author suggests the following alternative method:—A known quantity of the substance is dissolved as usual in hydrochloric acid, and the cadmium and iron separated by precipitation with sulphuretted hydrogen. The precipitate of cadmium sulphide, which always contains zinc sulphide, is washed with hot water, dissolved in hydrochloric acid and filtered. The solution is heated to boiling and an excess of boiling, dilute caustic soda added; the cadmium hydroxide which separates is filtered off and repeatedly washed with 1 per cent. caustic soda, then with hot water, till no longer alkaline. The filter containing the precipitate is then transferred to a Rose's crucible, and the water driven off by gentle warming, after which the crucible is heated to redness while a stream of oxygen is passed into it. From the weight of cadmium oxide so obtained the amount of cadmium present in the sample can be readily found.

In the determination of the cadmium in the so-called "pure cadmium" of the zinc works, the precipitation by sulphuretted hydrogen may be omitted, as this product contains only a small trace of iron which does not influence the determination.—O. H.

*Estimation of Cadmium in Calamine.* W. Minor. Chem. Zeit. 14, 34.

THE amount of cadmium in calamine is generally not taken into account in estimating the zinc, but is determined with

it, in the ammoniacal solution obtained after the precipitation of the iron. As the cadmium is a very valuable by-product, it is suggested that it should be determined separately. The following method is proposed:—The ammoniacal filtrate containing zinc and cadmium is made slightly acid with hydrochloric acid and the hot solution poured into an excess of hot aqueous soda. The insoluble cadmium hydroxide is determined as oxide (see preceding abstract). The zinc is determined in the filtrate, after neutralising most of the excess of soda with hydrochloric acid, by titration with sodium sulphide.—N. H. J. M.

*Estimation of Nitrogen by Schultze-Tiemann's (Schlössing's) Method.* F. Coehius and T. Moeller. Chem. Zeit. 14, 33.

THIS method has been found, especially by those who have to do with explosives, to give too low results; control analyses made with pure potassium nitrate gave, as a rule, 13.6 per cent. of nitrogen (instead of 13.86), the results often being as low as 13.5 or 13.3 per cent. Long experience in analysing gun-cotton by the method, showed that the concentration of the reagents is of great importance, and that the amount of substance used should be suited to the size of the apparatus. The following were employed by the authors:—A long-necked flask of 350 cc. capacity, a measuring tube of 150 cc. capacity, a ferrous chloride solution (70 grms. of salt to 100 cc. of water), hydrochloric acid (about 37 per cent.). The amounts taken were:—Pure saltpetre, 0.3—0.4 gm.; ferrous chloride solution, 5—15 cc., with twice the amount of acid. When 25 to 50 cc. of water were used, the results varied between 13.76 and 13.86 (mean, 13.81) per cent. of nitrogen, and the experiment took 30 to 40 minutes. With 80 to 150 cc., percentages of 13.39 to 13.05 were obtained (mean, 13.21), the analyses took from 70 to 90 minutes.—N. H. J. M.

*On the Volumetric Determination of Nitric Acid by a Solution of Diphenylamine in Sulphuric Acid.* J. A. Muller. Bull. Soc. Chim. 1889, 670.

THIS method may only be used in the absence of other oxidising substances, as these would also give a colouration with a solution of diphenylamine in sulphuric acid. A small quantity of hydrochloric acid does not interfere, but the colour is altered and destroyed by hydrobromic and hydriodic acids. Spiegel, to whom the process is due, states that organic substances do not influence the colouration, but the author found that a few drops of a 7.5 per cent. solution of dextrin altered the colour materially.

To make an estimation, 5 cc. of a solution containing 0.2 gm. of diphenylamine in 1 litre of concentrated sulphuric acid are placed in a test tube and 1 cc. of the nitrate solution added; similar solutions are made containing a known quantity of nitric acid, and the colours, after standing one hour, compared. If care be taken to prepare all the tubes in exactly the same way, and the nitrate solution contain from 1—5 mgrms. of  $\text{N}_2\text{O}_5$  per litre, results may be obtained to within 0.5—1 mgrm. of  $\text{N}_2\text{O}_5$  per litre.

If any other oxides of nitrogen be present they must be oxidised by permanganate. Provided the solutions be colourless, this method may be applied to the determination of nitrates in soils and drainage waters.—A. L. S.

*Some Applications of Caustic Soda or Potash and Carbon to the Qualitative and Quantitative Analysis of Minerals.* C. A. Burghardt. Lit. and Phil. Soc. Manchester, March 4, 1890.

AFTER many experiments, the author found that in the analysis of all refractory silicates, several refractory oxides and compounds of oxides, and some other insoluble mineral compounds, the best results were obtained by mixing the finely divided mineral with about 10 per cent. of its weight of finely divided charcoal, and projecting the mixture carefully into a silver crucible containing fused caustic soda or potash in the proportion of six times the weight of the mineral powder taken for analysis. The crucible and its

contents were then carefully heated over an ordinary Bunsen's burner until the reaction arising is complete. This point is easily ascertained by the fact that all further evolution of combustible gas has ceased, and the mass in the crucible has become dry and generally white or grey in colour.

From the experiments made, the author believes that hydrogen gas is evolved from the caustic alkali, and possibly a small quantity of carbon monoxide, whilst undoubtedly carbon dioxide is largely produced by the combination of the carbon with the oxygen of the caustic alkali and the oxygen of the oxides present in the minerals taken for analysis. The author believes that during the course of the reaction, metallic sodium or potassium is liberated, and that these metals whilst in *statu nascenti* combine either with the metallic oxide (as in the case of the oxides of zinc and tin) or with the acid present in the mineral.

The details of some experimental trials of the process are next given.

*Tin-Ore from Cornwall.*—Two samples of roasted "Black tin" were reduced to an exceeding fine powder and submitted to Bughardt's process. ("Black tin" is the tin ore which has been concentrated by washing away the lighter mineral impurities, drying and afterwards roasting to secure freedom from sulphur, arsenic and antimony.) For the analysis, 0.5 grm. of the mineral was taken, and the heating in the silver crucible with the caustic alkali only took place over the ordinary Bunsen lamp for 30 minutes. Lixivation with water, followed by a second fusion of the residue, was sufficient to bring the mineral into a complete state of resolution.

*Wolframite* (principally tungstate of iron).—This is a difficult mineral to analyse by the older methods and much time is expended.

By the author's process the iron is entirely separated from the tungstic acid *at once*, and remains behind on the filter after solution of the melt and subsequent filtration, as ferric oxide and magnetic oxide.

*Chrome Iron Ore* was treated with caustic soda with an addition of ammonium nitrate to ensure full oxidation of chromic oxide to chromic anhydride. Heating for half an hour over the ordinary Bunsen lamp proved sufficient. Other minerals which proved amenable to the caustic soda treatment as described, were Rutile, Barytes, Kaolin, Tale, Cyanite, and double silicates like Tourmaline, Hornblende, Garnet. Excellent results have been obtained with Fluorspar and Pitchblende. By this method the costly and troublesome blowpipe and bellows are dispensed with.

#### *Action of Phenols on Honey and on the Substance produced by Evaporating to Dryness spent Sulphite Solutions.* A. Hbl. Chem. Zeit. **14**, 3.

THE author has already (this Journal, 1887, 455) described the action of phenols, in presence of sulphuric and hydrochloric acids, on the carbohydrates. When honey is treated with an aqueous solution of phloroglucinol and much strong hydrochloric acid, the mixture acquires, after some time, a pale red colouration, which becomes more and more distinct. Resorcinol produces, after a long time, a splendid light-red colour. Other phenols react with honey only when heated. Aqueous aniline sulphate solution colours a solution of honey yellow after some time.

When the yellow transparent mass, obtained by carefully evaporating to dryness spent sulphite-lye, is dissolved in water, and heated with aqueous phloroglucinol and strong hydrochloric acid, a violet-red colouration is produced; other phenols give inconsiderable colourations. With concentrated sulphuric acid, the colour reactions are more distinct:—aqueous resorcinol solutions and alcoholic solutions of orcinol, thymol, and cresol, in presence of much strong sulphuric acid, give strong dark red colourations, and an alcoholic solution of  $\alpha$ -naphthol produces a splendid, intense red-violet colour. The same reactions are obtained when the spent sulphite-lye itself is employed.—N. H. J. M.

#### *Coal Oil and Lignin.* A. Hbl. Chem. Zeit. **14**, 34.

THE author previously (this Journal, 1889, 1012) proposed urea and antipyrine as reagents for woody cell-membrane. Coal oil also gives rise to production of colour with lignin. Wood and paper treated with coal oil diluted with alcohol and strong hydrochloric or moderately dilute sulphuric acid acquire an intense red colour, the reaction being strongest when concentrated alcoholic solutions of coal oil are used. The colour reactions are given by all substances which yield coal oil when destructively distilled: for example, albuminoids, horn, wool, &c. It is probable that the colour is due to the action of pyridine bases in the coal oil on cinnamic aldehyde contained in the wood (compare this Journal, 1889, 640).—N. H. J. M.

#### *Tobacco Juice as a Reagent for Lignin.* A. Hbl. Chem. Zeit. **14**, 67.

THE author has described (Chem. Zeit. **14**, 34; see preceding abstract) a colour reaction given by lignin and bone oil in presence of hydrochloric or fairly concentrated sulphuric acid, and considers it due to the presence of pyridine bases present as empyreumatic products in the bone oil. Considering that the nicotine and albuminoids in tobacco might yield similar substances on destructive distillation, he substituted tobacco juice from a reservoir pipe for the bone oil, and found that, when fresh, a fairly intense red colouration was obtained, sufficing even for the detection of wood-cellulose in paper. The colour also appears when cinnamic aldehyde or an alcoholic solution of oil of cinnamon is treated with fresh tobacco juice and concentrated hydrochloric acid. Nicotine itself needs some time for the development of the colouration.—B. B.

#### *Detection of Cotton-Seed Oil in Lard.* A. V. Ashóth. Chem. Zeit. **14**, 93–94.

Muter and de Koningh (Analyst, 1889, 61) have described a method for the detection of cotton-seed oil in lard, an outline description of which is as follows:—The sample is saponified, the fatty acids precipitated as lead salts which are then washed and shaken out with ether; the ethereal solution of such fatty acids as form lead salts soluble in that liquid is decomposed by hydrochloric acid, the two layers of liquid separated, the solution of fatty acids in ether washed, its volume noted, a known quantity taken, and titrated with standard alkali; another portion is run off, the ether evaporated in a stream of carbon dioxide, and the iodine number of the fatty acids left determined.

The author experimented with single samples of pure lard and cotton-seed oil (source not stated), and found for the former 54.31 per cent. of fatty acids with an iodine number of 93.66, and for the latter 69.20 per cent. of fatty acids with an iodine number of 136.69. In both cases the fatty acids are calculated as oleic acid. Mixtures gave results fairly near those demanded by their composition, so that in one containing 50 per cent. of cotton-seed oil was found 48.66 per cent., in another 20.52 instead of 25 per cent., and 5 per cent. in place of 12.8 per cent. in a third. The author views the method as of considerable value.—B. B.

#### *Drying Oils, their Properties, Tests, and Applications.* L. Borucki. Tech. Mittheil. f. Malerei, **6**, No. 74 and 75.

THE author describes the origin, chemical composition, properties, and purification of these oils. He gives the following description of a good drying oil: it should be clear and transparent and should not become turbid either on heating or on long standing (absence of albuminoids, &c.); shaken with water or alcohol it should give no acid reaction, in the first case mineral acids are absent, in the second free fatty acids or resin; there should be no appreciable quantity of ash; treated according to Livache, it should dry in 24 hours, and the varnishes should yield a solid mass in the same time (absence of non-drying oils, resinous oils, and mineral oils). Livache's test is made as follows, 1 grm. of lead powder is prepared by precipitating lead acetate with zinc, washing first with water and then with alcohol and ether and drying in vacuo. This is placed on a watch

glass, weighed and 0.6 gram. of oil dropped on it from a burette so that each drop remains separate from the rest; it is then placed in a light room at the ordinary temperature. A drying oil thus treated ceases to lose weight after 18 hours, a non-drying oil not until six days.—A. L. S.

**Testing Cocaine.** K. Mezger. *Pharm. Zeit.* **34**, 697. To distinguish cocaine from other alkaloids, 0.05 gram. of cocaine hydrochloride is dissolved in 5 cc. of water and five

drops of a 5 per cent. solution of chromic acid added; as each drop is added a distinct precipitate is formed, which dissolves immediately. On then adding 1 cc. of hydrochloric acid (conc.) a dark orange-coloured precipitate of cocaine chromate is formed.

In the following table the behaviour of a number of alkaloids towards chromic acid, chromic acid and hydrochloric acid, and towards potassium chromate is shown. The solutions tested must be neutral.

0.05 Grms. dissolved in 5 cc. of Distilled Water.	I. 5 Drops of a 5 per cent. Solution of Chromic Acid.	II. Addition of 1 cc. of HCl (conc.) to I.	III. 5 drops of a 9.20 Per Cent. Solution of $K_2CrO_4$ .
Cocaine hydrochloride .....	Distinct precipitate with each drop, which then disappears.	Orange yellow, amorphous precipitate.	Clear.
Ecgonine hydrochloride ....	Clear.	Clear.	Clear.
Quinine hydrochloride .....	Yellow precipitate.	Precipitate dissolves; on standing separates again in needle-shaped crystals.	Yellow precipitate.
Quinidine hydrochloride ....	Yellow precipitate.	As with quinine.	Yellow precipitate.
Cinchonidine hydrochloride	Yellow precipitate.	As with quinine.	Turbidity with each drop added; on standing a crystalline precipitate.
Cinchonine hydrochloride ...	Clear.	Clear.	Pale yellow precipitate.
Hydroquinine hydrochloride	Yellow resinous precipitate.	Clear. Crystals separate after long standing.	Yellow precipitate.
Apomorphine hydrochloride	Red-brown precipitate turns green after standing a minute.	Dirty brown precipitate.	Dirty brown precipitate.
Brucine hydrochloride .....	Yellow precipitate.	Orange-yellow precipitate. Solution dark red.	Crystalline precipitate forms gradually.
Strychnine hydrochloride ...	Yellow crystalline precipitate forms after a minute's standing.	Yellow crystalline precipitate.	As with brucine.
Veratrine hydrochloride ....	Yellow precipitate.	Yellow precipitate.	Precipitate forms gradually.

Sparteine and atropine sulphates and the hydrochlorides of caffeine, pilocarpine, and codeine give no reactions with these reagents. Morphine hydrochloride gives a green colouration with all three reagents owing to the reduction of the reagent; narceine gives a crystalline precipitate after long standing with chromic and hydrochloric acids.

—C. A. K.

**Detection of Rosin Oil in Fatty and Mineral Oils.** Hölde. *Mittheil. der könig. tech. Versuchsanstalten*, 1890, 19—22.

THE author finds that in his method for the detection of rosin oil in vegetable or mineral oils (this Journal, 1888, 526), it sometimes happens that the colouration does not readily appear. A continuous and vigorous shaking, however, causes the acid to assume a distinct red colour when rosin oil is present. For detecting the presence of small quantities of rosin oil, he recommends shaking with stronger sulphuric acid of sp. gr. 1.624, whereby (in testing a series of 11 different sorts of rosin oil) he always obtained a distinct red colouration after shaking for a short time. To apply a still stronger acid is not advisable, as it would affect the other oils present. This reaction is not as delicate as that with anhydrous acetic acid and sulphuric acid (this Journal, 1888, 136, 526 and 871; 1889, 572), but being extremely simple the author considers it useful for technical purposes as a preliminary examination. Quantities as small as 1.5 per cent. of rosin oil could in all experiments be detected by this reaction, and mostly even 0.5 and 1.0 per cent. Castor oil is the only exception, its limit being 5 per cent. But in this case, after shaking with the above-named acid, the castor oil itself, and not the acid, assumes a red colour, even if as little as 1 per cent. of rosin oil be present, whilst pure castor oil remains yellowish-white. The author thinks that intentional adulterations of less than 1 per cent. are practically not to be found.

When sulphuric acid of 1.624 sp. gr., after having been shaken with the oil, is of a yellow or yellowish-brown colour, rosin oil is not present in the above-named limits; but if the

acid become red or brown, another portion of the substance should be shaken with double its volume of 86—90 alcohol, to extract rosin oil, if present. This extraction is necessary, because the presence of train oils, earth-nut oil, and mineral oils containing asphalt substances, may give similar colour appearances in the original substance. Castor oil must be treated in the same way, if the acid has changed the colour of the oil to red or brown, which colour may originate from the presence of train oil. The test cannot be applied for those tar-oils which are soluble in alcohol with brown or black colours. The alcoholic extract is then treated with sulphuric acid of 1.624 sp. gr., the presence of rosin oil being shown by a red or violet colouration. This colour is more constant than that obtained by the test with anhydrous acetic acid and sulphuric acid of 1.53 sp. gr., which changes after a few moments. For the detection of smaller quantities of rosin oil, the alcoholic extract is evaporated, then the test with sulphuric acid of 1.624 sp. gr. applied, and a comparative test with anhydrous acetic acid and sulphuric acid of 1.53 sp. gr. made. The author finds, with Morawsky, that anhydrous acetic acid and sulphuric acid of 1.53 sp. gr. produce red-brown colourations in train oil and in earth-nut oil. Therefore, when these substances are present, and rosin oil is to be tested for, an alcohol extraction has to be made prior to the application of the above reagents. The alcoholic extracts of train oil and of earth-nut oil do not show the colourations characteristic for rosin oil.

The author furthermore states that the phosphoric acid test for train oil is uncertain, for on the one hand rosin oils produce red colourations with this acid, and on the other hand distinct colourations only appear when large quantities of train oil are present in other oils. The author mentions that this is contrary to the experience of Schaeidler, who found that even  $\frac{1}{10000}$  of train oil can be detected by the reaction with phosphoric acid. Whether the above reaction with phosphoric acid is characteristic for rosin oils has to be proved. (Compare this Journal, 1890, 112.)—H. S.



*Boessneck* has suggested that a method might possibly be based on the solubility of antimonious oxide in tartaric acid in the proportion of one molecule to two of the acid, and the author has experimented in this direction. He finds, however, that variable quantities of antimonious oxide are dissolved, e.g., 60—66.9 per cent. instead of the theoretical 77.6 per cent., the action appearing to diminish as the proportion of free tartaric acid decreases.—B. B.

*On the Estimation of Glycerol in Crude Glycerin.* F. Filsinger. *Chem. Zeit.* **14**, 197.

To ascertain the amount of glycerol in crude glycerins, the author compares a series of results obtained by Fickert and Hirsch between the modified permanganate method (*Chem. Zeit.* **13**, 127) and Benedikt and Cantor's acetin method as applied by O. Hehner (this Journal, 1889, 6—8). Throughout the series, which consists of crude glycerins of different origin, the acetin method gives higher results than the permanganate method, viz.:—

No.	Permanganate Method.	Acetin Method.
	Per Cent.	Per Cent.
1	70.2	(a) 74.7
	..	(b) 75.4
2	78.5	(a) 80.6
	..	(b) 81.9
3	76.1	(a) 80.6
	..	(b) 80.9
4	72.1	76.8
5	(a) 73.9	(a) 78.4
	(b) 73.3	(b) 78.2
	..	(c) 78.5
6	73.8	78.1
7	70.5	74.9
8	72.1	73.2
9	(a) 77.6	79.5
	(b) 78.0	..
10	(a) 82.2	86.7
	(b) 81.9	..
11	(a) 50.6	(a) 54.8
	(b) 50.4	(b) 53.4
12	(a) 66.3	69.9
	(b) 66.7	..
13	(a) 69.6	76.6
	(b) 70.1	..
14	(a) 74.8	77.4
	(b) 75.2	..
15	(a) 67.1	(a) 73.4
	(b) 66.5	(b) 73.7
	..	(c) 73.8
16	77.2	79.8
17	71.2	(a) 76.7
	..	(b) 77.4

The author was in a position to testify to the correctness of the permanganate method in several instances by the practical yields from the crude materials employed. The results obtained confirm his view that the acetin method gives percentages of glycerol in crude glycerins which are too high, and he states that these results are

contrary to those obtained by Hehner and Lewkowitsch. He objects to Hehner's proposal in analysing crude glycerin to strike an average between the results obtained by the acetin and the bichromate methods, but recommends further examination which might lead to a more correct modification of the acetin method.—H. S.

*Determination of Cellulose.* G. Lange. *Zeits. Physiol. Chem.* **14**, 283.

Ten grms. of the substance in which the cellulose is to be determined are placed in a stoppered retort, together with three or four times its weight of pure caustic potash and 30—40 cc. of water, and the temperature raised to 140° C. by means of an oil-bath. At this point reaction sets in with considerable frothing, and is generally complete after continuing the heating for about an hour at 180° C. After cooling to about 80° C. the contents of the retort are washed into a beaker, acidulated with dilute sulphuric acid, made slightly alkaline with weak caustic soda to dissolve any substance other than cellulose precipitated with it on acidulation, and the purified cellulose filtered off by the aid of a filter pump. By washing with hot and cold water, drying, weighing, and determining the ash retained, the quantity of pure cellulose is arrived at.—B. B.

*Estimation of Quinine in Quinine Tannate.* S. Neumann. *Zeits. Anal. Chem.* **28**, 663—668.

The quinine in tannate of quinine cannot be accurately estimated by Orillard's method, which is recommended by the German Pharmacopœia owing to the fact that the alkaloid cannot be completely extracted with alcohol. The following method can be conveniently employed for the analysis of the tannate:—2 grms. of the finely-divided salt (*Quinimum tannicum*) are decomposed with 20—25 cc. of potash of sp. gr. 1.24, the mixture is then diluted to about 60—80 cc. and shaken vigorously with 100 cc. of pure ether in a well-stoppered bottle, in order to dissolve the liberated quinine. The presence of solid particles in suspension, either in the ethereal or alkaline solution, shows that the sample is impure or that the salt has not been completely decomposed. After some time 50 cc. of the ethereal extract are transferred to a tared beaker by means of a pipette and the solution is allowed to evaporate in a warm place; the residue is then dried at 100° and weighed. The residue should be completely soluble in dilute hydrochloric acid and in dilute sulphuric acid.

Quantitative experiments with sulphate of quinine showed that the author's method gives results which agree well among themselves, but which are generally about 2.5 per cent. too high; this is owing to the fact that the ether is slightly soluble in water and that evaporation takes place during the process.

A number of different samples of commercial tannate of quinine examined by the method described above gave a percentage of quinine more than double that found by Orillard's method.—F. S. K.

*Butter Fat.* S. Bondzynski and H. Ruff. *Zeits. Anal. Chem.* **29**, 1—6.

To determine the easily volatile acids contained in butter fat the authors take advantage of the fact that these are the most readily soluble and carry out the determination as follows:—4—5 grms. of the butter are saponified with 50—60 cc. of semi-normal alcoholic potash, the excess of alkali neutralised by semi-normal hydrochloric acid, and the alcohol evaporated off. The resulting soap is decomposed by an excess of hydrochloric acid, the insoluble fatty acids filtered off, washed with hot water, dissolved in alcohol, and the solution titrated with alcoholic potash. From the difference between the number of cc. of alcoholic potash required by the total fatty acids and the number required by the insoluble acids, the amount required to neutralise the volatile acids is got. This value can be got directly by

dissolving the soap first obtained, after evaporating off the alcohol, in water, and adding the same quantity of semi-normal sulphuric acid as of caustic potash required for the saponification. The insoluble fatty acids are then filtered off and washed, and the volatile acids determined by titration in the filtrate. The two methods are said to yield results agreeing amongst themselves and with those got by the ordinary method of distillation. If the insoluble fatty acids be weighed before titration, the amount of the glycerides is got and the amount of the glycerides of the volatile acids can then be obtained by difference.

The volatile acids are only present in butter as glycerides, and it is only after prolonged keeping that their presence in the free state becomes evident. Samples kept for 6—8 days at a temperature of 25° show no trace of free volatile acid, although the decomposition of the butter is considerably advanced. Butter, when quite fresh, contains some free insoluble acids and also free oleic acid. The turning sour of butter is essentially due to the increase in the quantity of insoluble fatty acids and especially to that of oleic acid. Two samples tested gave when fresh 0.50 per cent. and 0.27 per cent. of insoluble fatty acids, and 0.17 per cent. and 0.16 per cent. of oleic acid; after 20 days' keeping at the ordinary temperature they contained 1.29 per cent. and 3.60 per cent. of the former, and 0.47 per cent. and 0.71 per cent. of the latter respectively.

Phenolphthaleïn is recommended as the indicator to be used in testing for the free acids in preference to litmus, the latter being quite useless in testing for the free oleic acid. The importance of determining the free acid in butter as a means of determining its freshness is pointed out. The method employed is described in detail in the paper.

To determine the quantity of hydroxy acids in fresh butter the authors adopt Benedikt's method (this Journal, 1888, 594). The butter is saponified, the insoluble fatty acids filtered off, and the water in the filtrate removed by repeated filtrations through dry filter paper. The resulting product is then treated with acetic anhydride. An experiment gave 18.2 as the acetyl value for butter.—C. A. K.

*Contributions to the Analysis of Fats.* H. Noerdlinger. Zeits. Anal. Chem. **29**, 6—13.

This is a continuation of previous work on the free fatty acids of fats, oils, &c. (this Journal, 1889, 806). The free fatty acids are determined by extracting the total fats with petroleum ether, and phenolphthaleïn is used as the indicator in the titration with alkali; the results are calculated as oleic acid. In order to judge of the rancidity of fats and oils of commerce by the amount of free acids they contain, it is necessary to know the percentage of free acids in the pure products. The results of experiments in this direction is shown in the following table, both for oils and oil-cakes. The numbers given are the mean values found:—

	100 Parts contain		Per Cent. of Free Fatty Acids on Total Fat.
	Free Fatty Acids.	Total Fats.	
I.—OILS.			
Rape ( <i>Brassica rapa</i> ).....	0.42	37.75	1.10
Cabbage ( <i>Brassica campestris</i> )...	0.32	41.22	0.77
Poppy ( <i>Papaver somniferum</i> )....	3.20	46.90	6.66
Earth-nut ( <i>Arachis hypogæa</i> )— (a.) Seed .....	1.91	46.60	4.15
(b.) Outside pale yellow husk...	1.91	4.13	43.10
Sesame ( <i>Sesamum orientale</i> ) .....	2.21	51.59	4.59
Castor ( <i>Recinus communis</i> ) .....	1.24	46.32	2.52
Palm-nut ( <i>Elaeis guineensis</i> ) (with 6 per cent. husks) .....	4.19	49.16	8.53
Coprah ( <i>Cocos nucifera</i> ) .....	2.98	67.10	4.42

	100 Parts contain		Per Cent. of Free Fatty Acids on Total Fat.
	Free Fatty Acids.	Total Fats.	
II.—CAKES.			
Rape .....	0.93	8.81	10.55
Poppy .....	5.66	9.63	58.89
Earth-nut .....	1.42	7.65	18.62
Sesame .....	6.15	15.44	40.29
Palm-nut .....	1.47	10.39	14.28
Earth-nut meal (extracted).....	1.55	18.68	8.29
Cocoa.....	1.31	13.11	10.51
Linseed.....	0.75	8.81	9.75
Castor .....	1.27	6.53	20.07

The oils obtained by pressing only contain a portion of the free fatty acids. The so-called "technical oils" which result from a second and third pressing of the seeds also contain free fatty acids, but the greater portion thereof remains behind in the cakes.

In the case of "extracted oils," as opposed to oils obtained by pressing, it is pointed out that the composition of the fat extracted is the same as that left behind in the seeds (*i.e.*, both contain the same percentage of free acids), since both glycerides and fats are equally dissolved by the solvents. The test for free acids, together with a microscopical examination, serves therefore to distinguish "extracted" from "pressed" meal; and further, by testing the oils also for free acid "extracted" and "pressed" oils can be distinguished. This holds as long as the product is unadulterated. A number of analyses of palm-nut cake and of cocoa-nut cake are given which bear out this point.

—C. A. K.

*Cassia Oil and its Adulteration.* Schimmel and Co. Zeits. Anal. Chem. **29**, 93—94.

CASSIA oil is adulterated with colophonium and other substances, in China. To examine the oil, 50 grms. are fractionated and the distillation continued up to 280°—290°. The main bulk of the oil distils over between 240° and 260°. The residue in the fractionating flask is weighed; if it solidify on cooling the oil is adulterated; pure cassia oil also leaves a residue, but this remains liquid on cooling. Gilbert (Chem. Zeit. **13**, 1406) has shown that cassia oil adulterated with colophonium satisfies the requirements of the Pharmacopœia, and considers that the determination of the acid equivalent lends the best aid to the detection of the adulterant. Two grms. of the oil are dissolved in 30 cc. of alcohol, and titrated with alcoholic potash, using phenolphthaleïn as indicator; 1 gm. of the pure oil requires from 10—13 mgrms. of the alkali, whilst a sample containing 20 per cent. of colophonium needs 40 mgrms., and colophonium itself (sp. gr. 1.08) 150 mgrms.

Schimmel and Co. value cassia oil by a determination of the cinnamyl aldehyde contained. This is arrived at by difference, the sample being treated with acid sodium sulphite and the uncombined portion of the oil weighed. No details of the method are given. It is considered that a sample of the oil with less than 75 per cent. of aldehyde is suspicious, and with less than 70 per cent. adulterated. The chief constituent of cassia oil beyond cinnamyl aldehyde is cinnamyl-acetic ether— $\text{CH}_3\text{CO.O.C}_9\text{H}_9$ .—C. A. K.

*The Incompatibility of Codeine and Morphine Sulphate.* Claassen. N.Y. Pharm. Rund. 1890, 40.

THE author points out that codeine is capable of decomposing morphine sulphate as well as morphine salts generally with separation of morphine which is insoluble in water. This

reaction is sufficiently complete to admit of its application to the estimation of the amount of codeine present in a liquid. For this purpose an excess of morphine sulphate is added to the warmed liquid with frequent shaking, and after cooling the mixture is allowed to stand for 48 hours and the separated morphine is collected on a tared filter. If after addition of more morphine sulphate to the liquid no further deposition takes place in 24 hours, the weight of the morphine obtained multiplied by 0.9868 gives the amount of anhydrous codeine, or multiplied by 1.0462, the amount of codeine crystallised with one molecule of water. Thus, 100 parts of anhydrous codeine decomposes 126.76 parts of morphine sulphate. In cases where codeine salts or morphine or morphine salts may be present, excess of magnesia is added and the mixture evaporated to dryness. The residue is now stirred up with some water, shaken several times with ether free from alcohol, the ether distilled off and the residue exhausted with hot water. The codeine in the resulting solution can then be determined as above specified.—O. H.

#### PATENT.

*A Process and Apparatus for Freeing Wool from Grease.* L. Pinagel, Aachen, Germany. Eng. Pat. 20,936, December 31, 1889. 8d.

See under V., page 386.

### New Books.

DIE CHEMIE DES STEINKOHLENTHEERS MIT BESONDERER BERÜCKSICHTIGUNG DER KÜNSTLICHEN ORGANISCHEN FARBSTOFFE, VON DR. GUSTAV SCHULTZ. Zweite vollständig umgearbeitete Auflage. Zweiter Band. Die Farbstoffe. Siebente (Schluss-) Lieferung. Braunschweig: Friedrich Vieweg und Sohn. London: H. Grevel and Co., 33, King Street, Covent Garden. 1890.

THIS, the concluding part of the whole work, commences on page 1153, and from this page to page 1267 we have an Appendix continued from the preceding part and concluded. Thus ends the Appendix devoted to the newest patented discoveries and inventions in Coal Tar products and Colours. On page 1268 commences a scheme of analysis for the Artificial Organic Colouring Matters, which ends on page 1275.

From page 1276 to page 1354 extends a comprehensive index of German patents taken out on Coal Tar preparations, and arranged in tabular form under the following headings:—No. of the German Patent. Date. Applicant and Holder of the Patent. Title of the Patent. Class. Remarks. The work terminates with a copious and complete Alphabetical Index for the entire work.

A POCKET-BOOK FOR PHARMACISTS, MEDICAL PRACTITIONERS, STUDENTS, &c., &c. (British, Colonial, and American.) By THOMAS BAYLEY, Assoc. Roy. College of Science, Consulting Chemist, Analyst, and Assayer. London: E. and F. N. Spon, 125, Strand. New York: 12, Cortlandt Street. 1889.

THIS little work is arranged as a pocket-book, is bound in limp morocco leather and gilt, price 6s. On the inside of cover is an Almanack, which is followed by 13 pages of blue-lined blank paper for memoranda or notes. Then come the Title-Page, Preface, and 523 pages of Subject-Matter,

Tables, &c., Alphabetical Index, and nine pages of blank blue-lined paper for notes. The nature of the contents will be gathered from the following synopsis:—

Notes on Materia Medica; Notes on Ordinary Food Materials, Condiments, &c.; Galenical Preparations. Official Drugs, arranged according to Botanical System. Glossary of Botanical Terms. List of Latin Terms and Technical Phrases, &c. Glossary of Medical Terms. Antidote Bag. Poisoning, Domestic Treatment, Symptoms and Treatment. Posological Table. Boiling Points of Liquids. Melting Points of Official Substances. Specific Gravity of Liquids, &c. Solubility of Medicinal Substances.

ANALYSIS:—Testing for Official Substances, Animal Secretions, Calculi, Urine, &c., Alkaloids, Table of A.S. Volumetric Test Solutions of B.P. Behaviour with Indicators. Alkaloids, Properties, and Reactions. Atomic Weights of Elements. Weights and Measures. The Text closes with a selection of Useful Recipes. The little work closes with an Alphabetical Index.

A HANDBOOK FOR SUGAR MANUFACTURERS AND THEIR CHEMISTS. Containing Practical Instruction in Sugar-house Control, the Diffusion process, Selected Methods of Analysis, Reference Tables, &c. By GUILFORD L. SPENCER, A.C., of the U.S. Department of Agriculture. New York: John Wiley and Sons, 15, Astor Place. London: Kegan Paul, Trench, Trubner and Co., Limited. 1889.

SMALL 8vo. volume in Pocket-book form, bound in morocco leather, price 10s. It contains Preface, 117 pages of Subject-Matter, an Alphabetical Index, and concludes with 93 pages of ruled and headed, "Blank Forms for Practical use in Sugar-house Work," and 20 pages of blue-lined blank sheets for memoranda. The text of the 117 pages of Subject-Matter is entitled, "Chemical Control of Sugar-house Work," and it is paraphrased as follows:—1. Measurements. 2. Measurement of the Juice. 3. Measurement of the Juice in the Clarifiers. 4. Automatic Measurement of the Juice. 5. Automatic Determination of the Density of the Juice. 6. The Weight of the Juice, Automatic Determination. 7. Measurement of the Syrup. 8. Weight of the Syrup. Automatic Scale. 9. Masseccutes. Measurement and Weight. 10. Commercial Masseccute. 11. Sugar Weights. 12. Diffusion Work. Measurement of the Juice. 13. Diffusion Work. Automatic Registration of the Amount of Juice Drawn. Then follow:—Chemical Control. Estimation of Losses. Diffusion. Estimation of Cane-Sugar by the Optical Method. Estimation of Cane Sugar by Fehling's Method. General Analytical Work. Available Sugar. Examination of the Bone-Black. Examination of the Quality of the Water Supply and Remedies for Impure Water. Special Reagents. After this, from page 61 to page 117, follow a series of useful Tables and Tabulated Data.

With the text are interspersed 10 engraved illustrations.



## Trade Report.

### TARIFF CHANGES AND CUSTOMS REGULATIONS.

(From the Board of Trade Journal.)

#### SWITZERLAND.

##### Classification of Articles in Customs Tariff.

Note.—Quintal = 220·4 lb. avoirdupois. Franc =  $9\frac{4}{10}$ d.

The following decisions affecting the classification of articles in the Swiss Customs tariff were given by the Swiss Customs authorities in the month of January last:—

Tobacco extract.—Category 9. Duty, 10 francs per quintal.

So-called "sulfonal."—Category 9a. Duty, 7 francs per quintal.

Chromate of chrome.—Category 16. Duty, 30 centimes per quintal.

#### AUSTRIA-HUNGARY.

##### Customs Regulations concerning Creoline and Preparations of Creoline.

The *Moniteur Belge* for the 1st March says that the Austro-Hungarian Customs authorities have recently laid down the following Customs régime for imports of creoline, creoline soap, and pharmaceutical preparations of a base of creoline:—

Solutions of soap in coal-tar oil (naphthalene oil) contained in casks or other receptacles are liable to an import duty of 10 fl. per 100 kilos., conformably to Art. 331 of the tariff.

However, when in any way whatsoever these products, according to their inscriptions, labels, wrappers, &c., appear as medicaments, they will be treated according to Art. 336 of the tariff, which provides for a duty of 24 fl. per 100 kilos., and the sanitary restrictions relative to the source of origin of compounded medicines must be observed.

Creoline soap (soap for destroying parasites in animals, &c.) is liable to the same duty as fancy soap, namely, 15 fl. per 100 kilos., Art. 342b of the tariff, whilst in conformity with the general dispositions, Arts. 336 and 337 of the tariff do not apply. However, in the latter case, the restrictive sanitary measures will be dispensed with.

Pharmaceutical preparations made from creoline (pills, capsules, &c.) are subject, both as regards Customs treatment and the restrictive sanitary measures of their free traffic, to the regulations in force concerning medicines.

#### NICARAGUA.

##### Tallow for Soap Manufacture.

According to the *Moniteur Belge*, the Government of the Republic of Nicaragua has just published a decree declaring free of import duty tallow imported as the raw material used in the manufacture of soap; but the manufacturing importer who sells the tallow free of duty, in virtue of this decree or of former regulations, will incur a fine equal to the value of the quantity sold; and in case of repetition he will be doubly fined, and his authority to further import this article withdrawn.

#### CUSTOMS TARIFF OF MAURITIUS.

Note.—Kilog. = 2·204 lb. avoird. Hectol. = 100 litres = 22 Imp. gallons.

The following is a statement of rates of import duty now levied in the colony of Mauritius:—

Classification of Articles.	Rates of Duty now leviable.
Animal charcoal.....	Rs. Cts. 1,000 kilos. 0·25
Charcoal.....	100 kilos. 0·10
Lime.....	" 0·20
Matches, each box not containing more than 100 matches.....	Gross 0·75
Opium: crude.....	Kilo. 20·00
" refined.....	" 10·00
Manure of all sorts; and the following substances when imported for the purpose of being used in the preparation of manures or other colonial produce, or as disinfectants, viz.: Ammoniacal liquor.....	
Bones, bone-dust, bone-oil, and dissolved bones.....	
Carbolic acid.....	
Chloride of lime.....	
Chloride of manganese.....	
Chloride of soda, solution of soda.....	
Chloride of zinc.....	
Coal and wood soot.....	
Dried muscular flesh and dried blood...	
Ether.....	
Fish and other substances damaged and condemned by the Customs sanitary officers as fit for manure only.....	1,000 kilos. 0·25
Lime, carbonate of lime, sulphate of lime or gypsum, phosphate and superphosphate of lime.....	
Nitrates, silicates, and carbonates of potash and soda.....	
Percchloride of iron.....	
Permanganate of potash.....	
Phosphate of soda.....	
Sulphate of iron.....	
Sulphate and muriate of ammonia and other ammoniacal salts.....	
Sulphate of potash.....	
Sulphate of zinc.....	
Sulphuric acid.....	
Urate and sulphuretted urine.....	
All goods, wares, and merchandise not otherwise charged with duty, and not mentioned above, or not specially exempted, shall be liable to a duty of.....	7½ % ad val.

#### THE AMERICAN TARIFF BILL.

The *Oil, Paint, and Drug Reporter* publishes the full text of the drug section of the Tariff Bill adopted by the Ways and Means Committee of the American House of Representatives. The trouble in the States is that, in spite of the fact that their tariff is higher than that of almost any other nation, the imports continue to increase, and the financial condition of the country is such that it has become

imperative to reduce the revenue. Although the Democratic party, who proposed to adopt legislation in a direction towards free trade, were defeated at the last Presidential election, the anomalies of the existing tariff are so manifest that even the present Republican and Protectionist Government have found it necessary to produce a Tariff Reform Bill of some kind. In this measure they propose, along with a reduction of certain duties, the imposition of an advance in others; but, so far as can be judged at present, the chance that the Bill evolved from the deliberations of the Committee of the House of Representatives will be passed into law is exceedingly slight. The principal alterations proposed affecting the drug trade are that acetic acid is to be reduced from  $7\frac{1}{2}$  cents to 6 cents per pound according to its specific gravity, while the duty on ammonia is changed from an *ad valorem* to a specific rate. The duties on refined camphor are reduced from 5 cents to 4 cents per pound, on chloroform from 50 cents to 40 cents per pound, on raw glycerin from 2 cents to 1 cent per pound, and on refined glycerin from 5 cents to  $4\frac{1}{2}$  cents per pound; on morphia and morphia salts from 1.00 dols. to 50 cents per ounce, on castor oil from 80 cents to 50 cents per gallon, on castor beans from 50 cents to 32 cents per bushel of 50 lb., on ultramarine blue from 5 cents to  $4\frac{1}{2}$  cents per pound, and on varnishes from 40 to 35 per cent. *ad valorem* (with 1.32 dols. per gallon additional duty for every gallon of alcohol contained in them); on acetate of lead from 6 cents to 3 cents for white and from 4 cents to 2 cents per pound for brown; on bicarbonate of soda from  $1\frac{1}{2}$  cents to 1 cent per pound; on refined roll sulphur from 10 dols. to 8 dols., and on flowers from 20 dols. to 10 dols. per ton; and on hops from 22 cents to 15 cents per pound. On the other hand, it is proposed to increase the duty on linseed oil from 25 cents to 30 cents per gallon, that on linseed, poppy-seed, and other oil seeds from 20 cents to 30 cents per bushel, and on window glass by about 10 per cent. The duty on drugs not specified is maintained at 10 per cent. *ad valorem*, on alcoholic perfumes at 50 per cent. *ad valorem* plus 2 dols. per gallon, on liquid preparations of opium at 40 per cent., on unspecified alcoholic medicinal preparations at 50 cents per pound; on all other medicinal preparations, alkaloids, and essential oils at 25 per cent.; on toilet soaps at 15 cents per pound and other soaps 20 per cent. *ad valorem*. It is not proposed to revive the duty on quinine.

#### THE TARIFF BILL.

The following is the text of the clauses in the bill prepared by the Ways and Means Committee affecting "miscellaneous metals and manufactures of" under Schedule C.

Aluminium or aluminium manufactured or in crude form or contained in alloys in which aluminium is the component material of chief value, 35 per cent. *ad valorem*.

Antimony, as regulus or metal, three-fourths of one cent per pound.

Argentine, albata, or German silver unmanufactured, 25 per cent. *ad valorem*.

Brass, in bars or pigs, old brass, clippings from brass or Dutch metal, and old sheathing, or yellow metal, fit only for remanufacture,  $1\frac{1}{2}$  cents per pound.

Bronze powder, 15 cents per pound; bronze or Dutch metal, or aluminium, in leaf, 10 cents per package of 100 leaves.

#### Copper.

Copper imported in the form of ores,  $1\frac{1}{2}$  cents per pound on each pound of fine copper contained therein.

Old copper, fit only for remanufacture, clippings from new copper, and all composition metal of which copper is a component material of chief value, not specially enumerated or provided for in this Act,  $1\frac{1}{2}$  cents per pound.

Regulus of copper and black or coarse copper, and copper cement,  $1\frac{1}{2}$  cents per pound on each pound of fine copper contained therein.

Copper in plates, bars, ingots, Chili or other pigs, and in other forms, not manufactured, not specially enumerated or provided for in this Act, 2 cents per pound.

■ Copper in rolled plates, called braziers' copper, sheets, rods, pipes, and copper bottoms, also sheathing or yellow

metal of which copper is not the component material of chief value, and not composed wholly or in part of iron ungalvanised, 35 per cent. *ad valorem*.

#### Gold and Silver.

Bouillons, or cannetille, metal thread, file or gespinst, and epaulettes, galloons, laces, knots, stars, tassels and wings of gold, silver, or other metals, 35 per cent. *ad valorem*.

Gold leaf, 2 dols. per package of 500 leaves.

Silver leaf, 75 cents per package of 500 leaves.

#### Lead.

Lead ore and lead dross,  $1\frac{1}{2}$  cents per pound; *provided*, that silver ore containing lead shall pay a duty of  $1\frac{1}{2}$  cents per pound on the lead contained therein, according to sample and assay at the port of entry.

Lead in pigs and bars, glaziers' lead, lead wire, molten and old refuse lead run into blocks and bars, and old scrap-lead fit only to be remanufactured, 2 cents per pound.

Lead in sheets, pipes, or shot,  $2\frac{1}{2}$  cents per pound.

Metallic mineral substances in a crude state and metals unwrought, not specially provided for in this Act, 20 per cent. *ad valorem*; mica, 35 per cent. *ad valorem*.

#### Nickel.

Nickel in ore, 2 cents per pound for the nickel contained therein.

Nickel in matte, or other crude form not ready for consumption in the arts, 5 cents per pound on the nickel contained therein.

Nickel, nickel oxide, alloy of any kind in which nickel is the component material of chief value, 15 cents per pound.

Quicksilver, 6 cents per pound; flasks, bottles, or other vessels in which quicksilver is imported shall be subject to the same rate of duty as they would be subjected to if imported empty.

Type-metal,  $1\frac{1}{2}$  cents per pound for the lead contained therein; new types, 25 per cent. *ad valorem*.

#### Zinc or Spelter.

Zinc in blocks or pigs,  $1\frac{3}{4}$  cents per pound.

Zinc in sheets,  $2\frac{1}{2}$  cents per pound.

Zinc, old and worn out, fit only to be remanufactured,  $1\frac{1}{4}$  cents per pound.

#### Miscellaneous.

Manufactures, articles, or wares not specially provided for in this Act, composed wholly or in part of iron, steel, lead, nickel, pewter, copper, tin, zinc, gold, silver, platinum, or any other metal, and whether partly or wholly manufactured, 45 per cent. *ad valorem*.

The most important changes under this head are therefore:—

Aluminium, hitherto free; proposed rate, 35 per cent. *ad valorem*.

Antimony as regulus.—Present rate, 10 per cent. *ad valorem*; proposed rate,  $\frac{3}{4}$  cent per pound.

Copper ore, &c.—Present rate,  $2\frac{1}{2}$  cents per pound; proposed rate,  $1\frac{1}{4}$  cents per pound.

Regulus of copper.—Present rate,  $3\frac{1}{2}$  cents per pound; proposed rate,  $1\frac{3}{4}$  cents per pound copper contained therein.

Copper in rolled plates.—Present rate, 35 per cent. *ad valorem*; proposed rate, 2 cents per pound.

Lead in sheets, &c.—Present rate, 3 cents per pound; proposed rate,  $2\frac{1}{2}$  cents per pound.

Metallic mineral substances.—Present rate, 20 per cent. *ad valorem*; proposed rate, 25 per cent. *ad valorem*. Mica, 35 per cent. *ad valorem*; at present free.

Nickel ore, &c.—Present rate, 15 cents per pound; proposed rate, 2 cents per pound for nickel contained.

Nickel in matte, &c.—Present rate, 15 cents per pound; proposed rate, 5 cents per pound for nickel contained.

Quicksilver.—Present rate, 10 per cent. *ad valorem*; proposed rate, 6 cents per pound.

Zinc or spelter.—Present rate,  $1\frac{1}{2}$  cents per pound; proposed rate,  $1\frac{1}{2}$  cents per pound.

Zinc in sheets.—Present rate,  $2\frac{1}{2}$  cents per pound; proposed rate,  $2\frac{1}{2}$  cents per pound.

Zinc, old and worn.—Present rate,  $1\frac{1}{2}$  cents per pound; proposed rate,  $1\frac{1}{4}$  cents per pound.

The other alterations of interest to our readers are those of the different classes of acids, &c., as follows:—

Acids.—Present rate, 2 cents per pound; proposed rate,  $1\frac{1}{2}$  cents per pound.

Acids.—Present rate, 10 cents per pound; proposed rate, 4 cents per pound.

Acids.—Present rate, 4 cents and 5 cents per pound; proposed rate, 5 cents per pound.

Acids.—Present rate, 15 per cent. *ad valorem*; proposed rate, 6 cents per pound.

Blue vitriol, &c.—Present rate, 3 cents per pound; proposed rate, 2 cents per pound.

Zinc oxide, &c.—Present rate,  $1\frac{1}{4}$  cents and  $1\frac{3}{4}$  cents per pound; proposed rate,  $1\frac{3}{4}$  cents per pound.

Sulphate soda.—Present rate, 20 per cent. *ad valorem*; proposed rate,  $\frac{1}{10}$  cent per pound.

Sulphur.—Present rate, 10 dols. per ton and 20 dols. per ton; proposed rate, 8 dols. per ton and 10 dols. per ton.

Sulphur, refined.—Present rate,  $\frac{3}{10}$  cent per pound, proposed rate,  $\frac{1}{2}$  cent. per pound.

Fire-brick.—Present rate, 20 per cent. *ad valorem*; proposed rate, 1.25 dols. per ton.

Cement, Roman, Portland and hydraulic.—Present rate, 20 per cent; proposed rate, 8 cents per 100 pounds.

Lime.—Present rate, 10 per cent.; proposed rate, 6 cents per 100 pounds.

Plaster of Paris, ground.—Present rate, 20 per cent., 1 dol. per ton; calcined, proposed rate, 20 per cent., 1.75 dols.—*Eng. and Min. Journal, N.Y.*

#### THE INCREASED DUTY ON SPIRITS.

Subjoined is the actual text of Mr. Goschen's proposals in regard to the increased duty upon spirits:—

That, in addition to the duty of Excise payable under the Act of the twenty-third and twenty-fourth years of Her Majesty's reign, chapter 129, for and upon every gallon computed at proof of spirits distilled in the United Kingdom, there shall be charged and paid the duty of sixpence, and so in proportion for any less quantity.

That, in addition to the duties of Customs payable on spirits under the Customs and Inland Revenue Act, 1881, there shall be charged and paid the duties following, that is to say:—

	£	s.	d.
For every gallon computed at proof of spirits of any description as in the said Act mentioned (except perfumed spirits) .....	0	0	6
For every gallon of perfumed spirits .....	0	0	9
For every gallon of liqueurs, cordials, and other preparations entered so as to be chargeable under the said Act with the duty of 1s. ....	0	0	8

And so in proportion for any less quantity.

And the duties of Customs on the articles herein-after mentioned, being articles of which spirits are a part or ingredient, shall be proportionately increased, and shall be as follows:—

	£	s.	d.
Chloroform, the pound .....	0	3	1
Collodion, the gallon .....	1	5	0
Ether acetic, the pound .....	0	1	10
Ether butyric, the gallon .....	0	15	8
Ether sulphuric, the gallon .....	1	6	2
Ethyl iodide of, the gallon .....	0	13	7

And so in proportion for any less quantity.

#### EXTRACTS FROM DIPLOMATIC AND CONSULAR REPORTS.

##### MEXICO.

##### Drugs for Guadalajara.

The greater part of the drugs consumed in this important Mexican State are purchased from Germany, England, France, and the United States, although at present a few factories of pharmaceutical chemicals exist in Mexico, which turn out certain articles, principally acids. The largest pharmacists in Guadalajara also manufacture themselves numerous articles; for instance, nitrate of silver and gold salts, as well as a large variety of patent medicines and extracts. In the manufacture of the latter the native sugar-cane alcohol, testing 70°, is employed. Most of the leading pharmaceutical articles are obtained from Germany, and these include glassware, chemical balances, and other apparatus, antiseptic bandages, painters' brushes, coal-tar soap, salicylic acid and salicylate of soda, quinine, cocaine, camphor, borax, pepsine, iodides, opium preparations, caffeine, and vaseline. Essential oils come from Germany, but perfumeries mostly from France and the United States. The United States also supply india-rubber goods, fluid extracts, patent medicines, gelatin capsules and pills, Florida water, liquid paints and varnishes, and sulphuric acid, the latter in competition with that made in Mexico. They also furnish complete fittings for pharmacies. From England come heavy chemicals, quinine, and chloroform, the better classes of india-rubber goods, Windsor soap, scents, and surgical instruments. From France are received patent medicines, fine glassware, bandages, surgical and optical instruments, medicinal wines, quinine, Lourdes water, and many patent medicines and perfumes. — *Chemist and Druggist.*

#### MISCELLANEOUS TRADE NOTICES.

##### THE PRODUCTION OF ALFA IN ALGERIA.

The *Journal Officiel* for the 24th February has an article upon Algeria, in which the following passages, having reference to the uses and production of alfa, occur:—

Alfa, from time immemorial, has been utilised for its fibres. At the present day, alfa is employed in various ways. It is used for cordage and basket-ware; mats, brooms, shoes, &c. are made from it, or again, cords, thick carpets, as also hangings and curtains; the purified fibres can be converted into celluloid; there is obtained from it an excellent pulp for paper making, which is used pure or mixed with the pulp of rags, wood, or straw. It is well known that paper makers, finding a difficulty in obtaining the quantity of rags necessary for an increasingly large manufacture, have had recourse to a large number of vegetable fibres, which have rapidly grown in favour, namely, alfa, straw, wood, and jute.

Paper manufactured with alfa is supple, silky, tenacious, transparent, and very pure. It is, for the same weight, much thicker than any other kind. It acts very well for printing on, a quality much desired; it is also perfectly convenient for *editions de luxe* and for engravings.

Many of the best illustrated journals print on alfa paper, which is also used to make an excellent letter paper. An addition of alfa pulp greatly improves pure straw paper.

England consumes the largest part of the material furnished by the alfa countries, that is to say, Algeria, Spain, Tunis, Tripoli, and Morocco. Of the 225,000 tons of this product yielded in 1885, England took for her share nearly 200,000, and of this quantity of 225,000 tons exported from the alfa countries, the paper industry has taken 210,000, the other 15,000 have been used in the manufacture of cordage and basket-ware.

France uses very little alfa, because of the high cost of transport of chemical products and of coal.

In Algeria, the province of Oran has always preserved the alfa monopoly. At first, it was confined to the coast district, and the fields of alfa were at little distance from the sea; now the inland railways permit of an economic transport.

The value of the exportation of alfa from Algeria at present is estimated annually at 10 million francs. It is stated that on completion of all the ways of communication, Algeria will be able to produce annually 400,000 tons. Thus, in the department of Algeria, in the military territory, more than 6,000 hectares of alfa were wasted because of the difficulty of transport; this means a loss of 120,000 tons of the raw material.—*Board of Trade Journal*.

#### ASPHALT AND PETROLEUM IN MEXICO.

The proofs of the existence of petroleum in certain districts consist of two kinds, viz., large deposits of asphaltum (called here "chapopote") formed by the residue of oil, and flowing springs of the crude oil itself in a state of more or less density. The belt of country over which indications of oil extend is immense; in fact, the whole Atlantic coast of Mexico shows traces of it. But while these localities are distant from the coast and situated in a mountainous country in the southern part—for instance, in the State of Tabasco—in the northern portion of the Republic the deposits and springs are found on those extensive plains between the sea-coast and the foot of the mountains, very near to the sea and in most cases close to or on the banks of navigable rivers, such being the case in the district to which we refer, which must certainly be called the most important of all those around the ports of Tuxpam and Tampico. In these districts the deposits of asphaltum are of immense size and so favourably located that with a few miles of river navigation the open sea is reached. In some instances large vessels could go up almost to the spot; and in one case the deposits are in the immediate vicinity of the Tampico branch of the Mexico Central Railway. These deposits of asphaltum are of such pure composition that during the night they condense into a certain state of glossy hardness, whilst during the daytime the sun renders them so soft that the cattle passing over them sink down. The commercial value of this asphaltum has not been ascertained as yet, but it has been shipped with profit, in small lots, at various times, to the States and even to Europe, by the merchants of the coast. Its breaking or cutting on the spot amounts to next to nothing, and it can be thrown into the river and floated down to the sea-coast, where it may be collected again and loaded on the vessels. Some of the springs where crude petroleum runs out freely are situated on the very banks of rivers, the oil flowing into the same and covering the surface of the water for a great distance. A few samples of the different kinds of crude oil, some of which have been assayed in Pennsylvania, have turned out to be of a quality at least equal to the crude produced in that State. Some of these springs have a natural flow of a stream three inches in diameter.

Again, in the same districts, there exist in several places deposits or veins of a certain bituminous coal, which, upon careful examination by competent authorities, proved to be a very good class of "grahamite," which kind of coal has so far, if we are correctly informed, only been found in New Brunswick and somewhere in Virginia, which deposits are said to have given out. Its value is much greater than anthracite coal—perhaps three times as much—because of its superior qualities for the manufacture of gas. There is especially one deposit existing a few miles up the river from the city of Tampico, which, although not developed to any extent, has enough coal in sight to prove it to be an important deposit, worthy of careful investigation and further development.—*Ibid*.

#### REPORTS FROM BAKU.

Chem. Zeit. 14, 8.

According to the report of the Caucasian administration, the production of copper in 1888 amounted to 93,385 puds 17 lb.

On the 1st (13th) November there were in the peninsula Apochewn 432 productive naphtha borings; 231 in action,

110 at the time not in action, and in the case of 92 boring was going on. Four of the borings were in action as fountains. The maximum depth was 358½ metres, minimum depth 49 metres, average 192½ metres. The Government has offered to buy Nobel's naphtha-duct between Michailas and Kvirili.—N. H. J. M.

#### THE CONSUMPTION OF CUTCH.

The destruction of the cutch trees has been proceeding at such a rate during the last few years that the Government of British Burma has thought it necessary to adopt special measures for their protection.

During the last season cutch was also greatly mixed with a spurious article called "than," which deteriorated the quality considerably. The Government has, therefore, strictly prohibited the felling of "than" trees, and the market will not therefore be swamped this season by such inferior parcels as have accumulated in London and Liverpool, and now form a large portion of the stock there. The natives had also been cutting down the cutch trees indiscriminately, thus spoiling the forests. The Government, with a view to remedying this evil, have in some districts almost entirely withdrawn the licences from the native boilers, and only granted a very limited number in others.

These measures are reducing the output of cutch this season, and so far the shipments from Rangoon are less than one-third of those of 1889 and 1888.

The English stocks have yearly increased since 1887, owing to the vast amount of inferior cutch imported, which is unsuitable for most manufacturing purposes; but this nominally very large stock contains hardly 10 per cent. of really good consuming cutch.

The average consumption of cutch during the last four years is calculated to amount to 14,118 tons per annum.—*Ironmonger*.

#### THE OKRA PLANT AS A SUBSTITUTE FOR HENEQUEN.

Considerable attention is being directed to the okra plant, an annual of South American origin, which, heretofore, has been known chiefly, says the *South American Journal*, as furnishing an excellent vegetable for soups. It has recently been discovered, however, that this plant has an excellent fibre, and an American citizen asserts that he has invented a process for extracting okra fibre at a cost not exceeding 1 cent a pound. It is believed that okra fibre, especially if extracted at this price, will largely displace jute, and it is also likely to come into competition with henequen.—*Board of Trade Journal*.

#### PRODUCTION OF IRON IN INDIA.

See *Board of Trade Journal* for April, p. 465, quoting *Times of India* of February 21st, 1890.

#### SUGAR IN INDIA.

See *Journal of the Society of Arts*, April 4th, 1890, p. 495.

#### IMPORTANT PATENT DECISION.

The decision announced by the Supreme Court of the United States in the case of Pohl v. The Anchor Brewing Company is one of far-reaching consequences. The text of the decision is not yet printed, but the announcement is sufficiently full to show that the Supreme Court has held—overruling the Circuit Court of the Southern District of New York—that United States patents issued for inventions which have been previously patented abroad do not terminate when the foreign patents may happen to come to an end as a penalty for non-payment of taxes, importation in contravention to foreign laws, or other breaches of conditions subsequent on which those patents are issued.

The Westinghouse and Edison Companies have been in litigation for a long time on questions involving the one decided in this case, which is upon a patent for the washing of beer barrels. This case was availed of by the Edison and Westinghouse Companies, they having none of their own sufficiently forward to reach an early decision, and it was argued by their respective counsel. A judgment was rendered by Judge Wallace overruling his former decision

under the authority of *Bates v. Hammond*, which he interpreted as requiring him—apparently contrary to his convictions of what the decision ought to be—to declare the beer barrel patent expired here because of its forfeiture in Germany. The appeal from that decision was submitted out of its order under a rule of the court upon briefs signed by Mr. Grosvenor P. Lowrey, Mr. Benjamin F. Thurston, Mr. Clarence A. Seward, Mr. J. M. Denel, and Judge Davis on the one side, and by Mr. Wetmore, Witter and Kenyon, and Hess and Townsend upon the other. The fulness of the consideration which this question has now had seems to settle the law upon this point, and leave it certain that American patents can suffer no detriment by reason of those foreign patents, except to be limited to the term for which the foreign patent issues. Nothing done after that time by the foreign owner can prejudice the American interest.

It is believed that this decision is far-reaching to a degree that can scarcely be estimated. To the Edison Company alone the result is deemed of the greatest importance, since it is claimed by them that it rids them of the only serious difficulty in the way of establishing the principal Edison lamp patent; and doubtless other patents of a value nearly as great in various branches of industry which were compromised while this matter remained in doubt are now secured.—*Engineering and Mining Journal*.

#### THE ZINC WHITE INDUSTRY IN GERMANY.

The high prices of zinc have seriously restricted the trade in zinc white. The total production of the Silesian district last year amounted to only 715 tons, against 2,369 tons in 1888. Among the substitutes for zinc white, zinc dust, which is sold at three-fourths the price of the metal, is being more and more largely used. Another substitute, soapstone, used from time immemorial in China and Japan for painting metal-work, is also growing in favour in Germany.—*Industries*.

#### ANTIMONY ORES IN SPAIN.

The first lot of antimony ore, of great yield, from the Zalamea de Serena mines, in Estramadura, has been despatched to England. A large sample of this ore, sent to Germany, was analysed, and found to contain 15 grms. of gold per ton. As a rule, all the ores of that place, both antimony and silver lead, have shown more or less gold, but the company do not yet look upon their property as a gold mine.—*Ibid*.

#### THE NEW CALEDONIA MINES.

The New Caledonia mines, according to official reports, produced during the last fiscal year 8,423 tons of nickel ore, worth 125 frs. a ton; 3,020 of cobalt ore, worth 80 frs.; 236 of copper, at 300 frs.; 500 of lead, at 150 frs., and 2,475 tons of chrome ore, worth 40 frs. a ton.—*Chemist and Druggist*.

#### THE EXPORTS OF CHEMICALS.

The exports of chemicals from the Tyne for the first quarter of this year show a considerable increase in tonnage over the exports for the corresponding quarter of last year.

	1889.	1890.
	Tons.	Tons.
Alkali and soda ash .....	2,702	3,704
Bicarbonate of soda .....	26	24
Bleaching powder .....	3,911	6,424
Manure .....	1,963	2,955
Soda crystals .....	2,980	3,303
Sulphate of soda .....	187	1,107
Other chemicals .....	3,761	4,911
Total .....	15,539	22,428

Being a total increase of 44 per cent.

#### GLAZING A BLAST FURNACE LINING.

An experiment was tried at the Crown Point Iron Company's plant, Lake Champlain, N.Y., some time ago, on the occasion of their relining and starting in blast one of its large furnaces. After the fire-bricks were in place, a cheap article of graphite or plumbago, supplied by the Joseph Dixon Crucible Company, of Jersey City, N.J., was bought, reduced to a paste with water, and the interior of the furnace washed with the plumbago paste. It gave a slippery gloss to the fire-brick lining which refused to be coated with slag, and the charge passed down in less time and left the lining free and clear. The Crown Point managers claim quite a success for their scheme.—*Eng. and Min. Journal*, N.Y.

#### SALT SPRING IN THE MANCHESTER SHIP CANAL.

*Industries* states that a brine spring has been discovered in the cutting of the Manchester Ship Canal near Partington. The water from the spring is being tested in order to ascertain its strength and the probable richness of the assumed salt beds.

#### MINERALS IN SOUTH AFRICA.

The Transvaal appears to be very rich in minerals other than gold, and if we may place implicit reliance upon the statements recently made by Mr. W. H. Furlonge, of Johannesburg, it possesses most of the elements of success for the making of iron and steel. Mr. Furlonge remarks the almost entire absence of lime throughout the country, which would be very prejudicial to blast-furnace work, but silica is most abundant, and oxides of iron are to be found everywhere amongst the rocks. Many of the "bars" of oxides, containing 30 to 40 per cent. of iron, are alongside deposits of auriferous quartz, and the ironstone itself carries gold, so that it is one of the possibilities of the future Transvaal iron trade that gold may be raised with iron instead of being made out of it! In addition to these "bars" of ironstone, there are amongst the schists and shales of the De Kaap district of the Transvaal enormous deposits of iron ore, some of the stratified beds being hundreds of feet thick, and of almost pure hematite, with some magnetite. Mr. Furlonge says that some, at least, of these are Bessemer ores, and he predicts that the Transvaal will never be at a loss for iron ores. There is coal in inexhaustible quantities in the High Veldt, within about seventy miles of the iron-ore deposits, consequently it seems to be only a question of time for the two to be used in conjunction for the production of iron, or even of steel, by the pneumatic or open-hearth process. Of other minerals—lead, mercury (cinnabar), nickel, cobalt, copper, and antimony—the Transvaal has plenty, and Mr. Furlonge, who is a practical geologist, hints at the growth of a large silver-mining industry, seeing that some of the galena on the High Veldt contains silver up to as high as 600 oz. to the ton. The great want of this rich mineral country is railways, and as soon as they are made on a proper scale we may anticipate considerable developments on all sides.—*Ironmonger*.

#### EXTRACTS FROM GEIHE AND CO.'S REPORT.

##### Acid (Carbolic).

In Germany the use of carbolic acid has grown very considerably in the last few years, since this acid has been used more frequently than formerly in the preparation of stores for war. The home production has not been at all equal to fill the increased demands, but the assistance of foreign makers had to be invoked, with the result that the imports advanced from 698,200 kilos. in 1887 to 1,191,800 kilos. in 1888 and 2,432,100 kilos. last year. Of the 1889 imports, 2,267,800 kilos. came from England. The interest in synthetically-made carbolic acid has perceptibly fallen off since it became clear that the new article shared with the old one the fatal defect of reddening. It is true that the synthetic acid possesses some few advantages over the coal-tar product, but not sufficient to compensate for the important difference in price, particularly as the Imperial Department of Health has ascertained that the disinfecting properties of both acids are equal.

*Ambergris.*

It is but natural that the excessive prices demanded for this article should have had the effect of diminishing the demand, but however dear it may be there are always perfumers who will not do without this favourite article. If we consider, however, that by a suitable treatment of the combinations belonging to the aromatic acid series it has been possible to obtain a substitute for musk, it does not seem beyond the reach of possibility to manufacture synthetic ambergris also, the natural product consisting principally of a peculiar non-saponifiable fat. Such a discovery would have the result of putting an effectual limit to such exorbitant quotations as are now made, while the discoverer would be sure to reap a material reward from the constant demand of this article, which does not depend upon fashion.

*Borax.*

The present year has not brought any alterations in the price of borax, and so long as the union of the manufacturers continues to exist the market will retain its stability. Whether this combination can be maintained in future is questionable, because the German production grows every year at the expense of the British output. The German Imperial statistics show an increase in the exports of borax and boracic acid from 800,900 kilos. in 1887, to 1,172,500 kilos. in 1888, and 1,664,200 kilos. in 1889.

*Camphor.*

The large Hamburg refineries are now sold out, and it is difficult to form an opinion of the future of the article, as the principal consuming time is now approaching. It is also impossible to indicate the true causes of the advance. It is a fact that the consumption of camphor has enormously increased in consequence of its use in industrial purposes, both in blocks and in powder, while the imports remained small, and there is no means of judging whether and when it will again increase. The annual consumption of raw camphor in the United States is said to have averaged 2,100,000 lb. during the last four years, while the importation last year decreased by 1,200,000 lb. This deficiency has to be provided for from elsewhere, and this conclusion agrees with the reports spread from Hamburg that one large firm in the States will not only require for this year a surplus of 600 kegs of refined camphor over its usual consumption, but has not even covered as yet the principal part of its usual requirements of 800 kegs. As regards the question whether the supplies of raw camphor are kept back artificially in Japan, or whether the production has really diminished in the same measure as the supply, has hitherto been considered as an unsolved question; but the alleged diminution of the production is a favourite explanation of the smallness of the quantity offered. According to reports from an official source in Japan, it is quite possible that there may have been some limitation of the production, but the reduction in the quantity offered is entirely the result of the manoeuvres of native speculators in the primary markets. But already, last year, it was said that the advance in the price of raw camphor was caused by the wholesale destruction of trees by the natives. As regards the speculative rushing up of prices since the middle of February, that movement is said to have been caused by the fact that certain Japanese firms had requested their buyers in Europe to cancel existing contracts against payment of heavy damages, their requests being partly successful. The consequence of these cancellings was, of course, that the buyers had to cover their own contract sales of refined camphor, producing thereby a considerable excitement in the market. In view of the present elevated prices of camphor, it would not be surprising if experiments were to be made to find a substitute for it in the manufacture of smokeless gunpowder. To that purpose, about 8 per cent. of camphor was required, in addition to about 72 per cent. of nitro-glycerin and 20 per cent. of pyroxylin. The idea may be brouched to manufacture on a cheap scale, and for experimental purpose hydrochloric acid camphor, which in former years had a short run as artificial camphor. Perhaps the strong evolution of gas from this compound might render it specially suitable (?) for this purpose.

*Phosphorus.*

The dissolution of the union for the fixing of the phosphorus prices between the English and the French manufacturers has brought about a struggle of the respective works for the possession of the German and Austrian markets, which has resulted in a reduction in the quotations. The German Imperial statistics show that in 1889 the English makers succeeded in maintaining the field against their French competitors; but, on the other hand, the competition of the Russian works is becoming somewhat more perceptible. The imports of white and red phosphorus in 1889 were 266,800 kilos., against 136,300 kilos. in 1888. Of the former quantity, 212,500 kilos. came from England, 38,300 kilos. from Russia, and 2,100 kilos. from France.—*Chemist and Druggist.*

## BOARD OF TRADE RETURNS.

## SUMMARY OF IMPORTS.

	Month ended 31st March	
	1889.	1890.
Metals.....	£ 1,936,125	£ 1,929,528
Chemicals and dyestuffs.....	1,206,573	1,121,999
Oils.....	497,787	538,800
Raw materials for non-textile industries.....	2,638,199	2,605,441
Total value of all imports ....	36,265,387	36,140,334

## SUMMARY OF EXPORTS.

	Month ended 31st March	
	1889.	1890.
Metals (other than machinery) ....	£ 3,199,327	£ 3,493,151
Chemicals and medicines .....	680,477	711,152
Miscellaneous articles.....	2,940,056	2,800,032
Total value of all exports.....	21,459,490	20,007,922

## EXPORTS OF METALS FOR MONTH ENDED 31ST MARCH.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Copper:—			£	£
Ore..... Tons	10,433	12,507	112,364	113,943
Regulus .....	9,222	6,403	306,880	186,168
Unwrought ....	3,127	3,631	183,916	184,504
Iron and steel:—				
Iron ore .....	439,877	426,382	338,377	356,121
Iron bolt, bar, &c. ..	5,895	4,076	49,933	38,852
Steel, unwrought..	354	536	3,682	6,469
Lead, pig and sheet ..	12,874	13,245	168,601	172,428
Pyrites .....	59,138	63,248	112,967	112,147
Quicksilver .....	494,367	26,250	48,883	3,194

EXPORTS OF METALS FOR MONTH ENDED 31ST MARCH—  
*continued.*

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Tin..... Cwt.	50,455	51,330	£ 224,639	£ 250,085
Zinc..... Tons	3,720	3,925	69,103	88,206
Other articles... Value £	..	..	316,789	417,351
Total value of metals	..	..	1,936,125	1,929,528

IMPORTS OF CHEMICALS AND DYESTUFFS FOR MONTH  
ENDED 31ST MARCH.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Alkali..... Cwt.	5,868	1,940	£ 2,662	£ 1,946
Bark (for tanners, &c.) .....	32,021	39,427	15,385	16,647
Brimstone .....	67,725	12,578	14,712	9,546
Chemicals..... Value £	..	..	99,137	110,325
Cochineal .....	589	500	3,577	3,012
Cutch and gambier Tons	2,175	1,300	56,984	37,257
Dyes:—				
Aniline .....	..	..	21,737	21,685
Alizarine .....	..	..	29,687	29,598
Other .....	..	..	1,405	774
Indigo .....	24,366	19,407	514,307	348,634
Madder .....	1,402	1,187	1,727	1,512
Nitrate of soda....	311,440	659,672	165,515	269,674
Nitrate of potash .	37,510	32,017	31,460	28,260
Valonia .....	3,896	2,783	56,133	55,326
Other articles... Value £	..	..	192,145	187,803
Total value of chemicals	..	..	1,206,573	1,121,909

## IMPORTS OF OILS FOR MONTH ENDED 31ST MARCH.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Cocoa-nut..... Cwt.	21,620	19,799	£ 27,943	£ 25,099
Olive .....	1,740	2,568	63,957	97,351
Palm .....	69,475	72,069	71,931	74,771
Petroleum .....	8,042,009	6,609,239	183,310	173,178
Seed .....	967	1,389	27,682	40,876
Train, &c..... Tons	1,207	829	26,304	17,054
Turpentine .....	12,850	23,969	22,664	36,993
Other articles .. Value £	..	..	73,996	78,478
Total value of oils ...	..	..	497,787	538,800

IMPORTS OF RAW MATERIALS FOR NON-TEXTILE  
INDUSTRIES FOR MONTH ENDED 31ST MARCH.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Bark, Peruvian .. Cwt.	13,672	12,670	£ 18,596	£ 19,336
Bristles..... Lib.	209,990	201,052	33,383	29,190
Caoutchouc..... Cwt.	24,317	27,720	299,194	338,016
Gum:—				
Arabic.....	6,055	4,058	24,492	13,296
Lac, &c.....	11,954	12,839	40,010	49,059
Gutta-percha ....	3,119	5,240	29,113	70,753
Hides, raw:—				
Dry.....	63,212	48,703	175,877	127,016
Wet.....	43,344	51,980	101,857	118,493
Ivory .....	380	922	17,531	46,429
Manures:—				
Guano..... Tons	3,594	5,430	23,646	29,502
Bones.....	12,670	6,750	62,159	35,664
Paraffin..... Cwt.	21,679	51,699	27,738	65,258
Linen rags..... Tons	4,328	3,231	42,707	33,534
Esparto, &c. ....	26,547	16,945	136,157	82,079
Pulp of wood ....	8,107	9,507	48,396	56,111
Rosin..... Cwt.	192,453	167,223	39,675	35,674
Tallow and stearin ..	75,383	144,929	99,582	179,691
Tar .....	4,894	7,169	3,984	4,729
Wood:—				
Hewn .....	112,875	136,810	266,858	352,386
Sawn .....	108,165	84,232	276,760	219,404
Staves .....	6,245	5,599	25,041	35,796
Mahogany .....	4,450	5,455	39,896	51,154
Other articles... Value £	..	..	836,447	690,871
Total value .....	..	..	2,698,119	2,895,441

Besides the above, drugs to the value of 74,967, were imported, as against 71,041, in 1889.

EXPORTS OF METALS OTHER THAN MACHINERY FOR  
MONTH ENDED 31ST MARCH.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Brass..... Cwt.	7,879	7,429	£ 42,231	£ 36,620
Copper:—				
Unwrought .....	14,934	60,942	56,530	102,468
Wrought.....	10,660	21,430	47,538	69,631
Mixed metal ....	12,490	17,992	41,292	51,725
Hardware .....	..	..	274,930	231,231
Implements.....	..	..	101,267	107,278
Iron and steel..... Tons	345,274	324,721	2,425,670	2,546,610
Lead .....	5,300	4,060	77,680	62,859
Plated wares... Value £	..	..	32,956	37,904
Telegraph wires... ..	..	..	285,993	51,390
Tin .....	9,049	7,976	44,351	37,970
Zinc .....	7,989	12,397	5,796	11,841
Other articles .. Value £	..	..	63,984	85,624
Total value .....	..	..	3,499,327	3,493,151



## EXPORTS OF DRUGS AND CHEMICALS FOR MONTH ENDED 31ST MARCH.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
			£	£
Alkali..... Cwt.	528,296	417,209	131,292	128,492
Bleaching materials ..	113,494	109,129	43,370	29,417
Chemical manures. Tons	26,598	28,211	191,812	189,807
Medicines..... Value £	..	..	92,250	92,459
Other articles ... ..	..	..	228,744	270,986
Total value .....	..	..	680,477	741,152

## EXPORTS OF MISCELLANEOUS ARTICLES FOR MONTH ENDED 31ST MARCH.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
			£	£
Gunpowder..... Lb.	756,260	948,600	17,905	23,435
Military stores.. Value £	..	..	86,989	106,505
Candles..... Lb.	833,400	1,256,800	16,418	23,016
Caoutchouc .... Value £	..	..	98,392	94,297
Cement..... Tons	66,026	57,655	127,423	117,603
Products of coal Value £	..	..	96,750	133,351
Earthenware ... ..	..	..	194,828	139,641
Stoneware ..... ..	..	..	18,128	17,699
Glass:—				
Plate..... Sq. Ft.	378,568	256,550	22,700	12,642
Flint..... Cwt.	11,912	12,337	26,765	23,423
Bottles..... ..	82,033	75,276	38,696	35,011
Other kinds.... ..	17,995	13,674	14,336	11,963
Leather:—				
Unwrought .... ..	13,458	12,734	123,130	115,482
Wrought ..... Value £	..	..	37,180	29,307
Seed oil..... Tons	6,446	5,673	133,273	129,985
Floor cloth ..... Sq. Yds.	1,549,800	1,608,900	72,859	70,081
Painters' materials..... Value £	..	..	140,272	132,839
Paper ..... Cwt.	90,489	83,313	159,955	147,389
Rags..... Tons	4,920	3,427	41,812	26,470
Soap..... Cwt.	35,577	40,186	40,798	45,792
Total value .....	..	..	2,940,056	2,800,032

## TRADE BETWEEN SPAIN AND THE UNITED KINGDOM.

## IMPORTS INTO THE UNITED KINGDOM FROM SPAIN FOR THE THREE MONTHS ENDED 31ST MARCH.

Principal Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
			£	£
Chem. products unenumerated Value £	..	..	12,469	8,471
Copper ore and regulus ..... Tons	25,229	15,829	399,853	271,731
Iron ore ..... ..	990,734	1,138,142	703,911	907,027
Lead ore..... ..	170	320	1,260	2,870
Lead, pig and sheet ..... ..	18,913	19,174	242,233	251,201
Manganese ore.. ..	1,430	2,350	2,860	5,250
Olive Oil..... Tons	1,082	484	36,592	18,287
Pyrites of iron and copper ... Tons	201,643	156,487	493,593	305,730
Quicksilver ..... Lb.	1,389,555	1,534,914	151,403	209,010
Rags, esparto ... Tons	21,704	20,687	133,196	118,102
Zinc ore ..... ..	4	577	25	1,841
Total value .....	..	..	3,079,821	3,162,431

## EXPORTS OF HOME PRODUCE FROM THE UNITED KINGDOM TO SPAIN FOR THE THREE MONTHS ENDED 31ST MARCH.

Principal Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
			£	£
Alkali..... Cwt.	76,216	64,686	22,679	22,519
Caoutchouc manufactures .... Value £	..	..	4,123	3,233
Cement..... Tons	1,350	1,483	2,180	2,893
Chemicals (including dye-stuffs) ..... Value £	..	..	20,691	49,034
Products of coal .. ..	..	..	13,256	17,942
Glass manufactures ..... ..	..	..	1,667	1,629
Manure..... ..	..	..	60,294	52,456
Oil, seed ..... Tons	131	135	2,640	3,067
„ other sorts.. Value £	..	..	2,843	9,235
Painters' colours &c. .... ..	..	..	7,657	7,688
Paper of all sorts Cwt.	1,388	1,183	3,799	2,951
Sap..... ..	201	437	249	547
Oil and floor cloth ..... Sq. yds.	89,109	88,609	3,791	3,761
Total value .....	..	..	1,080,138	1,270,562

## Monthly Patent List.

\* The dates given are the dates of the Official Journals in which acceptances of the Complete Specifications are advertised. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the said dates.

### I.—GENERAL PLANT, APPARATUS, AND MACHINERY.

#### APPLICATIONS.

4225. J. von Langer and L. Cooper. Improvements in or connected with valves for hot gases. March 18.

4309. J. Foster. Improvements in and relating to evaporating and distilling apparatus. March 19.

4354. A. Eickhorn and R. Jaspar. Improvements in hydrometers. Complete Specification. March 20.

4532. E. G. Lawrence.—From S. Pick, Galicia. See Class VII.

4562. J. Coppard. Improvements in the construction of appliances for extracting the juices from fruits and other substances. March 24.

4594. E. Jones. Improved apparatus for separating liquids or solids from vapours or gases in which they are suspended. March 24.

4802. G. C. Topp. Improvements in hydrometers, saccharometers, and lactometers. March 27.

4822. H. A. A. Dombrain. See Class XX.

5038. H. Trott. Improvements in apparatus for operating valves for controlling the passage of fluids. April 1.

5142. E. Theissen. Improvements in apparatus for condensing steam and other condensable vapours or gases. April 2.

5267. A. Chapman. Improvements in the construction and working of double, triple, and multiple effect evaporating apparatus. Complete Specification. April 5.

5419. J. Guillaume. Improvements in the treatment of organic matters. Complete Specification. April 9.

5598. H. A. Fleuss. Improvements in freezing or refrigerating apparatus. April 12.

#### COMPLETE SPECIFICATIONS ACCEPTED.\*

1889.

5592. W. L. Wise.—From Escher Wyss and Co. Manufacture of clear ice, and apparatus therefor. April 2.

6391. W. Hucks. Method and means for separating solids from liquids. April 15.

8184. J. J. Hicks. Improvements in brewers' and other thermometers. March 26.

8687. E. F. Varaldi. Vacuum pumps. March 26.

8882. W. P. Branson and H. W. Neild. See Class VIII.—A. April 9.

9354. F. J. Brough. — From S. Jonsson. Centrifugal separating machines. April 9.

9375. G. King. Fastenings for retort lids. April 2.

1890.

1910. J. F. H. Gronwald. Sterilising apparatus. April 2.

### II.—FUEL, GAS, AND LIGHT.

#### APPLICATIONS.

4197. H. L. Müller and W. Adkins. Improvements in apparatus for the production of lighting gas. March 18.

4217. L. van Vestrout. Improvements in apparatus for charging inclined gas retorts. March 18.

4223. J. von Langer and L. Cooper. Improvements relating to the production of water-gas and generator-gas, and to apparatus connected therewith. March 18.

4224. J. von Langer and L. Cooper. Improvements in apparatus for the generation and enrichment of water-gas and producer-gas. March 18.

4236. W. E. Smith.—From S. T. J. Bray, Russia. Improvements in apparatus for burning liquid fuel. March 18.

4324. J. A. Kehuan. Improvements in apparatus connected with the manufacture of gas. March 19.

4326. C. A. Sahlström and A. F. Hill. An improved burner for liquid fuel. March 19.

4355. D. Hancock and J. B. Craig. Improvements in carburated air-gas produced from mineral oils or spirits. March 20.

4374. F. R. Engledow and The Engineering Joint Stock Company. Improvements in heating and heating apparatus. March 20.

4457. E. Edwards.—From F. Strohmer, Germany. Improvements in coke ovens and in the method of using the same. March 21.

4542. T. C. Fawcett and J. B. Swallow. A new manufacture of briquette or block fuel. March 24.

4598. W. Gray. Improvements in the manufacture of gas. March 24.

4708. R. M. Whitaker. Improvements in apparatus for utilising the waste heat from steam-boiler and other furnaces, and applying it to the heating of air to support combustion and consume smoke in those furnaces. March 26.

4928. The Manchester Oxygen Company, Lim., and W. M. Jackson. Improvements in the mode of using compressed gases for illuminating purposes. March 29.

4936. S. Girding. Improvement in a mantle or filament holder and burner for incandescent gas lamps. March 29.

5078. C. Hunt. Improvements in inclined gas retorts and in apparatus for charging the same. April 1.

5163. S. J. Woodhouse. Improvements in the mode and means employed for charging and discharging retorts used in the manufacture of gas. April 3.

5194. G. Waller. Improvements in apparatus for cooling and conveying coke discharged from gas retorts into suitable receptacles or places. April 3.

5239. J. Blum. The instantaneous production of water-gas. April 5.

5243. H. Fourness. Improvements in apparatus for carburetting water-gas. April 5.

5434. H. Williams. Improvements connected with the manufacture of water-gas and other gases for lighting and manufacturing purposes. April 10.

5453. W. Ackroyd, T. H. Ackroyd, and J. Willoughby. Improvements in the method of and apparatus for effecting or increasing the combustion of smoke in steam-boiler and other furnaces, and in the utilisation and transmission of heat produced therein, and for economising fuel and improving the draught. April 10.

5461. J. G. Hawkins and J. Barton. Improvements in vertical gas retorts, together with their lids, doors, and other attachments. Complete Specification. April 10.

5485. J. Westray. Improvements in apparatus for the better combustion of fuel, and supplying hot air to steam-boiler or other furnaces. April 11.

5543. E. Manbre. Improvements in the burning of liquid fuel, and in apparatus or furnaces therefor or appertaining thereto. April 12.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

9279. B. Loomis. Process and apparatus for manufacturing heating and illuminating gas. April 9.

9327. S. Fox and E. Blass. Manufacture of water-gas and apparatus therefor. April 9.

9454. G. Love. Apparatus for generating and consuming combustible gas. April 16.

11,456. N. Meiklejohn. Anti-dips for hydraulic mains employed in the manufacture of gas. April 9.

### III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

## APPLICATION.

4120. J. Laing. Improvements in the apparatus for destructive distillation of mineral oils at atmospheric pressure. March 17.

### IV.—COLOURING MATTERS AND DYES.

## APPLICATIONS.

4382. B. Willcox.—From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture of new derivatives of phenyl-amido-acetic acid. March 20.

4577. O. Inray.—From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. Improvements in the production of grey colouring matters. March 24.

4596. S. Pitt.—From L. Cassella and Co., Germany. The manufacture of sulphonated thionines. March 24.

4901. S. Pitt.—From L. Cassella and Co., Frankfurt-on-Maine. Improvements in the manufacture of dyestuffs. March 28.

4930. W. Hay, W. M. Alexander, and E. Bentz. Improvements in the preparation or admixture of indigo with certain substances for commercial and industrial purposes. March 29.

4946. O. Inray.—From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. Production of ethylether of cinnamic acid. March 29.

5155. The Clayton Aniline Company, Lim., and J. Hall. Improvements in the manufacture of colouring matters. April 2.

5455. J. Mills. A new or improved application of ceramic colouring matters, or colouring matters obtained from metallic oxides to india-rubber, gutta-percha, vulcanite, or other like substances or materials. April 10.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

7802. B. Willcox.—From The Farbenfabriken vormals F. Bayer and Co. Manufacture of azo-dyes and materials therefor. April 16.

7857. O. Inray.—From The Farbwerke vormals Meister, Lucius, and Brüning. Manufacture of a new colouring matter of the induline series. April 16.

7977. S. Pitt.—From L. Cassella and Co. Production of bluish black colouring matters. March 26.

8373. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik. Preparing colouring matters of the oxyketone group for dyeing and printing. March 26.

9428. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik. Preparing colouring matters of the oxyketone group for dyeing and printing. March 26.

9429. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik. Production of coloured compounds of galactophenone within or upon animal and vegetable fibres. March 26.

9612. J. Y. Johnson.—From F. von Heyden. Manufacture of beta-naphthol carbo acid. April 16.

9642. O. Inray.—From The Farbwerke vormals Meister, Lucius, and Brüning. Production of oxysulphonic acids and naphthalin. April 16.

9643. O. Inray.—From The Farbwerke vormals Meister, Lucius, and Brüning. Production of colouring matters from oxysulphonates of naphthalin.—April 16.

11,000. H. H. Lake.—From W. Oehler. Production of colouring matters. April 9.

12,549. T. R. Shillito.—From A. Feer. Manufacture of yellow to orange colouring matters.—April 2.

### V.—TEXTILES, COTTON, WOOL, SILK, Etc.

## APPLICATIONS.

4231. H. Smith, E. D. Smith, and H. D. Smith. Improvements in the method of and apparatus for washing wool and other fibres. March 19.

4452. H. A. Lowe. Improvements in the treatment of or finishing of manufactured or partially manufactured materials from cotton or other cellulosic fibres. March 21.

4958. T. Burrows. Means and machinery for removing the fibrous filaments from fibre-bearing leaves and stems of plants. March 29.

5271. J. Saleha. A new or improved process for cleaning and separating cotton from the cotton seeds. April 5.

5413. W. P. Thompson.—From H. Orval, France. Improvements in and apparatus for drying, cleaning, or otherwise treating wool. April 9.

5482. G. W. Arnott, P. A. Olivier, and G. Sengrave. An improved process for washing and scouring wool and fibrous materials containing grease. April 10.

5532. C. L. Field. A process for decorticating ramie and other fibrous stems. April 11.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

7058. B. W. Weatherdon.—From T. A. Compere. See Class IX.

7090. C. Dreyfus and J. Robinson. Manufacture of india-rubber or waterproof textile fibres and fabrics. April 9.

8106. V. Schevelin and P. Mindovsky. Treating vegetable fibres by acid, neutral, and alkaline residues of naphtha manufacture or specially prepared substances analogous to such residues. April 2.

### VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

## APPLICATIONS.

4409. J. O. Muller and C. O. Weber. Improvements in dyeing vegetable and certain animal fibres a fast aniline black, said fibres being either in the loose or manufactured state. March 21.

4450. W. Turnbull, W. Stockdale, and G. Duerr. Improvements in the process of mordanting cotton or other vegetable fibrous materials. March 21.

4610. G. Newsum. Improvements in apparatus for applying bronze and other powders to the surface of paper and the like. March 25.

4683. A. J. Boulton.—From A. Koblenzer and M. Koblenzer, Germany. Improvements in or relating to the dyeing of yarns. Complete Specification. March 25.

4854. W. E. Heys.—From E. Châtel, France. Improvements in machines for dyeing, bleaching, and similarly treating yarn and threads in cops and bobbins. March 28.

5054. C. Kellner. *See* Class XI.

5285. C. Kellner. Improvements in the process of and apparatus for bleaching vegetable fibres. April 5.

5385. W. E. Heys.—From E. Châtel, France. Improvements in apparatus for steeping, dyeing, and otherwise treating yarn and similar textile materials in cops and bobbins. April 9.

5547. B. Haigh. Improvements in dyeing and apparatus therefor. April 12.

#### COMPLETE SPECIFICATION ACCEPTED.

1889.

9149. S. Mason, jun. Apparatus for dyeing and otherwise treating wool. April 16.

### VII.—ACIDS, ALKALIS, AND SALTS.

#### APPLICATIONS.

4171. C. S. Negrier. Improved method and apparatus for concentrating sulphuric acid. March 17.

4394. F. Ricard. Improvements in the manufacture of soda by the ammonia process. March 20.

4434. J. J. Meldrum and T. F. Meldrum. Improvements in or connected with apparatus used in the distillation of ammonia. March 21.

4435. J. J. Meldrum and T. F. Meldrum. Improvements in or connected with apparatus used in the distillation of ammonia. March 21.

4473. H. H. Lake.—From La Société Anon. Jay et Jellathier, France. Improvements in apparatus for producing carbonic acid and similar gas. March 21.

4519. H. Precht. An improved process for the production of potash from potassium and magnesium carbonate. March 22.

4532. E. G. Lawrence.—From S. Pick, Galicia. Improvements in apparatus for evaporating liquors containing salts, and for separating such salts, when rendered insoluble by evaporation of the liquors. March 22.

4588. H. Precht. Improvements in the process or apparatus for the production of carbonate of magnesium and double carbonate of magnesium and potassium. March 24.

4652. J. C. Mewburn.—From C. C. Peck, United States. Improvements in purifying brine. Complete Specification. March 25.

4694. E. G. Lawrence.—From S. Pick, Galicia. Improvements in the manufacture or production of salts from brine. March 25.

4695. E. G. Lawrence.—From S. Pick, Galicia. Improvements in apparatus for drying salt. March 25.

4751. E. A. Cowper. Apparatus for producing fine salt from brine. March 26.

4759. H. Precht. An improved process for the production of hydrate of magnesia. March 26.

4780. B. H. Thwaite. The treatment of salt preparatory to its dechlorination by sulphurous acid. March 27.

4950. A. W. Ellis. Improvements in the distillation of ammoniacal liquors, and in the apparatus to be employed therein. March 29.

5058. E. Delplace and J. Delplace. Improvements in or connected with apparatus for the manufacture of sulphuric acid. April 1.

5072. L. Mond. Improvements in treating phosphatic minerals. April 1.

5205. T. Wilton. Improvements in saturators used in the manufacture of sulphate of ammonia. April 3.

5287. L. J. de Graef. Improvements relating to the manufacture of phosphates and to apparatus therefor. April 5.

5442. C. A. Burghardt. Improvements in the manufacture of nitrate of ammonium and of resulting by-products. April 10.

5469. R. H. Wilson. Improvements in the manufacture of sulphuric acid, and in apparatus therefor. April 10.

5488. W. Donald. Improvements in obtaining chlorine. April 11.

5571. A. Campbell and W. Boyd. Improvements in obtaining chlorine and in utilising calcium chloride or other by-products. April 12.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

6172. F. M. Lyte. Production of magnesia chloride and magnesia oxychlorides. April 9.

6433. W. Schlenking. *See* Class IX.

6968. J. Hargreaves, T. Robinson, and T. Hargreaves. Obtainment of sulphuretted hydrogen, sulphur and cement from black ash and alkali waste, and apparatus therefor. March 26.

7733. The Alkaline Reduction, Lim., and A. B. Cunningham. Recovery of soda from slags containing same. April 2.

8841. C. A. Burghardt. Manufacture of nitrate of ammonium and resulting by-product. April 16.

19,225. T. Wilton and G. Wilton. Manufacture of sulphate of ammonia and apparatus therefor. April 9.

1890.

1653. G. Kennedy. Saturators for sulphate of ammonia plant. March 26.

### VIII.—GLASS, POTTERY, AND EARTHENWARE.

#### APPLICATIONS.

4356. A. J. Wilkinson. Improvements in ovens for firing pottery. March 20.

4504. E. P. Lee. Improvements in kilns for burning bricks, tiles, and other clay goods. March 22.

4509. D. Rylands and B. S. Shepeote. Improvements in or applicable to machinery for the manufacture of glass bottles or other hollow glass ware. March 22.

4592. H. T. Parfitt. Improvements in kilns or ovens for firing glass, enamel ware, and for other purposes. March 24.

4792. D. Rylands and A. Hisselbee. Improvements in the manufacture of glass-lined fittings for glass-lined piping. March 27.

4800. C. Cockburn. Enamelling, rendering opaque or discernible, raised or indented designs on glass utensils, &c. March 27.

5079. S. Washington. Improvements connected with the manufacture of glass bottles and other hollow glass ware. April 1.

#### COMPLETE SPECIFICATION ACCEPTED.

1889.

8511. E. Bussy. Compounds for manufacture of pottery goods in general, and combination of ingredients for manufacture of white enamel or opaque colours as a surface or glaze for said bodies. April 2.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

## APPLICATIONS.

4741. W. A. Gibbs. Improvements in kilns for cement and the like. Complete Specification. March 26.

4995. G. A. Mansfield. Road paving by composite block process. Complete Specification. March 31.

5206. J. Chambers. A new or improved material for light roofing and such like purposes. April 3.

5349. C. O. Weber and G. F. Freeman. Improvements in the manufacture of artificial stones, slabs, or compositions for building and other purposes. April 8.

5515. P. M. Justice.—From S. W. Parker and A. Gates, New York. An improved material for building purposes. April 11.

5533. A. McLean. Improvements in asphalt paving. April 11.

5581. G. H. Skelsey. Improvements in kilns, more especially applicable to cement kilns. April 12.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

6433. W. Schlenning. Application of the residues of soda manufacture for building purposes. April 16.

7058. B. W. Weatherdon. — From T. A. Compere. Combining and treating hemp, flax, jute, &c., and employing same for roofing and building purposes. April 2.

8145. G. P. Gildea. Making cement from chalk, marls, and argillaceous limestones, and utilising poor clays for making cement, and apparatus therefor. March 26.

10,824. J. Brunton and L. Griffiths. Apparatus for mixing the materials used in manufacture of artificial stone, concrete, &c. April 16.

## X.—METALLURGY, MINING, Etc.

## APPLICATIONS.

4201. T. Teague. Improvements in smelting furnaces for treating tin ores, slags, or debris, or other metallic matters. March 18.

4386. J. von Ehrenwerth. Method for producing iron from ores. Complete Specification. March 20.

4420. J. T. Beilly and G. G. Henderson. The extraction of metals from their ores and apparatus therefor. March 21.

4560. T. Teague. Improvements in processes for refining raw tin and extracting and utilising the combined metals. March 24.

4631. R. H. Radford. Improvements in casting compound or composite metallic ingots and other metallic castings, and in machinery and appliances connected therewith. March 25.

4675. R. T. Tiefert. Improvements in the welding of metals. Complete Specification. March 25.

4690. H. J. Haddan.—From G. Conkling, United States. A process for concentrating magnetic iron ore. Complete Specification. March 25.

4752. D. Cameron.—From R. Pearce, United States. Improved process of extracting silver from copper ores, mattes, and other copper products. March 26.

4832. R. N. Oakman, jun. Improvements in furnaces for melting, heating, and welding metals. March 27.

5003. A. K. Eaton and K. A. Vanderbilt. Improvements in processes for obtaining chromium and chromium-alloys. Complete Specification. March 31.

5140. W. A. Thoms. Improvements in coating metallic and other surfaces with metals and alloys. April 2.

5199. The Electric Construction Corporation, Lim., T. Parker, and A. E. Robinson. Improvements in the treatment of copper nickel matte and the obtainment of products therefrom. April 3.

5306. W. E. Langdon. The detection of flaws or impurities in steel and iron. April 8.

5319. J. Lewthwaite. Improved method of operating upon and improving the quality of iron in the process of converting it into steel. April 8.

5407. O. Lumaghi. Improvements in apparatus for the reduction of argentiferous or other zinc ores. April 9.

5545. A. Turner, A. Baird, and M. B. Baird. Improvements in the manufacture of iron and steel. April 12.

5546. B. Mountain. Improvements in the manufacture of wire from copper, zinc, lead, brass, or other ductile metals or alloys. April 12.

5569. A. C. Kirk. Improvements in the manufacture of armour plates. April 12.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

5578. J. H. Pollok. Improvements in wet method of extracting gold. April 2.

7443. J. McCann. Blast furnace blocks and tuyères. April 16.

7666. G. Bamberg. Reducing aluminium by the action of molten metals, particularly iron, zinc, copper, and lead, on chloride of aluminium, or double chloride of aluminium and sodium. April 16.

7667. G. Bamberg. Reducing aluminium by the action of zinc vapour on chloride of aluminium or double chloride of aluminium and sodium. April 9.

8297. F. Bachschmid. Inlaying metals. March 26.

8492. J. Riley. Manufacture of steel or steel-like compounds. March 26.

8543. T. Twynam. Carburisation of steel or ingot iron. March 26.

8608. R. L. Consins. Process and apparatus for extracting precious metals from ores or quartz. March 26.

8822. E. Matheson. Manufacturing pots or crucibles of wrought iron or other malleable metals. April 2.

9358. R. E. Green. Production of alloys of aluminium. April 9.

9592. S. Alley. Apparatus for treating metallic ores or compounds with chemicals. April 9.

9632. T. Parker. Pickling and preparing iron for galvanising. April 16.

9784. W. White. Distillation of sodium and potassium, and their alloy. April 16.

11,788. D. Dennes.—Partly from T. K. R. Denver. Obtaining precious metals from their ores, and apparatus therefor. April 2.

19,334. W. L. Wise.—From B. Natuseh. Production of solutions of the chlorides of nickel and cobalt and other metals from ores or products containing them. April 9.

1890.

3782. L. Pszczolka. Obtaining uniform products from the Thomas converter, Siemens open hearth, and other basic furnaces. April 16.

## XL.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

### APPLICATIONS.

4191. T. D. Farrall. Improvements in method and mechanism for generating heat, steam, and light by electricity. Complete Specification. March 18.

4246. Sir C. S. Forbes, Bart. Improvements in primary batteries, and in apparatus connected therewith. March 18.

4383. J. Pitkin and H. C. L. Holden. Improvements in the preparation of active or storage material for use in secondary batteries or accumulators. March 20.

4384. J. Pitkin and H. C. L. Holden. Improvements in plates for secondary batteries or accumulators. March 20.

4648. P. F. Degn. Improvements in the construction of dynamo-electric machines. March 25.

4657. H. H. Lake.—From E. Thomson, United States. Improvements relating to the soldering or uniting of metals by electricity and to apparatus therefor. Complete Specification. March 25.

4943. W. Balch and S. S. Bromhead. Improvements in batteries for electrical purposes. March 29.

4949. L. Weigert. An improved electrolytic solution for galvanic batteries. Complete Specification. March 29.

5049. A. de Méritens. Means for effecting the sterilising of liquids by electricity. April 1.

5054. C. Kellner. Improvements in the process of bleaching fibrous material by aid of electricity. April 1.

5228. W. P. Thompson.—From L. Hoppe and G. Hoppe, Germany. Improvements in galvanic elements. April 3.

5400. R. Schorch. Improvements in dynamo-electric machines. April 9.

5420. C. Kellner. Improvements in the process of, and means for, the manufacture of cellulose with the aid of the electric current. April 9.

### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

13,818. V. H. Ernst. Secondary batteries. April 2.

1890.

1281. V. H. Ernst. Secondary batteries. April 2.

1878. J. B. Williams. Electric insulating compounds. April 9.

## XII.—FATS, OILS, AND SOAP MANUFACTURE.

### APPLICATIONS.

4607. W. E. Crane. An improved process of filtering oil. March 25.

4809. J. Y. Johnson.—From M. Duclou, France. Improvements in the manufacture of soap. Complete Specification. March 27.

5226. W. P. Thompson.—From R. Krause, Prussia. A new or improved lanolin compound, and process for preparing and utilising the same, specially applicable to the preparation of lubricants. Complete Specification. April 3.

5375. E. Noppel, B. Grosche, and J. Bigler. Improvements in apparatus for purifying and refining oil. Complete Specification. April 8.

### COMPLETE SPECIFICATION ACCEPTED.

1890.

540. J. L. Wade. Improved non-freezing and lubricating oil. April 2.

## XIII.—PAINTS, PIGMENTS, VARNISHES, AND RESINS.

### APPLICATIONS.

5342. E. Bruce. A varnish or enamel for leather. April 8.

5380. A. F. St. George. An improved method of and apparatus for preparing copal and other gum-resins and resins, and manufacturing varnish. April 9.

5387. D. Fulton.—From D. F. Schmid, United States. An improved manufacture of anti-corrosive compound. Complete Specification. April 9.

## XIV.—TANNING, LEATHER, GLUE, AND SIZE.

### APPLICATION.

5004. A. Foelsing. Process for clarifying and bleaching tannin extracts or tannin liquors. March 31.

### COMPLETE SPECIFICATION ACCEPTED.

1889.

8191. J. Kristen. Process and means for dyeing hides. March 26.

## XV.—AGRICULTURE AND MANURES.

### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

8782. A. Booty. Manurial composition or stimulant for vegetable produce. April 2.

19,225. T. Wilton and G. Wilton. See Class VII.

1890.

2238. C. W. Doughty. Means and proportions of materials used for the deodorisation, evaporation and decomposition of human excrement, and its transformation into a nearly inodorous fertiliser. March 26.

2450. F. Dorr. Treating cereals. April 9.

## XVI.—SUGARS, STARCHES, GUMS, Etc.

### APPLICATIONS.

4181. S. Spitzer. Improvements in the method of and apparatus for the manufacture of starch and other products. March 17.

4513. J. Duncan. Improvements in the treatment of molasses or syrup obtained from beetroot. March 22.

4754. A. J. Boulton.—From L. Bon, Cuba. Improvements in or relating to apparatus for drying sugar-cane trash. Complete Specification. March 26.

5282. G. F. Redfern.—From M. Wienrich. Improvements in washing and cleaning raw sugar. April 5.

5283. G. F. Redfern.—From M. Wienrich, United States. Improvements in the manufacture of sugar. April 5.

## XVII.—BREWING, WINES, SPIRITS, Etc.

## APPLICATIONS.

4108. J. McKinless. Improvements in maturing spirits and other liquors and apparatus connected therewith. March 17.

4550. E. Rudgard. A new or improved nourishing stout or beverage. March 24.

4746. S. Joseph and H. Citron. Fermenting process in rarefied air-space. March 26.

4876. W. E. Gedge.—From E. J. Fraser, United States. New apparatus for improving wine and other alcoholic liquors. March 28.

4982. M. Hanford. An apparatus for cooling and aerating the hot worts for the production of malt liquors. March 31.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

221. A. Perry. Preparation of rice and other grain or cereals for brewing, &c., and apparatus therefor. April 16.

9181. E. Luck. Purification of alcohol and alcoholic liquors, and apparatus therefor. April 2.

9280. S. Herschler. Apparatus for agitating and turning over malt, &c. March 26.

10,762. T. Vogel. Apparatus for treating yeast. March 26.

15,772. J. Bruce. Manufacture of yeast. April 2.

## XVIII.—CHEMISTRY OF FOODS, SANITARY CHEMISTRY, AND DISINFECTANTS.

## APPLICATIONS.

*A.—Chemistry of Foods.*

4266. P. Jacoby. Automatic apparatus of domestical and industrial use for to sterilise milk. Complete Specification. Filed 18th March. Date applied for, 16th August 1889, being date of application in France.

4554. J. Shoveller. An improved cooked food for horses, cattle, and other animals. March 24.

4701. E. Sonstadt. Improvements in stoppering or closing vessels containing solid or liquid articles of food or other organic matters in order to exclude micro-organisms, and in sterilising such matters conjointly with the stoppering. March 25.

4979. G. W. Thomas. Improvements in the preservation of eggs. March 31.

*B.—Sanitary Chemistry.*

4151. F. P. Candy. Improvements in the manufacture of materials for use in the purification and clarification, and decolouration of water and sewage and other liquids, and for other uses. March 17.

4500. R. S. Brownlow. Improvements in the construction of apparatus for removing the solid and flocculent matters from impure water. March 22.

4732. W. Birch. Improved apparatus for filtering sewage and other liquids. March 26.

4836. J. Preece. Improvements in the treatment of sewage. March 27.

5113. J. Stilling. Improvements relating to a method of destroying microscopie organisms for disinfecting and other purposes, and to substances used therefor. April 2.

*C.—Disinfectants.*

4151. F. P. Candy. See Class XVII.—B.

5438. J. Wheeler. An improved antiseptic deodorant and germicide, also particularly for preventing the ravages of moths, book worms, and other insect pests. April 10.

## COMPLETE SPECIFICATIONS ACCEPTED.

*A.—Chemistry of Foods.*

1889.

5356. H. W. Harr. Manufacturing food in a concentrated cooked form. March 26.

6280. J. F. H. Gronwald and E. H. C. Oehlmann. Method and apparatus for preparing milk so as to enable it to be preserved indefinitely. April 16.

6984. A. Vasarhelyi and J. Jambor. Preserving milk. April 2.

8882. W. P. Branson and H. W. Neild. Condensing milk or other liquids. April 9.

8963. T. Williams. Smoke-curing fish and flesh foods, and fuel compounds therefor. April 16.

9303. T. D. Constat. Preserves and condiments. April 9.

*B.—Sanitary Chemistry.*

1889.

6994. C. H. Harsey, for The Barry Patent Manure Company, Lim. Clarification or purification of sewage and other analogous foul waters. April 2.

1890.

2238. C. W. Doughty. See Class XV.

*C.—Disinfectants.*

1889.

8527. W. Black and W. L. Rennoldson. Preparation of disinfectants or anti-septics. March 26.

16,427. H. Staples, J. Staples, and W. Staples. Manufacture of a cattle cleanser, sheep dip, or disinfecting fluid. April 2.

## XIX.—PAPER, PASTEBOARD, Etc.

## APPLICATIONS.

4360. J. Robertson. Improvements in glazing paper and in machinery or apparatus therefor. March 20.

4914. W. Crosland. An improved method of graining or making a rough surface on paper. March 29.

4959. C. Kellner. Improvements in boilers for the manufacture of sulphide cellulose and in the method of heating the same. March 29.

4960. C. Kellner. Improvements in the process of and means for purifying paper pulp. March 29.

5053. C. Kellner. An improved process of softening brittle paper pulp, and apparatus therefor. April 1.

5128. C. Kellner. Process of modifying the properties of fibres used in the manufacture of paper. April 2.

5196. J. G. Walliker. Manufacturing bilge-shaped barrels from paper or other pulp. April 3.

5500. E. G. Wrigley. Improvements in the manufacture of absorbent sheets, rolls, or pads of paper, for blotting, filtering, and other similar purposes. April 11.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

5418. W. L. Wise.—From J. M. Badon. Transparent printed paper having the appearance of stained glass, and apparatus for manufacturing same. April 2.

5643. A. J. Boulton.—From Messrs. Jung and Lindig. Prevention of corrosion in vessels, tubes, valves, &c., used in the manufacture of sulphite cellulose. April 2.



XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES,  
AND EXTRACTS.

## APPLICATIONS.

4822. H. A. A. Dombrain. Improvements in apparatus for extracting fatty and other matters from substances by means of volatile solvents. March 27.

5366. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. Production of guaiacol ether. April 8.

5367. H. H. Lake.—From W. V. McKenzie and H. J. Braker, United States. Improvements in refining apparatus for camphor or similar substances. April 8.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

6494. O. J. Steinhart. Manufacture of ethyl mercaptan. March 26.

1890.

1422. A. A. Vale.—From The Chemische Fabriks-Aktiengesellschaft. Process of manufacturing cumarone. March 26.

1583. S. Radlauer. Preparation of ethyl chloral urethane. April 16.

XXI.—PHOTOGRAPHIC PROCESSES AND  
MATERIALS.

## APPLICATIONS.

10,393A, 1889. J. S. Fairfax.—From F. Crane, United States. Improvements in the films or supports used for photographic negatives or prints. Previously included in No. 10,393 of June 26, 1889.

4606. J. Leslie. Improvements in the manufacture of transparent flexible films for photographic purposes. March 25.

## XXII.—EXPLOSIVES, MATCHES, Etc.

## APPLICATIONS.

4243. W. Schückher. Manufacture of grained smokeless explosive. Complete Specification. March 18.

4798. C. A. Burghardt. Improvements in the manufacture of nitrate of ammonium and of a resulting by-product. March 27.

4808. H. J. Haddan.—From E. Passburg, Germany. Improved vacuum drying apparatus for explosive substances. March 27.

5157. A. Noble and G. Stewart. Improvements in the manufacture of pellets or plungers for percussion fuses. April 2.

5209. H. H. Lake.—From H. S. Maxim, Austria. Improvements relating to the manufacture of explosives, and to apparatus therefor. April 3.

5292. W. C. Morison. Improvements in ammunition. April 8.

5364. J. R. France. Improvements in the manufacture of nitro-cellulose and apparatus for that purpose. April 8.

5376. H. de Chardonnet. Improved processes for the nitration and the denitration of cellulose, and for regaining the acids employed, and apparatus employed in connexion therewith. April 8.

5422. K. K. Malmström. Improvements in percussion fuses. April 9.

5535. R. Low. Improvements in percussion fuses. April 11.

5552. S. V. Dardier. Improvements relating to time and percussion fuses. April 12.

## COMPLETE SPECIFICATION ACCEPTED.

1889.

1884. P. Jensen.—From H. G. Underwood. Solidified pyrotechnic powders, and method of converting same into solid form for convenience in transportation, storage, and combustion. April 2.

## XXIII.—ANALYTICAL CHEMISTRY.

## APPLICATION.

4840. E. Chameroy. Improvements in apparatus for weighing and measuring the volume and density of bodies. Complete Specification. March 27.

# THE JOURNAL OF THE Society of Chemical Industry:

## A MONTHLY RECORD

FOR ALL INTERESTED IN CHEMICAL MANUFACTURES.

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## NOTICES.

In accordance with the provisions of Rule 18 of the Bye-laws, notice is hereby given that those Members of Council whose names are placed in italics in the annexed list will retire from their respective offices at the forthcoming Annual General Meeting.

Mr. E. Rider Cook has been nominated to the offices of President and Treasurer; and Sir Lowthian Bell, Bart., F.R.S., has been nominated Vice-President under Rule 11.

Mr. David Howard, Mr. B. E. R. Newlands, and Mr. J. C. Stevenson, M.P., have been nominated Vice-Presidents under Rule 8; and Mr. R. R. Tatlock and Mr. T. W. Stuart have been nominated Ordinary Members of Council under Rule 17, in the place of Mr. B. E. R. Newlands and Mr. J. C. Stevenson, M.P., nominated Vice-Presidents.

The Foreign Secretary has been nominated for re-election.

### ANNUAL GENERAL MEETING.

Notice is hereby given that the next Annual General Meeting will be held in Nottingham on Wednesday, Thursday, and Friday, the 9th, 10th, 11th July next.

The following is a synopsis of the arrangements:—

- Wednesday, July 9th.
1. Annual Meeting, University College - 11 a.m.
  2. Midland Railway Works, Derby; train leaves Nottingham - 1.10 p.m.
  - Or, Messrs. Morley's Hosiery Factory, Messrs. Turney's Leather Works, and Messrs. Blackburn's Hosiery Machine Works - 2.30 p.m. to 4 p.m.
  3. Conversation at Nottingham Castle - 8 p.m.

- Thursday, July 10th.
1. The Burton Breweries; train leaves Nottingham - 9.50 a.m.
  2. The Butterley Iron Company; train leaves Nottingham - 1.25 p.m.
  - Or, The Birks Company's Embroidery Works, Messrs. Manlove's Engineering Works, The Nottingham Company's Hosiery Factory, and Messrs. Dobson's Lace Factory - 10 a.m. to 11.30 a.m.
  - And The Corporation Gas Works, Messrs. Doughty's Lace Factory, and Messrs. Morley's Hosiery Factory - 2.30 p.m. to 4 p.m.
  3. The Dinner at the Exchange Hall - 7.30 p.m.

- Friday, July 11th.
- Excursion to Welbeck Abbey and Clumber; train leaves Nottingham - 8.40 a.m.
- Or, Excursion to Wollaton Hall and Newstead Abbey (if possible).
- Or, Messrs. Lambert's Lace Dressing Rooms, The Midland Lace Company's Frilling Factory, and Messrs. Lindley, Wright, and Cox's Lace and Hosiery Finishing Works - 10 a.m. to 11.30 a.m.
- Tickets of membership will be issued with the Ballot Lists in time for the meeting.

Members who require extra sets or back numbers of the Journal are requested to make application to the General Secretary only, to whom also changes of address should be communicated.

Notice is hereby given, for the information of members and advertisers, that the advertisement columns of this Journal have been contracted for by Messrs. EYRE and SPOTTISWOODE, the Society's printers and publishers, to whom all communications respecting them should be addressed.

The Secretary is instructed to negotiate for the purchase of copies of the Society's Journal for January 1883, and January, February, and April 1886. Members possessing odd copies of these numbers are particularly requested to communicate at once, stating price required, with Mr. Cresswell. The stock of all other numbers is at present sufficient for the Council's requirements.

#### LIST OF MEMBERS ELECTED, 23rd MAY, 1890.

- Aldrick, E. J., 15, Charles Street, Millwall, E., clerk in chemical works.
- Anderson, Eugene E. J., Gasworks, Maryhill, by Glasgow, assistant manager.
- Anderson, Wm. F., 20, Montague Road, Dalston, N.E., engineer.
- Barrie, Daniel McL., c/o Pollok Patents Gold Extracting Co., Ltd., Johannesburg, S.A.R., chemist.
- Benyon, Jos. A., c/o And. Allan, Ionontch, Montreal, Canada.

- Brasher, F. W., 20, Mainstone Terrace, Cattedown Lane, Plymouth, chemical engineer.
- Caldwell, H. M., 3, New North Road, Huddersfield.
- Chester, W. R., Gas Offices, George Street, Nottingham, gas engineer.
- Dudley, Dr. Chas. B., 1219, 12th Avenue, Altoona, Pa., U.S.A., chemist.
- Earnshaw, Edwin, 24, Mark Lane, E.C., accountant.
- Eastlake, A. W., Ropienka Oel Gruben, Post Olszanica, Galizien, Austria, petroleum works manager.
- Fergusson, J. Henry, 14, Batavia Buildings, Hackins Hey, Liverpool, director of gas company.
- Field, Walter D., Short Hills, N.J., U.S.A., technical chemist.
- Frankenburg, Alf., 62, Waterloo Road, Manchester, rubber works manager.
- Glen, Jno., jun., Glengowan Printworks, Airdrie, N.B., calico printer.
- Hedley, Jno., jun., c/o Coignet and Co., 150, Fenchurch Street, E.C., chemical merchant.
- Hodgson, Wm., Wheelgate, Malton, Yorks, manager Malton Farmers Manure Co., Ltd.
- Holloman, Fred. R., Sugar Refinery, Rawcliffe Bridge, Selby, Yorks, chemist.
- Laycock, Dr. W. F., Guncotton Works, Stowmarket, analytical chemist.
- Losanitsch, Prof. S. M., Belgrad, Servia.
- McLellan, Duncan, Annock Bank, Helensburgh, N.B., chemist.
- Moodie, Wm. E., Croftingea Works, Alexandria, N.B., analytical chemist.
- Moulton, G. J., Henderson Street, Macclesfield, manufacturing chemist.
- Reade, Thos., Oakleigh, Compton, near Wolverhampton, manufacturing chemist.
- Sanford, P. Gerald, c/o The National Explosives Co., Ltd., Hayle, Cornwall, chemist.
- Schofield, J. E., Old Street, Newchurch, near Manchester, assistant chemist.
- Stearns, Fred. K. (F. Stearns and Co.), Detroit, Mich., U.S.A., manufacturing pharmacist.
- Sutherland, J. A., 88, Gloucester Street, Glasgow, chemical works manager.
- Wachtel, Gregory, Elabonga, Government of Viatka, Russia.
- Wells, Jno., 33, Northampton Square, Clerkenwell, E.C.
- Whiteley, Jno. W., 210, North Hill Street, Prince's Road, Liverpool, chemist.
- Williams, Herbert C., Chemical Laboratory, Ruskin Road, Tottenham, N., chemist.

#### CHANGES OF ADDRESS.

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Watson, D., Journals to 244, Great Clowes Street, Manchester.

Webster, Wm., jun., 1/o Lee Park; The Grove, Belmont Hill, Lee, S.E.

Whittaker, C. J., 1/o Alkali Works; Globe Chemical Works, Church, Acreington.

Wilson, Alf., 1/o Stafford; The Firs, Norton, near Stourbridge.

Zinkeisen, Wm., Journals to Bismarck Strasse, 31, Charlottenberg, Berlin.

## London Section.

### CHEMICAL SOCIETY'S ROOMS, BURLINGTON HOUSE.

*Chairman: David Howard.*

#### *Committee:*

<i>Sir F. A. Abel.</i>	R. Messel.
<i>H. E. Armstrong.</i>	R. E. R. Newlands.
<i>W. Lant Carpenter.</i>	B. Redwood.
W. Crowder.	T. Royle.
<i>W. J. Dibdin.</i>	John Spiller.
<i>C. Graham.</i>	Wm. Thorp.
S. Hall.	C. R. Alder Wright.
C. C. Hutchinson.	

*Hon. Local Sec. and Treasurer: Thos. Tyrer, Battersea, S.W.*

The names in Italics are those of members of Committee who retire at the end of the current Session.

The following have been elected to fill the vacancies and will take office in July next:—*Chairman: T. Tyrer; Secretary: T. W. B. Mumford; Committee: J. Dewar, A. G. Green, C. W. Heaton, D. Howard, W. S. Squire, T. E. Thorpe.*

#### SESSION 1889-90.

1890.

June 2nd:—Discussion on Messrs. Cross and Bevan's paper on the Chemistry of Hypochlorite Bleaching.

July 9th, 10th, and 11th:—Annual General Meeting in Nottingham.

*Meeting held Monday, 21st April, 1890.*

MR. DAVID HOWARD IN THE CHAIR.

### DISCUSSION ON MR. BOVERTON REDWOOD'S PAPER ON THE OIL-FIELDS OF INDIA.

(This Journal, April 1890, 359.)

THE CHAIRMAN considered that there could be no doubt as to the immense importance of the subject which Mr. Redwood had brought forward. The petroleum industry was so unmistakably one of the questions of the future, from many points of view, that it was a matter of the greatest importance to find our great Indian dependency so amply provided with this valuable product. It was to be hoped that these resources would be exploited with skill and energy, so that early and favourable results might be obtained. He was glad to see several gentlemen present who could speak with authority on the subject, and therefore would not further take up the time of the meeting himself.

MR. CHARLES MARVIN said that every one would, he was sure, concur with the remarks of the Chairman as to the importance of Mr. Redwood's paper. The Russian Government had some years ago published a bibliography of the various works dealing with petroleum. This bibliography was a bulky tome of some 300 pages, but the number of references to Indian petroleum that might be collected from the volume would not occupy more than two pages of the Journal of this Society. So far as he knew, no paper of importance dealing with the chemical aspect of Burmese petroleum had up to the present time been published in this country. All the more value, therefore, attached to Mr. Redwood's paper on the subject. The author had drawn the attention of the meeting to the four principal petroleum fields of India—those of Baduchistan, the Punjab, Assam, and Burma. He himself had always regarded the petroleum question from the political and popular point of view, rather than from the chemical. Regarded from that standpoint, it was interesting to note that Russia was developing railways on both sides of the Caspian which

## Death.

Joseph Jackson, Ashbank, Rudheath, Northwich.

would have to depend for fuel on the petroleum supply of Baku. Ten years ago the country east of the Caspian Sea was a prey to the man-stealing Turcomans. These had been replaced in power by the Russians, who had now a railway stretching for a distance of a thousand miles from the Caspian, which was consuming 10,000,000 gallons of liquid fuel a year from Baku alone. Such an immense consumption of oil from Baku for fuel purposes was due to the fact that the country traversed by the Merv railway was, like Badkhistan, almost devoid of coal and timber. That did not matter much so far as the Russians were concerned, because they had abundant supplies of liquid fuel to depend upon. The English, however, had nothing but the relatively limited supplies furnished by the Badkhistan region. As Mr. Redwood had pointed out, the Indian Government was engaged in carrying out boring operations there, but unfortunately the oil found was of such a character that it would not readily pass through the pipes, and was therefore troublesome to use as liquid fuel. He thought that the attention of the members of the Society might be advantageously directed to the problem of how to satisfactorily use this oil sludge for fuel on the Quetta railway, now running to within 70 miles of Candahar. Next in political importance came the oil-fields of Rawul Pindie, and those other northern fields to which the author had not referred, but which had been recently discovered, lying in the Waziri country, near the Khyber Pass. It was of course impossible to say what these oil-fields might yield; the present indications were very slight, but it was to be hoped that further investigation might prove that a copious supply was in existence there. Next to these two districts, which, as the meeting would see, were situated along the principal communications of India in the direction of Russia—from Quetta to Herat, and from Peshawur to Cabul, came the oil-fields of Assam. These also were of great political and commercial importance. For some time past the Government had been pushing a railway up the Brahmahpootra, and another up the Irrawaddy, towards the Assam frontier. Sooner or later there would be a connexion of the two railways in that quarter, and the petroleum fields of Makum would then be admirably placed for supplying fuel to that system of Indo-Burmese railways. After these in importance came the petroleum fields of Yenangyoung, which were exciting the greater amount of interest at the present time owing to the operations which were being carried on there by Messrs. Finlay, Fleming, and Co. He himself had long held the opinion that these fields would yield satisfactory results. As Mr. Redwood had pointed out, drilling operations had only been carried to a depth of 800 or 900 ft., whereas in the case of American borings 1,500 ft. was a common depth, and in many cases 2,000 ft. was reached. At such a depth as that, it was impossible to say what might be the yield of the Yenangyoung wells. As an illustration to support this opinion, he would remind the meeting of what had occurred at Baku. Professor Abich, one of the greatest geologists of Russia, visited that place when the drillers were commencing operations some years ago, and told them that it would be quite useless to drill below a certain point, because they would not find any oil in the lower strata existing there. The drillers disregarded his advice, and the result had been those enormous fountains of oil which were at present the wonder of the world. Mr. Redwood had given an immense amount of original information to the meeting, but, owing obviously to pressure of space, had omitted one point which seemed to him of such importance that he might perhaps be permitted to draw attention to it. It was this, that the crude petroleum of every country had its own special characteristics. The petroleum of the United States differed as much from that of Russia as the latter did from the petroleum of Burma. The oil of each country had its own special problems, requiring to be solved by the chemical expert. In the case of the United States, where the oil industry was about 30 years old, most of those problems had been overcome. The refiners there got the utmost possible yield of oil—results so exhaustive that until the Americans recently discovered certain extensive fields at Lima, Ohio, yielding petroleum of a sluggish character, not well adapted for lamp oil, they had no spare oil suitable for application as liquid fuel. The

petroleum of Russia was of an essentially different character, yielding a small proportion of illuminating oil, but large quantities of heavy lubricating oils. So far as Russia was concerned, the chemical problems connected with the oil supply had not yet been solved. She had one great chemical expert; he referred to Professor Mendeleeff, who had devoted a certain amount of scientific attention to the oil industry, but he was so much occupied with his professorial duties at St. Petersburg as to be unable to give the technical problems the requisite care at the refineries themselves. There were at present about 200 oil refineries at Baku, but the amount of chemical skill available for the industry being very limited, the refiners had so far devoted their attention to the task of merely extracting kerosene and overcoming the difficulties of transport, rather than to the solution of the problem of getting the largest possible results from their crude oil. In the case of the Burmese oils, apart from the local operations of Messrs. Finlay, Fleming, and Co., which had been of the most enterprising character, and the painstaking researches of Professor Dewar and Mr. Redwood at home, nothing had been done towards solving the problems connected with them. As Mr. Redwood had pointed out, those oils were of a peculiar character, and would require the most careful consideration of chemical experts in this country to render them of general commercial value. He felt sure that the admirable lecture to which they had listened would lead to many members of the Society taking a deeper interest in the subject. If they did, they would find that petroleum was undoubtedly one of the coming subjects of the future, and the study of it could not fail to lead to interesting and lucrative results.

MR. KIRKMAN FINLAY (of Messrs. Finlay, Fleming, and Co.) desired to thank the author and the speakers who had followed him for their kind remarks with respect to himself and his firm. It had afforded him and his partners great pleasure to add, in however small a degree, to the great store of knowledge possessed by Mr. Redwood with regard to the oil-fields of Burma. They felt strongly that it was better that the world should know the whole truth about those fields, and not be led away by irresponsible conjecture. Capital and knowledge were indispensable in conducting the enterprise there. If such were forthcoming the results would in all probability be satisfactory; if not, adventurers would be introduced and the results might be entirely different. It was their desire therefore to give the Society every information in their power with respect to these oil-fields.

MR. WATSON SMITH said that during the reading of Mr. Redwood's admirable paper it had occurred to him, as it apparently had also to the last speaker but one, that the commercial interest of this Burmese petroleum would depend to a considerable extent upon its containing considerable quantities of solid paraffins. The Caucasian petroleum was, he believed, very deficient in that class of hydrocarbons, whereas it now seemed that the crude petroleum of Burma was tolerably rich in them. It appeared to him that this Burmese petroleum was a kind of compromise, chemically speaking, between the crude American and the crude Caucasian oils. The fact called attention to by Mr. Redwood, of the close proximity of petroleum wells yielding oils containing high boiling and heavy hydrocarbons to others, the oils of which consisted of very light hydrocarbons of low boiling point, was of extreme interest, and seemed to him to present much that was suggestive. In Pennsylvania there were immense beds of anthracitic coal lying in close proximity to the petroleum springs and deposits. They now learnt that in Burma they not only had the petroleum wells, but, according to Mr. Redwood, large deposits of coal closely contiguous to them. He had awaited, during the reading of the paper, a statement of that fact, or the converse of it, and it just seemed to supply a missing link in connexion with the ideas already referred to, which had occurred to him. It would be of great interest to know whether this Burmese coal was, like that of Pennsylvania, of an anthracitic or even semi-anthracitic character. If so, the suggestion was that probably the petroleum was due to a slow kind of destructive distillation from these coal measures with subsequent condensation in contiguous

layers or strata of earth. Of course, in such case, in what particular strata the oils or crude products were deposited would naturally depend upon the physical condition of those strata as regards penetrability, &c. There was a further analogy which led one to believe in the probability as to correctness of this view, namely the fact that oils containing light products, as stated by Mr. Redwood, were found in close proximity to those containing heavy and high-boiling products. In that case also, one could imagine that subterranean heat, possibly resulting in emanations of steam or hot water, may have caused distillation of the heavier petroleum to take place, the lighter products of which, at first vaporised, would gradually and subsequently pass through and settle in the more impervious of the neighbouring strata, and there accumulate. But his theory might still hold good even if the Burmese coal only approached the anthracitic character, were, say, a slightly bituminous steam-coal, the distillation being then one of a less degree than that supposed to have occurred in Pennsylvania, though similar in character and effect. He might mention one fact which would be of interest in connexion with a slow distillation of petroleum oils. Those who had been practically engaged in the distillation of such oils might have observed, when employing ordinary glass retorts, that besides the usual distillation process there was a tendency to what he might term a "creeping over," due to capillary attraction. One could imagine it possible that this might afford an explanation of the passage of these light oils from the strata containing the heavier oils.

Mr. BOVERTON REDWOOD, in reply, said that he entirely agreed with Mr. Watson Smith as to the importance of the question whether these Burmese petroleum oils contained solid hydrocarbons in considerable quantity. He had already pointed out that a large proportion of the petroleum found in Upper Burma was of that class. He had indicated, further, that, according to information kindly given to him only that day by Dr. J. B. White—who, if he were present, would perhaps give the meeting the benefit of some further information—there was reason to believe that the petroleum now being obtained at Digboi would also be found to contain large quantities of solid hydrocarbons. With respect to the comparison made by Mr. Watson Smith between the petroleum of India and that of America, he thought it important to bear in mind that there was this fundamental difference between the two, that throughout India the petroleum occurred in the Tertiary formation, whereas in Pennsylvania, though not in California, it was found in the comparatively very old Devonian and Silurian formations. The speculation in which Mr. Smith had indulged with respect to the possible relations between petroleum and coal were of an extremely interesting character, and he hoped that Mr. Smith would pursue the inquiries he had suggested, and give the Society the results on some future occasion.

Dr. J. B. WHITE (Director of the Assam Railways and Trading Company) said, that in reply to Mr. Watson Smith's inquiry, he could state that the oil which they were now obtaining at Digboi contained a much larger proportion of solid paraffin than was formerly found in American petroleum. The specific gravity of the Assam oil indicated on Mr. Redwood's diagram was that of oil which had been exposed to the air for 10 or 12 years. He had himself taken the specific gravity of the oil from a flowing well some 17 years ago, and it was then  $\cdot 8683$ ; and he had no doubt that the oil now obtained was much thinner and would give a much larger proportion of illuminating oil than was yielded by the samples previously sent over for examination. As to the question which Mr. Smith had raised with respect to the distance of the oil deposits from the margin of the great coal-field, he might say that in Assam, generally speaking, it lay all along the coal-field for a distance of about 120 miles. So far, however, from being of the anthracitic character which Mr. Smith had suggested, the Assam coal was decidedly bituminous, closely resembling the coal of South Wales—so closely indeed, that it had driven South Wales coal out of the Calcutta market. They had no coal resembling anthracite in Assam, and he had reason to believe that there was nothing of that kind in

Burma either. He could not sit down without thanking Mr. Redwood for the very able paper which he had read, and which he hoped would have the effect of directing attention to the immensely valuable deposits of oil which undoubtedly existed.

Mr. BOVERTON REDWOOD hoped that he might be allowed to make a few further remarks with regard to the question of the specific gravity of Assam oil raised by Dr. White. He might mention that within the past 10 or 12 years he had had to examine samples of that oil submitted by the Assam Railways and Trading Company, of which Dr. White was a director. He understood that the samples were obtained from surface collections, and he had no doubt that oil obtained from a fresh-flowing well would be found to be of far lower specific gravity. So far as he knew, the only samples which had come to this country were those which he had analysed and had been kindly permitted to refer to in his paper. He hoped shortly, however, to have an opportunity of examining the oil obtained from the well at Digboi, and might perhaps be able to supplement his remarks of to-night by giving the results obtained.

#### NOTE ON THE EXPULSION OF AMMONIACAL COMPOUNDS FROM SULPHURIC ACID USED IN KJELDAHL DETERMINATIONS.

BY E. R. MORITZ.

SOME time ago, in conjunction with Professor Meldola, I communicated to this Society\* a method for the freeing of the sulphuric acid used in the Kjeldahl method from the ammoniacal compounds which are apt to be contained in it. This method consisted in digesting the acid with a small proportion of potassium nitrite. Shortly after its publication Professor Lunge criticised it adversely,† contending that the nitrous acid would not be completely expelled owing to the formation of nitroso-sulphuric acid; and, further, that the nitrous acid locked up in this way would seriously interfere with the accuracy of any Kjeldahl determinations which might be made with the sulphuric acid so treated. Lunge bases these views upon the results of experiments in which the amount of nitrous acid remaining in the acid after digestion with nitrite was estimated by potassium permanganate. He finds this acid retained by the sulphuric acid to equal approximately 95 per cent. of that originally added. It was our intention to go over the ground immediately after the publication of Lunge's criticism, but the pressure of many and more immediately important duties has prevented us from doing so until quite recently. Although Professor Meldola has left the recapitulation of the work in my hands, it has been submitted to him from time to time; and I may add that he endorses all that now follows.

I do not deny that nitroso-sulphuric acid is formed during the treatment of the sulphuric acid with the nitrite, but I argue that this is a matter of no moment so long as the compound is broken down and the nitrous acid expelled before the digestion is completed. The experiments which follow show, I think, that such is the case:—

Experiment 1.—0.3 per cent. of ammonium sulphate was added to pure sulphuric acid. The above acid was then digested with potassium nitrite; after digestion it gave no reaction (after neutralisation and dilution) with Nessler solution, and no reaction (after dilution) with starch and potassium iodide.

Experiment 2.—The same acid as above was digested with potassium nitrite (0.05 grm. per 10 cc.). After digestion the residual nitrous acid was determined by potassium permanganate. The sulphuric acid was found to contain 0.0008 grm. nitrous acid per 10 cc.

\* Journal of the Society of Chemical Industry, Vol. VII., p. 63.

† Zeits. f. angew. Chem. 1888, No. 23.

Experiment 3.—The same acid, after similar digestion, was used as in an ordinary Kjeldahl estimation, pure cane sugar being the substance operated upon. The nitrogen calculated upon 100 parts of cane sugar was 0.01 part.

Experiment 4.—The same acid, after treatment with potassium nitrite, was used as in an ordinary Kjeldahl determination, urea being the substance operated upon. The same sample of urea was also subjected to another Kjeldahl determination, pure sulphuric acid being used. The following results were obtained:—

—	Theory.	Pure Acid.	Treated Acid.
Percentages of nitrogen .....	45.67	46.38	46.38

Experiment 5.—A set of determinations were then made as described in Experiment 4, uric acid being substituted for urea. The following results were obtained:—

—	Theory.	Pure Acid.	Treated Acid.
Percentages of nitrogen .....	33.33	33.77	33.71

Experiment 6.—A sample of malt extract was now analysed by Kjeldahl's process, both with treated acid, and with some pure acid.

—	Treated Acid.	Pure Acid.
Percentage of nitrogen .....	1.09	1.11

The above experiments show, I think, that the treatment suggested is competent to free the acid from combined ammonia, while no nitrous acid remains in the sulphuric acid to interfere with the accuracy of Kjeldahl determinations subsequently made with it.

I must add, however, that I have come across cases where the whole of the nitrous acid was not expelled in the time originally suggested (two hours), owing probably to insufficiently high temperature of digestion or other accidental causes of that kind. But as it is exceedingly simple to test each batch of acid for nitrous acid before employing it as an analytical reagent, this constitutes no practical argument against the process originally suggested. For if the nitrous acid is not completely expelled in the time suggested, the digestion can be continued until the whole of the impurity is found to have been removed.

I may add that the acid used in the foregoing experiments was digested in beakers covered with clock glasses, 0.05 grm. of potassium nitrite having been added to each 10 cc. of previously ammoniated acid.

I have to thank my assistant, Mr. H. T. Pentermann, for carrying out the above experiments.

#### DISCUSSION.

The CHAIRMAN said that only those who had tried the experiment knew how difficult it was to get pure sulphuric acid; whereas in the Kjeldahl determination absolute purity was required. Therefore this paper was of great practical importance. The smallest traces of ammonia would entirely vitiate the results of the process; and it would appear that Dr. Moritz had clearly made out that Professor Lunge's objection to the use of nitrites in purifying the sulphuric acid must be based on the results of an imperfect boiling. The presence of ammonia or nitrate was alike objectionable; but in this case, if proper precautions were taken, there appeared to be no danger of either. If any members present had had experience of the process, he would be glad to hear their opinion on that point.

Dr. J. W. LEATHER had had no difficulty in obtaining pure sulphuric acid straight from the dealers, and therefore did not see the use of trying to purify impure acid. He got pure acid at a nominal price, something under 8d. a pound, it having become much cheaper recently.

Mr. BERTRAM BLOUNT wished to point out, with regard to the temperature needed to effect a complete decomposition in the Kjeldahl process, that the advantage of using a high temperature lay in the fact that the decomposition of the substance was thereby rendered more perfect. According to a private communication made to him, such a temperature could be readily obtained by adding to the sulphuric acid a suitable proportion of potassium sulphate. Acid potassium sulphate was formed, which body, having a higher boiling point than sulphuric acid, caused the organic substance present to be decomposed more completely than would otherwise be the case.

Mr. W. FOSTER would like to ask whether there was any limit to the use of Kjeldahl's method for the estimation of nitrogen. It had occurred to him recently to use the process, and he had not found it to answer as he expected. In the case of the hard cokes left after the destructive distillation of organic bodies, such substances were not attacked by strong sulphuric acid in such a way as to warrant him in considering the results good. He would be glad to know, therefore, whether any other member had used the process for such purposes. For soft substances, the method seemed to him to be well adapted; but he wanted to know how it could be applied to such bodies as were generally got by the destructive distillation of such substances as petroleum, for instance. He believed that a paper had recently been read upon the subject by a German chemist whose name he could not for the moment remember, and he thought that there must be some modification of the method for dealing with hard substances such as he had mentioned.

Mr. E. J. BEVAN said that in reply to the question put by the last speaker, he would suggest that the use of Nordhausen acid might perhaps get over the difficulty. Mr. Cross and he had devised a process for the estimation of carbon by digesting the carbon compound with sulphuric acid and chromic acid, and measuring the mixture of carbon monoxide and dioxide evolved. In the case of coals, when ordinary sulphuric acid was used no action took place, but with Nordhausen acid a considerable amount of the mixed gases was given off.

Mr. W. FOSTER explained that the cokes to which he had referred were so hard as not to be easily attackable under any circumstances, and certainly Nordhausen acid would not attack them—at least the action appeared to be very slow. The cokes were in the finest state of division. Nordhausen acid had always been used, and, in some instances, potassium permanganate had been finally added in small quantities.

Mr. BERTRAM BLOUNT suggested that in such case Mr. Foster might find that potassium sulphate would answer the purpose.

*Meeting held on Monday, May 5th, 1890.*

MR. DAVID HOWARD IN THE CHAIR.

#### THE CHEMISTRY OF THE DINSMORE PROCESS FOR THE MANUFACTURE OF COAL GAS.

BY WATSON SMITH,

*Lecturer in Chemical Technology in University College, London, &c.*

At a meeting of the Liverpool Section of this Society, held December 4, 1889, a paper was read by Mr. Isaac Carr, in which a description of the plant was given as well as the general *modus operandi* for carrying out the Dinsmore



process. For this I will refer you to this Journal, 1889, 960—962. Mr. Carr there makes good his claims for increased luminosity whilst using inferior species of coal not usually employed in gas-making, such as Arley coal, mingled with 25 per cent. of its weight of Arley slack. These claims as regards increased luminosity were supported by the experiments of Mr. Wm. Foster, M.A., of Middlesex Hospital. However, the statements as to the quality of the tar products were somewhat vague, and rumours got abroad of a rather sensational character, either as to failures of supply or failure as to quality of coal-tar, if this process were generally adopted. In order to combat these notions, and also to ascertain the condition of the coal tar produced, I undertook for Mr. Carr and the Widnes Gas Company an investigation of the tars produced at the works of this company, of which Mr. Carr is the manager. My results were communicated to a meeting of the Manchester District Institution of Gas Engineers on March 1st, and appeared in the "Journal of Gas Lighting," of March 11, 1890, 436—440. Since this I have tried certain experiments with the view of ascertaining some of the conditions under which the constituents of vaporised tars may undergo transformation, and what the nature of such transformation is, and the results I hope to lay before you this evening.

The Dinsmore process, as modified by Mr. Carr, consists in subjecting coal to a kind of double destructive distillation, the first operation being to carbonise the coal and produce gases and tar vapours; the second to further transform some of the constituents of the tar vapours into a sufficiently permanent illuminating gas, besides furnishing to the gas of the first heating a certain quantity of highly illuminating vapour, which may raise the total illuminating value by several candles. The question will be asked, and was asked at Liverpool, when Mr. Carr read his paper, "Has not this been done before?" I may reply, "It has all been tried before, but certain insuperable difficulties remained which hitherto had prevented success." It remained for Mr. Isaac Carr to engraft upon the best that had been so far done in this direction, in short, upon the Dinsmore process as it was, one or two apparently trivial improvements, to give rise to the Dinsmore process as it is. It may be instructive to go back to one of the earliest recorded attempts to subject coal to such a double destructive distillation as that referred to. This I find was in the year 1830, when James Down (Eng. Pat. 5966) patented a process of which the following is an abstracted record, his object then being to convert tar into gas, since tar was almost a valueless substance, if not a nuisance. The patentee made or generated gas out of certain portions of the "residuum" by passing crude or "nascent" gas with its vapour of tar and ammonia through a long stratum of ignited charcoal or coke, thereby evolving an additional quantity of gas free from the impurities "of the said nascent gas." A kind of box with divisions in it, which would stand the heat well, was arranged, and nearly filled with charcoal or coke, and connected with the ordinary retorts. The idea was to furnish a long stratum of ignited charcoal or coke, and the containing partitioned box was to be set like an additional retort, and to be fired with the waste heat of the other two ordinary retorts from one and the same furnace set below these. Down does not claim increased illuminating power, but merely says he thus does away with the necessity for a separate purifying apparatus. No doubt he also very much desired to consume as much as possible of his tar in the process.

In 1850, Michiel (Eng. Pat. 13,066) proposed to collect the tar and then feed it by a pipe into a hot retort or other heated vessel, in order to gasify it. He proposed to let in half a gallon of tar per given period of time, indicated for the workmen by an index; and in this manner he would work it all up. These are two of the most reasonable processes patented at the earlier dates.

I consider the earliest one, *viz.*, that of Down, an admirable conception in theory, though absolutely unrealisable in practice on the lines laid down by that inventor, for the divided box would most certainly be stopped up with thick pitch, sooty matter, and possibly coked or semi-coked pitch, and probably after one working of the retorts.

In the original Dinsmore process it was, I believe, proposed to run in tar to the retorts, and the result was then

obstinate stoppages in the ascension pipes from the ducts. So that the Dinsmore process, until modified by Isaac Carr, was by no means a success.

Carr's modifications consist, in the first place, in such a setting and arrangement of the extra heating apparatus termed the duct, that the heat applied is not excessive, and is, moreover, a graduated heat, strong as the crude gas enters, and less strong as it reaches the ascension pipe. The ascension pipe is, moreover, surrounded by a water-jacket, the effect of this cooling arrangement being to prevent the excessive charring and pitch-formation, otherwise causing the choking-up of this pipe. But just below this ascension pipe is a vertically descending pipe, leading from the duct downwards, so that any pitch formed in the duct and ascension pipe can run down towards this outlet, and, descending by it, slowly ooze out below the surface of water by which the extremity of the pipe is sealed. It is on these last-named devices by Isaac Carr that, in my opinion, the whole success of the Dinsmore process depends.

More recently, Mr. F. Pritchard, manager of the gas works of the Huyton and Roby Gas Company, near Liverpool, has made a further addition by a modification of the duct E. (Compare ducts E, E, Figs. 2 and 4.)

In connexion with and intersecting the ascension pipes is a collecting chamber D (Fig. 3), uniting together the various retorts of each bank, and fitted with valves C, to prevent back-flow of gas from the other retorts when any one of the bank is being separately drawn and charged. (See also Fig. 1.) The end of the collecting chamber is connected with the duct E, which is made of cast iron, and fitted with a cast diaphragm or midriff E, (Fig. 1), cast in solid, and running to within 8 inches of the far end of the duct. The gas thus passes along the under side of the diaphragm, up the space at the far end, along the top side, and through the water-jacketed ascension pipe G, to the hydraulic main. A temperature of from 700° to 800° F. is found to yield the best results in the iron duct, whereas in the fire-clay duct double that temperature is necessary. Since only such a low heat is used, the life of the duct will be probably of many years' duration. It is estimated that the cost of applying the system to existing systems of through-retorts is about 6*l.* per mouthpiece. It is found necessary to open the duct once a day, to remove the pitch which condenses in and runs down the water-jacketed pipe, and for this purpose a valve C, separating the duct from the collecting chamber, is closed, the gas meanwhile passing to the hydraulic main in the ordinary way, by the ascension pipe, with small dip pipes at J, leading direct from the retorts. There is a heavier seal on these pipes than on those leading from the duct at G to the hydraulic main.

Now, when I have recounted all these precautionary arrangements to prevent stoppages, even when using a duct heated only to dull redness, you may well imagine what the fate of a process in which Down's earlier apparatus was used would be.

That water-jacketed ascension pipe looks like a daring adventure, but it is really very ingenious, and scientifically correct. That which usually collects in the ordinary ascension pipes is what may be termed semi-coked pitch, a something between pitch and gas-coke, or pitch much thickened with soot. The water jacket, with water entering cool and escaping boiling, causes condensation of a little more tarry matter than would otherwise separate, and this, so to say, reduces the melting point of the whole, which consequently keeps "on the move" downwards, and so, slowly but continually, slides down into the duct, or, at all events, keeps the ascension pipe open to the gas current. The duct consequently plays, in the Dinsmore process, not only the part of a carburetting apparatus, but also a part precisely analogous to that of a dust-chamber; it is a "pitch-chamber," as well as a carburetter.

Now, with regard to the capacity as a pitch collector or chamber, the function of the duct is a mechanical one and readily comprehensible, but the case is different when we regard it as a carburetting apparatus, as an instrument for increasing the luminosity and, to some extent, the volume of the gas.

As regards actual increase of illuminating power, there can be no question after the exhaustive tests and determina-

tions of Mr. Wm. Foster, M.A., and later by Mr. Francis Jones (this Journal, 1889, 961). Mr. Francis Jones finds a larger quantity of acetylene in Dinsmore than in ordinary gas. The prolonged heat probably accounts for the reduction of olefiant gas to acetylene, as well as the formation of the latter by other modes. Now, seeing that there is some increase in volume, and certainly an increase in illuminating power, it would seem that certain hydrocarbon constituents of the gas are broken up into less luminous constituents, which swell the volume to the decrease doubtless of illuminating value, while certain constituents of the crude gas otherwise condensing to form tar, are also decomposed to yield highly illuminating compounds retaining the gaseous form, in contact with so large a volume of permanent gases. But the puzzle has been this, that hitherto gas manufacturers and chemists have been assuming that continued high temperatures in the retorts whilst decomposing and breaking down hydrocarbon substances, generally separating some carbon, of necessity must give rise to bodies of less and less illuminating value as the decomposing and disrupting action goes on, and now Mr. Carr, with the Dinsmore process, succeeds in *doing both*, namely, breaking down with increase of volume and diminution of illuminating power, and at the same time adding illuminating power, derived from the tar vapours, over and above that lost in increase of volume. Before proceeding to show how this is quite possible, let us inquire into the condition of the tar produced in the Dinsmore process, for, by subjecting crude gas, which includes the tar in the state of vapour, to continued high temperatures, it may be confidently expected that the resulting tar, which is condensed, will bear its own testimony. I have analysed two samples of tar from the Widnes gasworks, of which Mr. Carr is manager, that marked A. being actual, or, as it is termed, surplus Dinsmore tar, and that marked B. being ordinary tar, containing  $\frac{1}{3}$ rd of the Dinsmore product.

	A.	B.
	Surplus Dinsmore.	Ordinary Tar. ( $\frac{1}{3}$ rd of Dinsmore.)
	Per Cent.	Per Cent.
Water .....	1.1	7.1
Light oils .....	1.3	5.4
Creosote oils .....	16.5	17.8
	(rich in naphthalene.)	
Anthracene oil .....	12.1	8.6
Pitch .....	63.0	61.1
Specific gravity of the two tars .....	<div style="display: flex; align-items: center;"> <div style="font-size: 3em; margin-right: 10px;">}</div> <div> A .. 1.157 B .. 1.150 </div> </div>	

**The Creosote Oils.**—These contain somewhat over 50 per cent. of naphthalene. The specific gravity of the oils after the separation of naphthalene:—A. = 1.030; B. = 1.025.

**Carbolic Acid.**—This, as is usually the case with tars very rich in naphthalene, as, for example, the London tars, is chiefly found in the creosote oils. The light oils were also extracted. A. contains 2.5 per cent., B. contains 5.4 per cent. of crude carbolic acid.

**Anthracene.**—A. contains 0.79 per cent., B. 0.32 per cent. of real anthracene. The anthracene from A. was obtained without any coking of the pitch; that from B. after incipient coking.

The results were quite what I expected to find. It is clear that the phenols (see analysis of A., carbolic acid) have been drawn up in the extra-destructive distillation process. It is also probable that the increase of anthracene is at all events in part due to the reduction of higher phenols, and it must be remembered in this connexion that Noelting has discovered the phenols both of anthracene and phenanthrene in coal-tar heavy oils (this Journal, 1884, 234). The benzene in A. has been doubtless considerably

drawn upon along with others of the benzene hydrocarbons, by the gas produced, to the increase of its illuminating power.

It is clear that A. as a tar is very little behind B. in point of value. The lower specific gravity of B. than that of A. indicates what is really the case, viz., that B. is richer in naphthalene than A., and contains less crude anthracene. The light oils from A. and B. gave the following results on distillation:—

LIGHT OILS.  
(Per cent. by volume.)

	A. (Sp. gr. = 0.930.)	B. (Sp. gr. = 0.923.)
Below 100° .....	4	4
From 100°—120° .....	11	11
„ 120°—130° .....	16	14
„ 130°—150° .....	20	27
„ 150°—175° .....	18	150°—180° .. 24
„ 175°—195° .....	14	180°—185° .. 7

Residue = solidified naphthalene.

The best criterion for the tar distiller as to how far the distillation must be driven to obtain such results as those given, is to be found in the quality of the pitch left behind as residue.

The pitch from A. and B. was tested, and found to be good hard pitch.

PITCH.

	A.	B.
	Tensile Strength = 150 lb. per sq. in.	Tensile Strength = 132½ lb. per sq. in.
Action of heat {	Softens at 67½° C.	68° C.
	Twists „ 71½° C.	72° C.
	Melts „ 94° C.	95° C.

I may mention that other samples of Dinsmore and ordinary tar from the Widnes works were also tested by Mr. W. R. Chester, manager of the gasworks of the Corporation of Nottingham, and our results were mutually confirmatory.

Some interesting results were obtained with a sample of tar taken directly from the Dinsmore duet at a temperature of about 350° F. The specimen contained no first and scarcely any of the second light oils. These must have been carried forward, and only condensed further on. What was actually condensed was a product containing a very large quantity of anthracene, with scarcely any naphthalene. Now, naphthalene is generally looked for in such high-boiling tars, but doubtless this hydrocarbon had been nearly all carried forward by the hot gas and vapours, the less volatile crude anthracene remaining behind. The remarkable thing is that amongst the anthracene oils was a large proportion of phenols with carbolic acid, showing that crude phenol and cresol will condense at a temperature at which naphthalene is carried forward in the state of vapour, if in addition to heat there be a current of gas. In this tar there were 2.8 per cent. of water; light oils, nil; heavy oils, only 1.7 per cent.; creosote oil, 14.6 per cent. (sp. gr. 1.04); anthracene oil, 25.9 per cent.; pitch, 55 per cent. The amount of crude carbolic acid was extraordinary, viz., 5.1 per cent., and it was contained chiefly, as stated, in the anthracene oil.

With regard to the tar obtained when a duet is used like that devised by Mr. Pritchard, in which the gas has to pass over a still greater heated surface, I have tested a small sample of such tar and found it of good average quality.

# THE DINSMORE PROCESS.

Fig. 1.

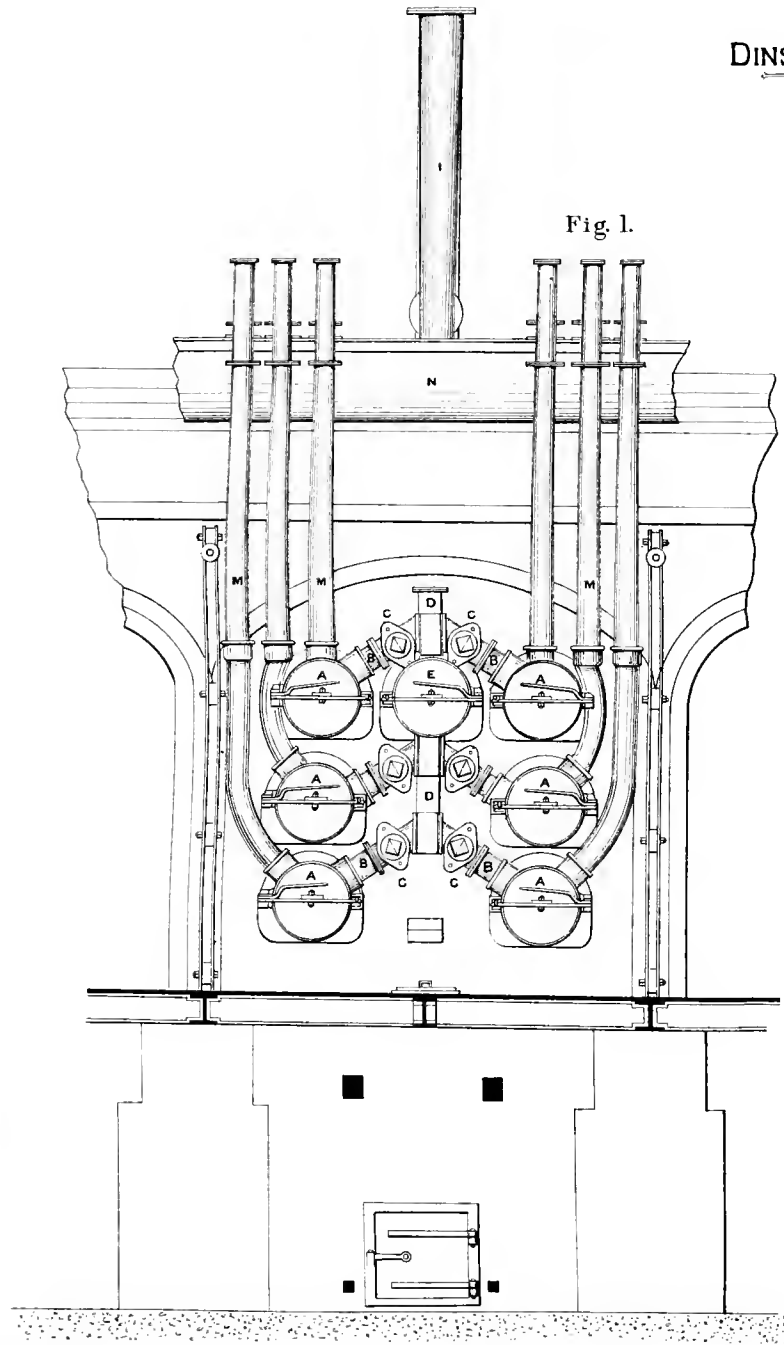
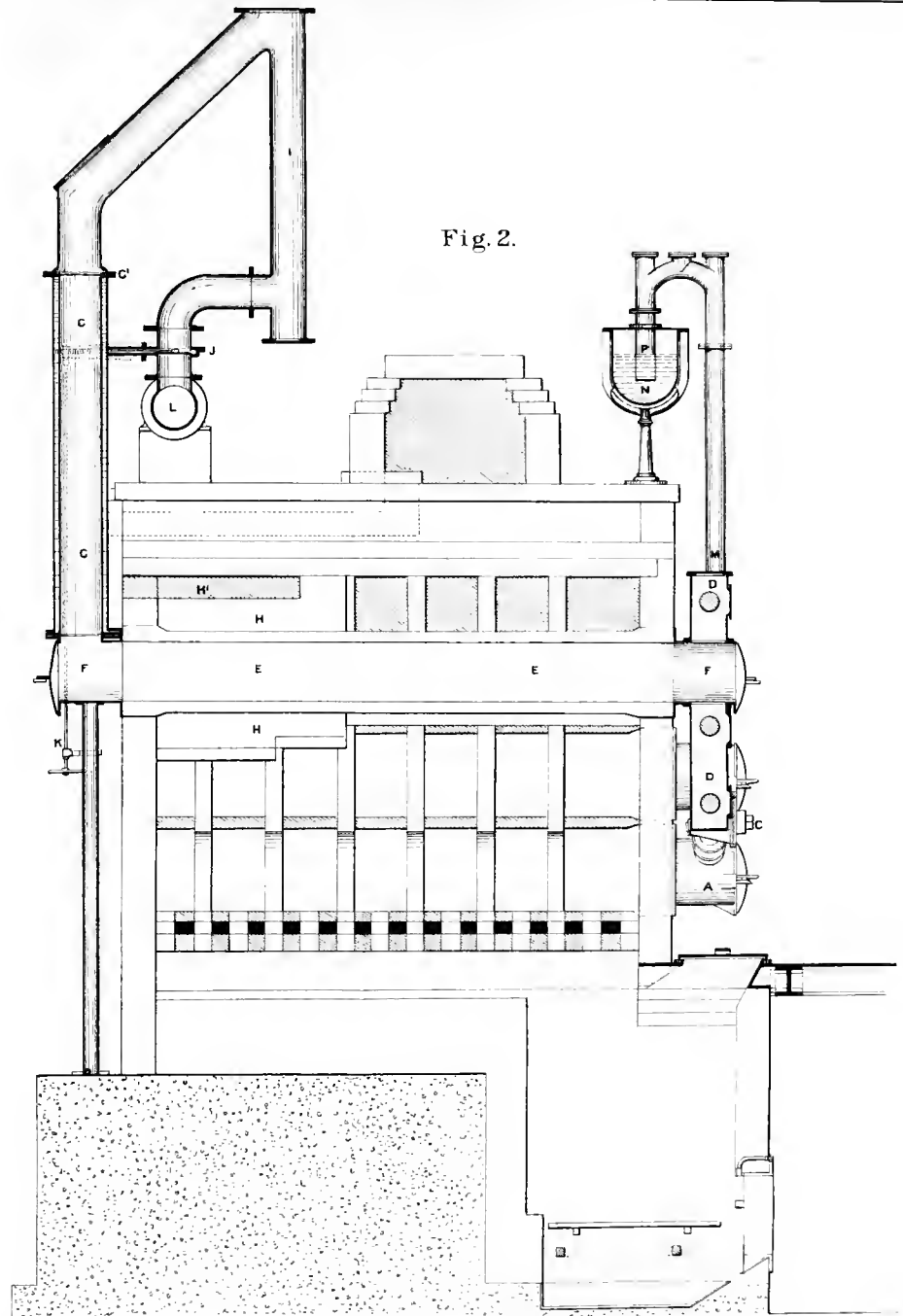


Fig. 2.



THE DINSMORE PROCESS.

Fig. 3.

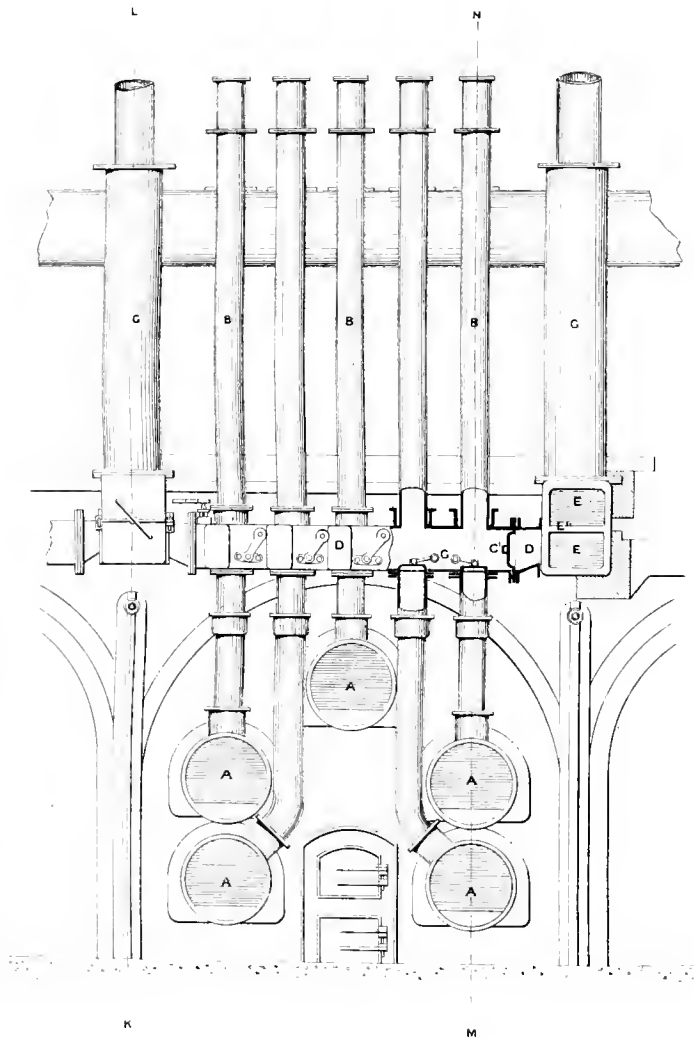
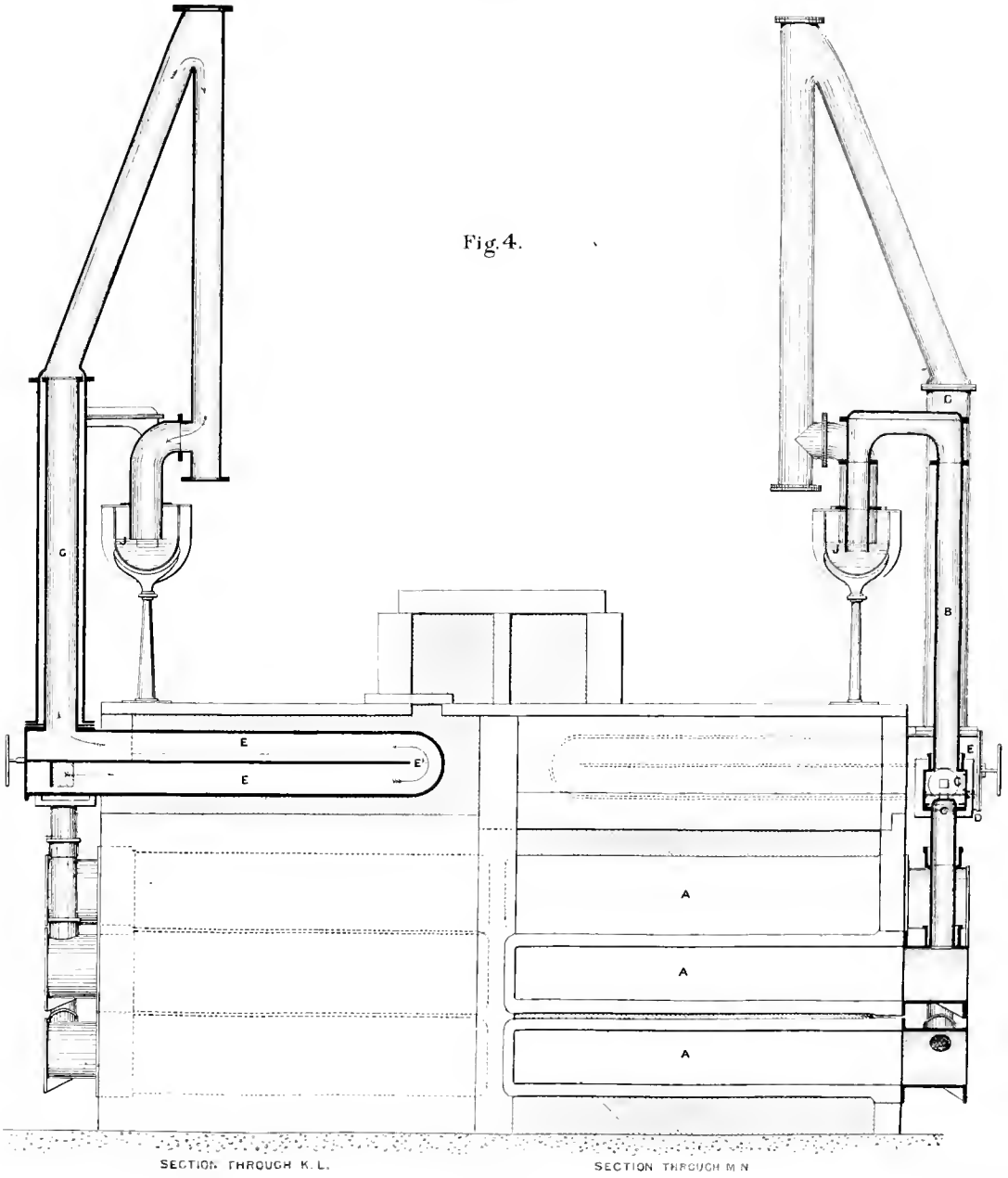
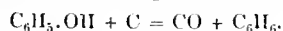


Fig. 4.



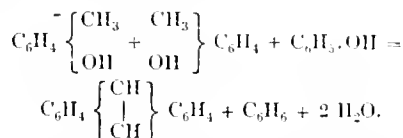
Having given a fair notion of the character of the tar produced by the Dinsmore process, I must only add that the coal used by Mr. Carr was not the usual gas coal, or Lancashire cannel, but a common steam coal from the St. Helen's district, ordinarily yielding about 9,000 cubic feet of 15-candle gas. By the use of this coal in the Dinsmore process Mr. Carr produces 9,800 cubic feet of from 20 to 21 candle gas. It may not be known to many present, and so I will just mention the fact, that it is now pretty accurately ascertained that the celebrated Lancashire cannel coals, such, *e.g.*, as Wigan cannel, are getting near their end, and consequently begin to rise in price. This is a very cogent reason alone for the energetic way in which the Dinsmore process has been perfected and pushed forward.

Proceeding now to the chemical changes taking place in the Dinsmore duct, let us ask, "What are the results of a prolonged red heat on the vaporised products of the gas retort?" The direction, it may be replied, is one and the same in all cases, with but few exception, *viz.*, to the decomposition or resolution of the more complex hydrocarbons or carbon compounds into less complex and more simple forms, the extreme limits being carbon (soot, coke), carbonic oxide and hydrogen. This final ideal stage is never fully reached, of course, and yet to some extent and in some degree it is, for the products named are always present, and increase with increased heats; and there are some carbon compounds more easy of such ultimate resolution than others. The paraffins may suffer resolution and yield olefines and hydrogen, and these again may be further reduced to acetylene and hydrogen. Mr. Francis Jones found in the Dinsmore gas about 0.14 per cent. of acetylene, and he adds that the acetylene in the gas of the Manchester Corporation does not exceed 0.05 per cent. Acetylene being an illuminating constituent, this is a point favourable to the Dinsmore gas; but whilst no doubt its greater quantity is due to the prolonged heat in the duct, yet it may also be due in part to the class of coal carbonised. But let us once more regard the acetylene which Francis Jones finds present to an extent nearly three times that usual in ordinary gas. Berthelot has shown that when heated in a glass tube to dull redness, it is converted into benzene,  $3 \text{C}_2\text{H}_2 = \text{C}_6\text{H}_6$ . It also plays an important part in the formation of toluene, the xylenes, and probably of anthracene. Now when we regard the tar analyses we observe the diminution of the phenols in the Dinsmore tar, besides that of the first light oils containing the benzenes. Let us now ask, what becomes of the phenols which have vanished from the Dinsmore tar? Experiment has proved that phenols may be reduced, either alone, to form the corresponding hydrocarbons, or in conjunction with certain non-saturated hydrocarbons to form aromatic hydrocarbons of more complex types. The Dinsmore duct we may consider as a red-hot tube lined with carbon in a finely divided, or at all events porous form, and the crude gas itself, as it comes from the retorts, as filled with a vapour of finely divided carbon or sooty particles. The duct thus serves to prolong the contact of the red-hot carbon with the constituents of the gas. I have proved by actual experiment that when pure carbonic acid vapours are passed through a red hot glass tube containing iron turnings, or carbon, the result is the same, *viz.*, reduction to benzene is effected, according to the equation—



In fact this is the most convenient way, I know, for preparing pure benzene free from thiophen for lecture experiments, and I have long used it. Cresol in like manner yields toluene. I have also tried the experiment of passing a mixture of carbonic acid vapour and carbonic oxide through a red-hot tube with pumice stone, and I found scarcely any action to take place, whilst with a mixture of hydrogen gas and carbonic acid or cresol, reduction takes place at a high temperature with formation as with carbon, of benzene or toluene, as the case might be. Tervet has shown that the last gaseous products yielded by the almost exhausted fuel in a gas retort, consist of nearly pure hydrogen. He patented a process for increasing the yield of ammonia from the coal being carbonised in the gas

retorts. He proposed to turn the final products of a finishing retort consisting mainly of hydrogen into a retort which had been charged and working for about an hour, and thus by supplying hydrogen at the right period, he claimed an increased production of ammonia, and, indeed, he proved by experiment that this increase is an undoubted reality (this Journal, 1883, 445—449). If, then, the retorts in the Dinsmore process are worked consecutively through the duct, we should have along with the usual average gas from one retort, the finishing gas of the other retort (if two were working through one duct). The hydrogen at the high temperature would then certainly reduce phenols to great advantage. I am informed that the retorts are thus worked consecutively. Again, crude phenol (phenol and cresol) may contribute to form anthracene. Köhler has proved this (this Journal, 1885, 335). Probably in this reaction two molecules of cresol and one of phenol interact:—



Finally, it will be observed that an essential difference between the ordinary gas process and the Dinsmore process is that in the ordinary case we have each retort acting independently and discharging independently its issues into the hydraulic main, where, of course, no further chemical change in the gases can be expected to occur; but in the Dinsmore process we have the final issues (hydrogen) of one retort mingling in a red-hot pipe with the earlier issues of a neighbouring retort, and we have now seen that under the circumstances interaction may take place. The latter slightly- or non-luminous constituents of the finishing retort thus enrich themselves and become carburetted.

#### DISCUSSION.

The CHAIRMAN said that the chemistry of gas manufacture was apparently an inexhaustible field of interest. Whether the total destruction of benzol and toluol was desirable was, however, a serious question, upon which the Dye Industry would have something to say. Papers of the nature of that just read by Mr. Watson Smith were specially valuable as indicating to gas engineers the conditions under which they can produce such constituents as might be required at the moment. At one time the demand might be for hydrocarbons for use in various industries, at another the chief object would be to produce as much gas and as little of by-products as possible. Mr. Foster being present, would, he hoped, give the meeting the benefit of his wide knowledge and experience of this subject.

Mr. W. FOSTER said that he was glad of an opportunity of explaining certain proceedings on his part which, as Mr. Watson Smith had observed, had given rise to some comment. In November 1889 he visited Widnes to report on the character of the gas then being made by Mr. Carr. The table before the meeting\* was prepared by him for the purposes of the paper read by Mr. Carr at Manchester. Nothing was said on that table as to the illuminating power of the gas under notice, but the point was fully gone into at the time, and was embodied in the original report. He was at Widnes for four days, and spent the whole of the time with Mr. Carr at his works. He wished to explain, however, that he was not able to go fully into the question of the tar at the time, though he took samples of tar both from the Dinsmore duct and the works main, each of which was examined and the results incorporated in his report, a further description of his results and his own opinions being expressed in a discussion at Manchester on the reading of Mr. Carr's paper. On the termination of that discussion his obligation to the Dinsmore Company was discharged. A point about the Dinsmore process as practised at Widnes which struck him as specially admirable was that one retort of a bed of six was drawn and charged hourly. Each retort

\* See this Journal, Dec. 1889, 961, 962.

was heated for six hours, and during all that time gases were coming off in the different phases of destructive distillation and commingling in the central chamber or duct. When the workmen wanted to discharge a retort, all that was necessary was to turn a special tap, thus shutting off that retort from the duct and causing the gas, when the lid was again closed, to pass into the ordinary hydraulic main. The gas was never subjected to the action of water in the hydraulic main except during the few minutes occupied in taking out the coke and putting in the coal. That was a most important feature of the process, and required emphasising. (The speaker here entered into a description of the manipulation of the apparatus illustrated by the diagrams.) The gas made by the Dinsmore process at Widnes was decidedly richer in heavy hydrocarbons than gas made in the ordinary way from such coal, and from a chemical point of view was altogether better as an illuminating agent. The particular coal employed during his experiments was Abram Arley Mine slack, a cheap coal which would not ordinarily produce the quantity of gas which he had found it to produce by this process, the illuminating power of the gas being at the same time several candles higher. When his results became known, some gas engineers shrugged their shoulders. They held it impossible to heat coal gas without impoverishing it, because so many people had tried to do so before and failed, and in this respect, speaking broadly, such a view was correct. But the Dinsmore process in operation at Widnes consisted in heating for a limited period a stream of crude coal gas laden with tar particles and heavy hydrocarbon vapours, the latter of which were capable of being further decomposed by the action of heat. He had had this question before him in another form for years, and the thing was perfectly intelligible. In examining very rich coals (say from Kentucky), which were so rich as to be incapable of being distilled for gas by the ordinary apparatus, either larger surfaces or increased temperatures were necessary for fully developing the gas-producing qualities which they possessed. His experiments at Widnes had fully borne out Mr. Carr's assertions. That he did get more gas was verified by the measurements. The plant and everything connected with the process was thrown open to those who cared to see it, and there were absolutely no secrets about the matter. Moreover, the illuminating values as stated by him were fully borne out by the complete chemical analyses of the samples. He attached considerable importance to these figures because they had been arrived at after very long experience. (The speaker here referred in detail to the table of analyses, and showed that the results brought forward by Mr. Watson Smith were in accord with his own original report.) But there was one point about the experiments that was questioned, and very properly so, and that was the extreme lowness of the specific gravity. Nobody had yet accepted those figures as exact without reserve, and they were still under surveillance. It was, however, not his business to write history, but to try to make it; and he had therefore left the gas engineers to dispute over this matter among themselves. He had been taken to task because his observations on specific gravity and illuminating power were not in accordance with statements in the text-books. He would leave the engineers to settle that point, merely observing that the only way in which they could properly do so was to bring forward actual experiments proving the opposite. Having dealt with many sorts of coal, and finding that by means of high temperatures he could split up the tars, Mr. Carr's results were quite intelligible to his mind. Given a coal, the amount of tar would vary, and in such cases the amount of gas would vary also. Where there was more gas there would be less tar, because the amount of coke was found to be a pretty constant quantity. That had been shown over and over again. In the case of the carbohydrates, matters were not so simple, because the amount of coke was much smaller; but with ordinary coal the coke was fairly constant and the changes were rung on the tar. Supposing a hydrocarbon like ethane were decomposed by heat, thus  $\text{C}_2\text{H}_6 = \text{C}_2\text{H}_4 + \text{H}_2$ . The two volumes of the ethane furnished two volumes of ethylene and two volumes of free hydrogen, the total lighting efficiency of the new bodies being greater than that of the old. Further,

it was known that marsh gas was of itself non-luminous; yet if the amount of air supplied to it during combustion were modified it became luminous. The luminosity could only be explained on the assumption that free carbon or hydrocarbon vapours were produced. It was difficult to prove the production of these hydrocarbons, but nevertheless he believed it was a fact. So it was with other bodies. Therefore if marsh gas, which was a saturated hydrocarbon, could thus be made to yield a luminous flame, it might be inferred that it was due to the temporary production of other hydrocarbons presumably unsaturated. He would point to an equation illustrating this assumption. If they took marsh gas,  $\text{CH}_4$ , they might, hypothetically, suppose acetylene,  $\text{C}_2\text{H}_2$ , to be produced. Thus,  $2\text{CH}_4 = \text{C}_2\text{H}_2 + 3\text{H}_2$ . Marsh gas, as he had said, was non-luminous, but was rendered luminous by the least trace of acetylene. So that if by the Dinsmore or any similar process acetylene were produced, each four volumes of marsh gas would give two volumes of acetylene and six volumes of free hydrogen, not merely an increase in volume, but the communication of illuminating power to that which was previously non-luminous. Dr. Percy Frankland had determined the lighting values of the paraffin  $\text{C}_{10}\text{H}_{22}$ , and also of the olefine  $\text{C}_2\text{H}_4$ . The speaker thought it desirable to coin an expression for such lighting values, and therefore preferred the expression "specific illuminating power" of a given substance. The specific illuminating power of ethylene ( $\text{C}_2\text{H}_4$ ) was much greater than that of ethane,  $\text{C}_2\text{H}_6$ , and he had no hesitation in advancing the opinion that the specific illuminating power of acetylene,  $\text{C}_2\text{H}_2$ , would be found on determination to be much greater than that of ethylene,  $\text{C}_2\text{H}_4$ , because it was a less saturated substance. For these and analogous reasons he considered that the Dinsmore process, if they could only heat up to the necessary point and supply the duct with gases of these various characters, possessed all the elements of success. The difficulty was to regulate the temperature so as to resolve these saturated or analogous bodies into non-saturated bodies known to be luminous. As Mr. Watson Smith had pointed out at Manchester, there were several ways of representing the matter. He was still convinced, however, that the results of the process were, as he had stated in his original report, due to the breaking up of the hydrocarbons. He did not like to go further than that at the time. There were contracts at the back of his observations; and gentlemen engaged in trade would know what that meant. Therefore having regard to the tar, he had confined himself to stating that there was less benzene and the light naphthas, less light oils, more of the heavy oils up to anthracene, and more pitch than in ordinary tar, quantities by volume being given. Probably by mere accident the sample of Dinsmore tar which he had examined was more than usually viscous, it being difficult to get the water from the tar by allowing it to stand. With reference to the low specific gravity of the Dinsmore gas, he thought that his hearers, as chemists, would concede that if one could make acetylene or any similar hydrocarbon from marsh gas, thereby giving it a higher illuminating power, that was a possible explanation of the effect of the Dinsmore process. The carbon and hydrogen densities of the heavy hydrocarbons were very suggestive, and pointed clearly to the conclusion that they were bodies of the olefine rather than of the paraffin type. Mr. Watson Smith had referred to the absence of carbonic acid gas. There was some misunderstanding, as this body was always present in the Dinsmore gas. Column I. of the table gave the specific gravity of a specimen of Dinsmore gas after passing through a lime purifier. The same column gave the amount of  $\text{CO}_2$  and  $\text{H}_2\text{S}$  before the action of the lime, and the following figures of the same column gave the composition of the sample after the complete removal of  $\text{H}_2\text{S}$  and  $\text{CO}_2$ . The figures of the second column gave the composition of another specimen of Dinsmore gas after its passage through a lime purifier, while the third column gave the composition of a specimen of town gas (a mixture of Dinsmore and ordinary gas) after its passage through an oxide of iron purifier only. It would be seen that the crude gas contained 2.75 volumes per cent. of  $\text{H}_2\text{S}$  and  $\text{CO}_2$ . Some people who had argued this question had inquired: "Where are the sperm candles you claim to get from these few gallons of tar?" But the

question could not be so discussed, the expression "illuminating power equal to so many candles" being merely a convenient form of representing lighting values. The carbonic oxide was by no means exceptional. Its correct determination was troublesome. His friend Professor Thomson of King's had privately expressed to him his own experience of such difficulty. It was removed by means of euprous chloride. A mixture of gases containing carbonic oxide would even after three successive treatments contain a trace of carbonic oxide sufficient to vitiate the determination of the free hydrogen when based on the amount of oxygen needed.

Mr. F. SCUDDER said that he would like to be informed as to the illuminating power of the town gas as compared with the Dinsmore gas.

Mr. W. FOSTER replied that he would have given the figures, but feared to be wearisome. He would now give them from his report.

Column I.	
	Candles.
Dinsmore gas after purification with lime...	21.8
Column II.	
	Candles.
Dinsmore gas after purification with lime...	22.3
Column III.	
Town gas after purification with oxide of iron	18.6

He might add that he strongly believed in the process. One of its chief advantages would be that it would enable the cheaper classes of coal to play a very different part from what they did a few years ago. There was no merit in making 22-candle gas. At Widnes the Corporation was under contract to supply gas of much lower illuminating power. But the Dinsmore process enabled Mr. Carr to use a coal which he could not possibly employ in the ordinary method of distillation. He certainly robbed his tar to the extent of about 33 per cent. in order to attain this result; but if he could use a coal not usually considered as a gas coal, and obtainable at a lower price, and still make his gas as good or better, that was a very important point. Cannel coal was already becoming scarce in this country, and the freight from America was so great as to prohibit the use of American cannel in this country. Everything therefore tended to force the gas engineer to fall back on inferior coal. But, as the Chairman had said, it would always be a question of gas or tar; and that question must of course be settled by the relative market values of products.

Mr. F. SCUDDER remarked that the increase of illuminating power in the Dinsmore gas was about 17 per cent. But it was to be noted that the ordinary town gas contained 2 per cent. of carbonic acid. If that carbonic acid were taken out the illuminating power of the gas would be increased by 10 per cent. There was also another very ugly figure in the analysis of the town gas, namely, free nitrogen 1.1, whereas the Dinsmore gas showed only 0.5, which entailed little loss of illuminating power. If these two faults were corrected (and it was to be remarked that the quantity of nitrogen was extraordinary) it would be found that the illuminating power of the two gases was practically the same. It would be interesting to know what kind of hydrocarbons the 6.6 was composed of; for instance, what was the proportion of benzene vapour? He would also be glad to know how long the gas was kept before it was tested. He could quite understand gas being prepared with tar at 230° F., in which case the illuminating power would be increased; but if the gas were kept for two days the illuminating power would go back, and the hydrocarbons would condense from the gas. No experiments had been brought forward to show the permanent character of the Dinsmore gas.

Dr. C. R. ALDER WRIGHT had not had the advantage of hearing the earlier part of the paper, and was therefore running the risk of putting questions which need not have been put if he had been present from the first. He would like to know precisely what the terms "carbon density" and "hydrogen density" referred to in the table displayed

giving the relative compositions of "Dinsmore gas" and "town gas." He thought he understood, but was not quite sure. He assumed that they referred to the average state of condensation in the hydrocarbons other than marsh gas; so that, taking the first specimen, 5.3 per cent. of hydrocarbons containing carbon at the density of a little less than .3 would contain about the same amount of carbon as would be present in about 15 per cent. of CO. In other words, he understood "carbon density" to mean the average number of carbon atoms per molecule of the hydrocarbons. If that were the correct interpretation, he could not see that the figures were much in excess of those ordinarily obtained with gas coal, or gas coal with cannel. He remembered years ago making some analyses of Rochdale gas by Bunsen's method, where the amount of hydrocarbons absorbable by fuming sulphuric acid ranged between five and seven, and represented an amount of carbon condensation equivalent to from a little under to rather over three atoms of carbon in the molecule. In other words, the carbon was in a more condensed state on the average than in ethylene approximately in the same state of condensation as in propylene. Analogous results were obtained more than 30 years ago by Bunsen with Manchester gas, and described in his "Gasometry." He would like to know how the figures were obtained for the percentages of hydrocarbons, whether by fuming acid or bromine; and if there were any certainty that other hydrocarbons of the paraffin class (*e.g.*, dimethyl or ethane) were wholly absent; for if not, the ordinary method of determining hydrogen marsh gas and carbonic oxide would be wholly fallacious. Assuming that the town gas was made from the same coal as the Dinsmore gas—and even apart from that assumption—it was difficult to understand how one could materially diminish the amount of marsh gas and correspondingly increase the amount of hydrogen, and yet keep the density practically constant. It seemed at first sight as if the figures quoted were incompatible in that respect. Then as regarded the hydrogen density in the hydrocarbons, he would like to know how the figures had been determined. As they stood they indicated that the hydrocarbons belonged to the ethylene series; so that benzene and less hydrogenised hydrocarbons would seem to be almost wholly absent. He did not know if that was the result of the breaking up taking place in the duct; but if it were, it was then not difficult to understand how the total volume of gas produced was increased in the process. He did not gather that the actual yield of tar constituents had been calculated out per ton of coal. The percentages of the various constituents contained therein were intelligible enough as they stood; but if one reckoned them per ton of coal, it might be that in one case one would get only one-third the proportion obtained in another case, and so on; it was obvious that the percentage compositions alone did not tell the whole story. He was asking these questions simply for his own satisfaction, and trusted that they were not out of place.

THE CHAIRMAN felt that the course of the discussion had placed him in a condition of some difficulty, inasmuch as although Mr. Watson Smith was the author of the paper before the meeting, the criticisms and questions which it had called forth were addressed chiefly to Mr. Foster. Under the circumstances, therefore, he would call upon that gentleman to reply.

Mr. W. FOSTER replied that in the first place he would like to call attention to the town gas given in the right hand column. If the carbonic anhydride were removed the density would work out to .382. It was obviously paradoxical, however, to have a gas of that high value and very low specific gravity. Mr. Scudder had inquired about the heavy hydrocarbons, and the answer to him would apply also to Dr. Wright. The volume of those hydrocarbons was determined by absorption with Nordhausen sulphuric acid. The amount of carbonic anhydride produced by these bodies, and the amount of oxygen needed for their combustion, furnished the experimental data. The figures for the carbon and hydrogen densities gave the *average molecular* composition of the substances present. There was no attempt made to differentiate the substances classed under the head "heavy hydrocarbons," that being quite impossible



at the time. But the specific gravity of the sample was taken at the time by means of a special globe kindly made for him by Mr. Lennox, of the Royal Institution, to whom he was much indebted in many ways. The gas was always kept for a day in a holder exposed to the air laterally in the month of November. No attempt whatever had been made to nurse the gas. He had gone down to Widnes under somewhat exceptional circumstances, but though aware of some hostility to the process he had tried to keep a perfectly judicial mind, and had recorded everything as he had found it. If there had been any attempt at padding, the amount of carbonic anhydride in the town gas remarked upon by Mr. Seadder would have been different. He scarcely ever found a sample of coal-gas containing less than 1 per cent. of nitrogen. One did not like to call in question the work of a confrere; but a distinguished chemist, who was probably a member of that Society, had quite recently recorded as much as 10 per cent. He thought he knew how he had found that figure, and why. Certainly 1 per cent. could not be considered high, when it was remembered that coal-gas was passed through purifiers to which the air had access during the transfer of material for purification. Air was always introduced in small quantity during the charging of the retorts and through the action of the gas exhauster.

MR. WATSON SMITH, in answer to Dr. Alder Wright, desired to say that the yield of tar per ton of coal carbonised in the ordinary retorts is about 11 galls. The same coal carbonised by the Dinsmore process would yield about 7 galls. per ton. It must, however, be borne in mind that the coal from which these quantities of tar are obtained is of low class quantity, a good Arley mine coal yielding from 12 to 13 galls. of tar per ton. He also wished to say one word with regard to the absence of carbon dioxide in Dinsmore gas, which was the only point upon which he had seemed to be at variance with Mr. Foster. He had founded his views on that head, and it now appeared erroneously, at the Manchester meeting, from statements he had heard there and had taken to be originated by Mr. Foster. Now that Mr. Foster assured him that the result he had mentioned was not really in accordance with his figures, he, of course, withdrew the application of his theory accounting for it. At the same time he believed that the reaction would be in that direction, for if carbon dioxide passed into a duct containing red hot carbon it must be reduced to some extent.

MR. F. SCUDDER remarked that in that case the carbonic oxide would increase; whereas, according to the analyses, it was less in the Dinsmore gas than in the town gas.

MR. W. FOSTER replied that there was no evidence before them yet as to what the town gas was before the Dinsmore gas was mixed with it, and he thought that the Dinsmore Company were a little at fault in that matter. They would like to know exactly what an ordinary coal would do by the ordinary method, and then ascertain how the same coal behaved under the Dinsmore method. He was informed that Mr. Duxbury, of Darwen, had instituted experiments of the character he had suggested.

### BÖTTINGER'S NEW REACTION FOR TANNIC AND GALLIC ACIDS.

BY WATSON SMITH.

MR. WATSON SMITH illustrated this reaction by heating separately small quantities of the above acids with double their weight of phenylhydrazine for a few minutes to a temperature slightly over 100° C., adding a little water, and boiling for a few seconds, and then allowing a drop or two from either vessel to fall into large beakers containing water which had been made alkaline with caustic soda.

In the case of the tannin compound, a beautiful blue colouration was developed, gradually subsiding into yellow, whilst in that of the gallic acid hydrazide, an orange or golden yellow colouration was produced.

The details of this method and its *rationale* will be found on page 555.

### DISCUSSION.

THE CHAIRMAN said that the chemistry of these allied but totally different acids was always a matter of interest, but in the present case the meeting had to consider an entirely novel reaction. He hoped that if there were any members present who had studied these bodies and their reactions, they would give the meeting the benefit of their views as to the practical applicability of such a process as Mr. Watson Smith had described.

MR. CROWDER inquired whether the reaction was intended as a test for quantitative purposes, or whether it was simply a qualitative test?

MR. WATSON SMITH replied that at present the reaction was simply qualitative, but what it might come to ultimately he did not know.

MR. CROWDER asked if it was to be regarded as a qualitative reaction which might be used for a quantitative process?

MR. WATSON SMITH replied in the affirmative.

MR. C. F. CROSS inquired whether the reaction had been studied so far as to determine the constitution of the bodies?

MR. WATSON SMITH replied that it had been studied by Herr Böttinger, and that a detailed account of the research, so far as it had gone, was given in the article from which he had quoted. There appeared to be a certain amount of mystery in connexion with the substances which had as yet been only partially isolated.

### SOME CONSIDERATIONS ON THE CHEMISTRY OF HYPOCHLORITE BLEACHING.

BY C. F. CROSS AND E. J. BEVAN.

WITH the assistance of our friend and pupil, Mr. F. E. Nelson, we are investigating the reactions which take place in the processes of bleaching vegetable fibres, confining ourselves for the present to those in which the chloroxygen compounds are used.

In the subjoined brief account of our results we shall endeavour to elucidate the following points:—

(a.) Bleaching by means of the hypochlorites is attended with chlorination of the fibre constituents, more or less, according to the nature of the basic constituent of the bleaching solution and the condition of the fibre substance.

(b.) The chlorination is conspicuously less in the case of magnesium hypochlorite—prepared by double decomposition from bleaching powder—and still less in the case of solutions prepared by electrolysis of  $MgCl_2$  Aq.

(c.) The evidences of chlorination are: (1), a portion of the chlorine of the bleaching solution does not revert to the condition of hydracid, *i.e.*, chloride, and in consequence, a portion of the base with which it was combined as hypochlorite, is otherwise combined than as chloride in the exhausted mixture (solution and fibre substance); (2), "organic" chlorides are present in the washed pulp.

(d.) The factor of chlorination differentiates bleaching by means of hypochlorites from those processes in which the only possible factors are oxidation and hydrolysis, *e.g.*, bleaching with permanganates and hydrogen peroxide.

(e.) The cause of chlorination lies in the presence of ketonic oxygen in the fibre constituents (non-cellulose).

Also the following points of practical rather than theoretical import :—

(f.) The simple and accurate method of determining the "free base" in hypochlorite solutions, by direct titration after destroying the hypochlorite by means of hydrogen peroxide.

(g.) As a result of such determinations, that the proportion of free base to hypochlorite, in solutions made from bleaching powder at various concentrations, varies inversely with the concentration.

(h.) That bleaching, to be efficient, economical, and safe requires a careful regulation of the several factors; these are, translated from their more theoretical expression under (d), nature and proportion of the basic constituent, both free and combined; the temperature of the solution; and the nature (composition) of the negative or oxidising constituents.

(i.) The presence of organic chlorides in bleached esparto and straw pulps is a probable result of the bleaching process, requiring the careful attention of the paper-maker.

Before proceeding to our experimental matter, we may, perhaps, be allowed to offer a few considerations in *a priori* elucidation of these several heads of the subject.

(a.), (b.), (c.). If the chlorine of a hypochlorite solution act only indirectly, if, *i.e.*,  $\text{Cl}_2\text{O}$  act, as regards bleaching, as  $\text{O}_2$ , it will revert in the process entirely to the hydracid, that is, to  $\text{CaCl}_2$ . Chlorine or base otherwise combined, at the conclusion of the bleaching, than as calcium chloride, is an indication that the process is complicated by other reactions, *e.g.*, chlorination of the fibre constituents. Deficiency of "total chlorine," would indicate loss, either as volatile  $\text{ClO}$ -compounds, or an organic chloride of low molecular weight, *e.g.*, chloroform. By means of the inorganic constants alone, therefore, the problem of their mode of action admits of solution in general terms, and the experimental evidence which we have to offer in this paper is chiefly concerned with these.

Bleaching consists for the most part in the isolation of a pure cellulose from a compound cellulose. Such is the cotton bleach (cloth and yarn) and the linen "bottom bleach" (almost exclusively cloth), as well as the preparation of pulps for white papers. In these cases the bleaching process proper is preceded by a severe treatment of the raw material—generally a digestion with alkaline lye at high temperatures—which removes the greater proportion of the non-cellulose constituents, leaving the bleaching solution little more than residues of these to deal with. In contradistinction to these, there are the kinds of bleaching which are well called "whitewash" bleaches, in which the purpose is to whiten, as far as possible, a compound cellulose itself, by removing colouring matters, either adventitious (*e.g.*, chlorophyll and proteid residues) or products of change of the fibre substance. In such bleaches the purpose is to resolve the compound cellulose as little as possible, for resolution means loss of weight and, in cases where the fibre is made up of units of microscopic length—as in jute, with an ultimate fibre of 2—3 mm.—serious loss of strength. In this category we include, in addition to jute bleaching, whether for textiles or coloured papers, the various treatments of linen yarn known as "creaming," "half," and "three-quarters" bleach, and also some of the paper-makers' bleaches for low-grade papers. In all these cases the consumption of the bleaching-agent is relatively high. This, of course, immediately follows from the more-oxidisable character of the non-cellulose constituents of fibres. The distinction in this respect between cellulose and non-cellulose may be more definitely expressed in terms of their actual constitution, so far as ascertained. In the latter we have ketonic, aldehydic, and hydroxylic oxygen, either or all, with the "reactivity" characteristic of the presence of the CO, COH, and OH groups (Watts' Diet., New Ed., Art. Cellulose; also C. S. J., 1889, 199, and 1890, 1), whereas in cellulose we find none of the reactivity characteristic of an aldehyde or ketone; and even the hydroxyls, if present as such, are abnormally inactive. The directions of attack presented by such compounds to the alkaline hypochlorites are, in the case of the non-cellulose constituents, obviously,

oxidation and hydrolysis, and in lesser degree, also chlorination, the cellulose, on the other hand, remaining inert so long as a certain limit of intensity of oxy-hydrolytic action is not exceeded.

As subjects of experiment, we have confined ourselves for the most part to esparto and wood pulps prepared by the alkali process, as practised in a "news" mill; that is, pulps boiled so as to retain a maximum of non-cellulose constituents consistent with the degree of whiteness required for cheap printing papers. As bleaching agents, we have investigated the hypochlorites of calcium, sodium, and magnesium—the two latter prepared by double decomposition from the former—and, in addition, the solutions prepared by electrolysis from magnesium chloride.

(f.) A satisfactory method of estimating the oxides in presence of the hypochlorites (of the alkali and alkaline earths) has not, so far as we are aware, been proposed. The usual method of destroying the hypochlorite by ammonia, boiling off the excess and estimating the residual oxide, is open to an error resulting from the interaction of ammonia and hypochlorous acid, with formation of a volatile compound containing the elements of the latter combined with ammonia (Cross and Bevan, Proc. C. S. 1890, 22). That the results obtained are in excess of the actual amount of free lime present we have found by comparison of this method with those about to be described. The cause is obviously due to the loss of chlorine and the setting free of the equivalent of base.

The destruction of the hypochlorite by means of a solution of arsenious acid containing a known quantity of alkali, and subsequent titration of the total alkalinity, is unsatisfactory from the want of sharpness of the colour changes with the ordinary indicators. Sulphite of soda, oxidised by the hypochlorites to the neutral sulphate, appears to satisfy the required conditions; and the results obtained by subsequently titrating the free base in presence of phenolphthalein, are concordant and in fair agreement with those obtained by a method which we describe last, but which we should put first in order of simplicity and accuracy. This method consists in destroying the hypochlorite with hydrogen peroxide, which may obviously be used in excess without affecting the subsequent titration—and estimating the residual free base with acid; allowance being, of course, made in calculating the result for the acidity of the peroxide solution.

We subjoin the results of estimating the free lime in filtered solutions of calcium hypochlorite, which, besides illustrating the method, bring out a fact of some practical importance, *viz.*, that in dissolving bleaching powder the quantity of free lime taken up, calculated upon the chlorine strength, varies inversely with the concentration, *i.e.*, is approximately constant for the unit volume of solution.

Hydrogen peroxide: "10 vol." solution. Acidity, 1 cc. =  $0.34 \frac{\text{N}}{10}$  acid.

Hypochlorite solution: 10 cc. used in each determination.

—	Cc. $\text{H}_2\text{O}_2$	Free CaO per 10 cc.	Ratio. Free CaO to CaO as $\text{Ca}(\text{OCl}_2)$ .
10 grms. bleaching powder to 100 cc. ....	8.50	0.0070	1:20
7.5 grms. bleaching powder to 100 cc. ....	6.30	0.0073	1:14
5.0 grms. bleaching powder to 100 cc. ....	4.30	0.0087	1:8
2.5 grms. bleaching powder to 100 cc. ....	2.15	0.0078	1:4.5

The proportion of free to total combined CaO in the bleaching power itself has been found by O'Shea\* (C. S. J., 1883, 417) to be about 1:2.4; one-half of the combined CaO, however, reverts on solution to  $\text{CaCl}_2$ , the other half

\* These determinations were made by means of the ammonia method.

remaining combined with the chloroxygen group. The ratio of free to combined CaO expressed uniformly with those of the last column of the above table is 1:1.2; i.e., in a mixture of bleaching powder and water there is nearly as much CaO free, as in combination as hypochlorite.

It is obvious, therefore, that a sodium hypochlorite solution prepared by double decomposition from an unfiltered solution of bleaching powder will contain free soda in this proportion: prepared, on the other hand, from a clear solution, the proportions will be those of the above table.

Magnesium hypochlorite solutions similarly prepared show, on the other hand, a minimum basicity, owing to the relative insolubility of the oxide. These points are worthy of note in connexion with the practical work of bleaching.

We now pass to the experimental investigation of the bleaching process itself.

The points which we aimed at determining in the first place was, as already explained, (1), the proportion of base combined otherwise than as chloride in the exhausted mixture (solution and pulp); and (2), the distribution of the chlorine. As regards (1), the data required are relative rather than absolute, and we have taken as the measure of this factor the volume of standard acid required to neutralise in presence of methyl orange. The numbers obtained afford certainly minimum values for base not reverting to chloride, and a sufficiently exact comparison of the various solutions for the purposes of this investigation.

In our first experiments the conditions were such as obtain in the ordinary process of bleaching. The well-washed pulps (esparto and wood) were digested at ordinary temperatures

with the bleach liquor diluted to 20—30 times the weight of the pulp. The "available chlorine" taken varied from 2.2 to 6.6 per cent. of its weight. In calculating the results, due allowance was made for the free base in the bleach solution, and for the basicity of the water employed. The following were the general conclusions arrived at:—

The proportion of base not reverting is highest, and approximately the same, for soda and lime, lowest for magnesia, viz., from 20—to 30 per cent. for the two former, 10—15 per cent. for the latter. The proportion is higher in the early stages of the bleaching. As regards the distribution of the base between pulp and solution, an average of the numbers showed that in the case of lime,  $\frac{2}{3}$  was present in the pulp, in the case of soda only  $\frac{1}{3}$ , in the case of magnesia the distribution was approximately equal.

To establish an exact comparison between the several hypochlorites, in regard to this particular point, experiments were made in series and simultaneously, under uniform conditions, the base alone being varied.

The pulps were prepared by digesting in dilute acid and thoroughly washing off with distilled water; the bleaching solutions were also made up with distilled water. To give clearer expression to the results the numbers—for base—are given in cc. of normal acid required to neutralise. The particulars of the experiments are as follows:—

In each case we employed 20 grms. pulp and 500 cc. solution containing 1.010 grms. "available Cl," combined with base equivalent to 14.3 cc. normal acid. Experiments in each case simultaneous. Time, 18 hours.

Bleaching Agent.	Base as MOCI= cc. normal Acid.	Base after Exhaustion = cc. normal Acid.		Proportion of Base not reverting to Chloride.
		Pulp.	Solution.	
<b>A. Wood pulp:</b>				
Calcium hypochlorite .....	14.3	1.25	2.5	Per Cent. 26.0
Sodium hypochlorite .....	14.3	1.0	2.0	21.0
Magnesium hypochlorite.....	14.3	0.9	1.0	13.0
<b>B. Esparto pulp:</b>				
Calcium hypochlorite .....	14.3	2.2	2.0	29.5
Sodium hypochlorite.....	14.3	0.75	3.25	27.8
Magnesium hypochlorite.....	14.3	0.65	1.75	16.5

These results differentiate magnesia from lime and soda, the two latter appearing closely similar in their actions.

It was important to examine, in the next place, the mode of action of the bleaching solutions prepared by electrolysis magnesium chloride in dilute solution. We prepared two such solutions, uniform as regards chlorine strength with the above. In one we employed the pure salt alone; in the other magnesia was added in excess at the commencement of the electrolysis. The former solution, treated with the equivalent of sodium sulphite, was brought to exact neutrality; the latter was found to contain 12.5 per cent. of magnesia in excess of the equivalent of the chlorine set free by the electrolysis. Bleaching experiments were performed, using these solutions under conditions exactly uniform with the above. In the case of the first solution the mixture after exhaustion was neutral; in the second, the residual base was exactly that contained in the original solution in excess of the equivalent. *In these solutions, therefore, there appears to have been a complete reversion to chloride.*

The proof that the bleaching process is or is not attended with chlorination of the pulp constituents, is so far only inferential. Direct experimental evidence of chlorination in bleaching with calcium hypochlorite is afforded by the following observations:—Esparto pulp was freed from chlorine by digestion with dilute nitric acid (containing some sulphurous acid) and thoroughly washing. 6.4 grms. of this pulp was bleached under the usual conditions with 10 cc. of a solution of bleaching powder (7.2 per cent.), the total chlorine in which was estimated in a second quantity. After exhaustion the pulp was washed free from soluble

chloride, the original solution and washings being treated with silver nitrate. The washed pulp was "fused" with pure sodium hydrate, and the chlorine determined in the fused residue. The numbers obtained were:—

Cl in exhausted solution .....	0.2414
Cl in pulp .....	0.0255
Total Cl.....	0.2669
Total Cl in original bleaching solution.....	0.2750
*Difference .....	0.0081

The presence of an "organic" chloride in the pulp thus indicated was confirmed by taking a second portion, and after bleaching and washing till free from soluble chloride, drying and exhausting with alcohol. The alcoholic solution gave a copious precipitate of chloride with silver nitrate. On distilling off the alcohol from the remainder an oily residue was left, which was found to be partially soluble in sodium sulphite solution, with formation of sodium chloride.

Some years ago we called attention to the chlorination of the jute fibre in the bleaching of textiles with calcium hypochlorite, and the consequences attending the subsequent decomposition of the chloride in the fabric (Bull. Mulhouse, 1882). From what is now known of the constitution of the fibre substance there can be little doubt that it is the

\* This difference is a loss of Cl, either escaping during the bleaching process or in that of "fusing" the pulp with alkali, or it may be due to Cl in the exhausted solution not reacting with AgNO<sub>3</sub>. In any case the proportion is small.

ketonic oxygen which opens up a path to the chlorine, and the same cause would appear to be at work in the case of the esparto; for, on removing portions of the fibre, washing with water and sulphurous acid, and treating with magenta-sulphurous acid the characteristic reaction is obtained upon the fibre. The presence of a chloride in bleached pulps, not removable by washing, is a point of considerable practical importance. Such a chloride may be presumed to have acid properties, and its formation to account in part for the retention of the basic constituents of the bleaching solutions. The presence of the latter doubtless safeguards the fibre from being attacked by the hydrochloric acid, which would result from the hydrolysis of the chloride in the after processes of working up the pulp, but no doubt also tends to increase the discolouration ("going back") of the pulp resulting from the same cause. We would further remark that the presence of chlorine in a paper is also seen to be not necessarily an evidence of imperfect washing.

We now have to consider briefly the somewhat vexed question of bleaching efficiency. So far we have not spoken of the quality of the bleach obtained with the solutions which we have investigated. We do not, indeed, propose to go into a detailed account of the numberless experiments which we have made on this subject, in comparing the results obtained with the various systems used or proposed to be used. These more recent investigations have shown us that wide variations in efficiency are to be expected even when the actual bleaching agent is a constant quantity, and that no measure of the efficiency of a process will be complete which does not take into account the influence of the several factors. We cite one or two considerations in illustration. Thus *hydrolysis* will vary with the several bases, their proportion to pulp and solution, with the temperature, and evidently also with the condition of the pulp. *Oxidation*. The mode of action of the active constituent of a bleaching solution, we may assume, will not be seriously affected by variations of temperature within the ordinary range, nor directly influenced by the base with which it is combined. But the indirect influence of both, through the influence on the hydrolysis, may be considerable. We may cite experiments in illustration of these points.

(a.) *Temperature*.—Two identical solutions of bleaching powder were employed upon esparto pulp, the one being kept at 35° C. the other at 0°—4° C. The former was washed off when the solution was exhausted (2 hours), the latter allowed to remain until brought up to an equal colour (5½ hours). The residual chlorine in the latter was determined and found to amount to 20 per cent. of the original; also the proportion of base not reverting to chloride was found to be 40 per cent. less than in the experiment at the higher temperature.

(b.) *Quantity of base*.—Five solutions of sodium hypochlorite was prepared, in one the proportion  $\text{Na}_2\text{O} : \text{Cl}_2\text{O}$  was approximately normal, to the four remaining,  $\text{Na}_2\text{O}$  was added to increase the molecular proportion by 1, 2, 3, 4, units. At the conclusion of the experiment the solutions were found equally exhausted and the gradations of bleach were exactly in inverse order with the increments of base.

We should interpret these results as follows:—

(a.) At the lower temperature hydrolysis and tendency to chlorination are lessened: the same result as regards colour being obtained with a consumption of 20 per cent. less of the bleaching agent, the economy is due to limiting its work to that of simple oxidation, and the hydrolysis being a minimum, the oxidation is rather confined to the constituents upon the fibre, and not used up inefficiently in further oxidation of dissolved substances.

(b.) By the addition of alkali the hydrolytic tendencies are increased, and the sphere of oxidation is consequently extended, probably to the cellulose itself. It is well known in fact that with very alkaline oxidations the cellulose fibres are weakened, and fabrics are thus "tendered." The probable results therefore would be ineffectual oxidation—ineffectual, i.e., from being extended beyond the range of the colouring constituents, and even probably by promoting tendencies of the opposite kind to that desired, i.e., tendencies to fix the products of oxidation upon the cellulose.\*

\* In linen goods "dipped" before the "bottom" is "out" the difficulty of bleaching is much increased; the bottom, i.e., the non-cellulose residues, are fixed on the cellulose.

Lastly, the factor of *chlorination* in relation to bleaching efficiency is an element, *sui generis*, of the process of bleaching with the hypochlorites, upon the advantages or disadvantages of which we would not as yet pronounce any final opinion. Of course we may find that it plays a more important and necessary part than would appear from a study of the final products of the process. Further investigation of the hypochlorites, compared amongst themselves and with bleaching compounds into the composition of which chlorine does not enter, will lead to a definite solution of the problem, and this we have no desire to anticipate.

The presence of residual organic chlorides in bleached pulps is a disadvantage which we need not emphasise, and to the danger of which practical men will be alive. This matter certainly requires attention at their hands.

Generally we would remark that the familiar operations of the bleacher, to be recognised as definite chemical processes, require extended investigation and establishment upon a basis of exact knowledge, from which they will come to be presided over by instructed common sense and not by the much invoked genius of "fog."

## MOISTURE IN PAPER PULP.

BY MARTIN L. GRIFFIN, OF HOLYOKE, MASS., U.S.A.

MR. G. H. GEMMELL'S paper on moisture in paper pulp, read before this Society and published in the February number of its Journal, leads me to give the results of my experience as to the proper amount of atmospheric moisture allowable in paper pulp, and the methods of sampling and making the correction for atmospheric moisture. As the chemist to a soda fibre mill producing daily 30 tons of paper, this question was brought to my attention very early. Uniformity in results by different observers is not to be expected, because there are so many different conditions which enter into the question.

The term "atmospherically dry" as applied to paper pulp in its usual form is not satisfactory to me for this reason: it is not dried in the atmosphere, but usually passed over a machine to dry it, and while it is easy to dry a sheet of pulp in the air so that it does not feel moist, it is equally true that pulp may be atmospherically wet from such exposure. There is every gradation of moisture in this New England climate from the minute amount in January, when the thermometer is 25° below zero, to that during the rains of April and the thunder showers of July and August. I have a galvanised iron closet so arranged that neither snow nor rain can enter, yet freely accessible to air, for the exposure of samples; nevertheless, fog and mist, which we frequently have during certain seasons of the year, make samples so exposed feel wet to the touch. No one would call such fibre dry.

A certain amount of moisture should be allowed before a complaint is instituted, but this does not signify that the average limit should be aimed at any more than, when the law fixes the standard of purity of new milk at 13 per cent. milk solids, that all milk producers should reduce the quality to this standard. Every paper or pulp manufacturer would object to receiving lime air-dried or hide cuttings for sizing air-dry. Such cuttings easily absorb 20 to 25 per cent. moisture without feeling moist.

Not only do fibres from different sources acquire different proportions of moisture, but the same kinds differ in this respect according to the formation of the sheet and the pressure upon it. Some mills make a very light and soft fibre, while others make a stiff and harsh fibre from precisely the same kind of wood, and each takes up a different amount of moisture accordingly.

During the year 1884 I exposed daily about 10 samples of pulp dried at 212° in a room having windows open and with no fire, with the result that they gained by atmospheric moisture 6.38 per cent.

## MOISTURE TESTS.

Month.	Mean Departure Precipitation.	Increase by Moisture.
1886.	Inches.	Per Cent.
January .....	+ 2'02	5'74
February .....	+ 2'37	6'17
March .....	- 0'52	7'79
April .....	- 1'03	6'24
May .....	- 0'05	6'25
June .....	- 1'39	5'40
July .....	- 0'69	6'55
August .....	- 0'89	6'84
September.....	+ 0'03	8'30
October.....	- 0'73	8'28
November .....	+ 0'82	9'39
December.....	+ 1'05	7'55
Average.....	+ 0'99	7'04
1888.		
January .....	+ 0'78	5'66
February .....	+ 0'20	7'79
March .....	+ 1'77	8'69
April .....	- 0'72	6'76
May .....	+ 0'95	7'18
June .....	- 0'75	6'18
July.....	- 1'55	6'18
August .....	+ 1'19	6'99
September.....	+ 4'78	8'02
October.....	+ 1'71	7'02
November .....	+ 2'23	9'21
December.....	+ 1'04	9'21
Average.....	+11'76	7'40

Above I give the results of the average for each month in the year 1886 with the corresponding mean departure from the average precipitation for New England. There were from three to five samples previously dried at 212° exposed daily for a week in a well-aired brick storehouse, with cellar and good floor. These samples weighed 50 grms. each, were poplar fibre made by the alkaline process, and the sheets were comparatively thin, as pulp is usually made. It will be observed that the year 1886 was nearly normal in New England as regards precipitation, and substantially confirms my standard adopted, namely, 6'38 per cent.

I have also given the results for the year 1888, which were the same in method except that the samples were exposed in my air-closet referred to above, and the observations included weighing the same 20 samples daily without intermediate drying, allowing them to fluctuate according to the conditions of the atmosphere, and renewing them about once in three months.

It has been my experience that atmospheric changes will make as great a difference as 5 per cent. at times. 1888 was a wet year and the precipitation was 26'26 per cent. above the mean average during the whole year. The average increase by moisture for the year was 7'40 per cent.

As I sample most of the pulp in the consumer's mills, I remove a sample sheet about one-third the way in from the exterior usually, and in lots of 200 bales or rolls and

under 1 sample 10 per cent., above this number I take 20 samples.

To make the mathematical correction for atmospheric moisture, two methods may be employed. First, we may say that the standard is, in every 100 lb. commercial fibre, 94 lb. absolute fibre and 6 water, therefore, if we purchase fibre which dries so that every 100 lb. as invoiced weighs only 85 lb. containing 15 lb. water, to find what these 85 lb. would become atmospherically dry, the inquiry is stated thus: If 94 lb. become 100 on exposure to the air, what will 85 lb. become? The result is 90'42 lb. This method has nothing to do with percentage, and every reference to percentage should be avoided or mistakes will occur. The other method is purely by percentage. Let us consider the same example. Our standard by the first method was in 100 lb., 94 should be fibre and 6 water. If, therefore, 94 lb. fibre has gained 6 lb. water on exposure it has acquired 6'38 per cent. moisture and our percentage standard is 6'38. The problem is solved by finding 106'38 per cent. of 85, which gives 90'42 lb. as above.

My results have been generally accepted by domestic manufacturers. Foreign manufacturers have not as a rule come down to this standard, but no longer claim 12 per cent. in our markets here, and of late many invoices are dry according to the standard named in this paper.

For 1886 the excess percentage precipitation was 2'21 per cent.

The excess percentage of the average increase by moisture (7'04) over my standard (6'37) is 10'51 per cent.

For 1888 the excess percentage precipitation was 26'26 per cent.

The excess percentage of the samples over the standard was 16'17 per cent.

## DISCUSSION.

The CHAIRMAN said that members of the Society were always glad to have results of this sort which had been obtained in other climates and under circumstances different from those which generally ruled in England. If the phraseology of the paper were translated into the more ordinary language of this country, that would materially assist members in appreciating its full value. In a climate in which the difference between a wet and a dry day accounted for as much as 5 to 10 per cent. in the estimation of moisture in certain goods, the "precipitation of water which we call rain" became a matter of considerable importance.

Dr. C. R. ALDER WRIGHT did not see how a correction was to be applied to a bale of material only the outside of which was exposed to the air. If a truss of hay, for example, were exposed to a damp atmosphere for a few days, the outside of it might be thoroughly water-logged, while the inside might be in good condition. It would be better, he thought, though it might not be the usual practice of that trade, to adopt a system by which an average sample was estimated, and a *pro rata* deduction made for the moisture present, as was done in the case of the sale of pyrites and such like goods.

## Liverpool Section.

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The names in Italics are those of members of Committee who retire at the end of the current Session.

The following have been selected to fill the vacancies, and will take office in July next:—Chairman: A. Norman Tate; Vice-Chairman: F. Hurter; Secretary: Chas. A. Kohn; Treasurer: W. P. Thompson; Committee: C. L. Higgins, A. H. Knight, E. K. Muspratt, and A. Watt.

SESSION 1889-90.

Notices of Papers and Communications for the Meetings to be sent to the Local Secretary.

Meeting held on Wednesday, 7th May, 1890.

DR. J. CAMPBELL BROWN IN THE CHAIR.

### PHOTO-CHEMICAL INVESTIGATIONS AND A NEW METHOD OF DETERMINATION OF THE SENSITIVENESS OF PHOTOGRAPHIC PLATES.

BY FERDINAND HURTER, PH.D., AND V. C. DRIFFIELD.

#### I.—WHAT IS A PERFECT NEGATIVE? DENSITY, OPACITY, TRANSPARENCY.

THE production of a perfect picture by means of photography is an art; the production of a technically perfect negative is a science.

A perfect negative demands, in the first place, a perfect plate, and as the manufacture of dry sensitive plates is a large and rapidly-growing chemical industry, we need offer no apology for bringing this subject before this Society.

Our researches, which have covered a period of over 10 years, were made with a view to rendering the production of perfect negatives as far as possible a matter of certainty.

What is a perfect negative? A negative is theoretically perfect when the amount of light transmitted through its various gradations is in inverse ratio to that which the corresponding parts of the original subject sent out.

The negative is mathematically the true inverse of the original when the opacities of its gradations are proportional to the light reflected by those parts of the original which they represent.

Before we can clearly understand this definition, it will be necessary to state shortly the laws of absorption of light by opaque black substances, and to define clearly the meaning we attach to the terms opacity, transparency, and density of a negative. It is the more necessary to do this, as the whole of our investigations depend upon these laws.

For substances which do not reflect much light, such as black opaque bodies, or transparent coloured bodies, the relation between the light absorbed and the quantity of the substance present is very simple. If between the eye and a source of light we place a thin layer of dilute Indian ink, that layer absorbs light, and thereby reduces the intensity of the light transmitted. Assume that such a layer absorbs one-half of the light, then one-half of the light will be transmitted. Whatever may be the intensity of the original light, the intensity after passing this layer of ink will be one-half of what it was. The interposition of two such layers will reduce the light to  $\frac{1}{4}$  the original intensity; three such layers will reduce it to  $\frac{1}{8}$ , and so on, each layer reducing the intensity to one-half of what it receives.

Had the first layer allowed  $\frac{1}{3}$  of the light to pass through, then two such layers would reduce the intensity to  $\frac{1}{9}$ , three layers to  $\frac{1}{27}$ , &c. In general any number of layers would reduce the intensity of the light to a fraction, which is equal to the fraction the first layer allows to pass, but raised to a power, the index of which is the number of layers employed. If  $n$  equal layers were employed, and the first one reduced the intensity of the light to a fraction  $\frac{1}{m}$ , the  $n$  layers would reduce it to  $\left(\frac{1}{m}\right)^n$ .

If instead of using so many successive layers, the first layer were made to contain as much Indian ink as the  $n$  successive layers contain altogether, we should find that the one layer now reduces the intensity of light by exactly the same amount as the  $n$  layers did. The reduction of the intensity is of course due to the black particles, and depends simply upon the number of them which are interposed per unit of area. We can thus replace the number of layers by the number of particles, and the law takes this form:—The intensity  $I_x$  of light after passing  $A$  molecules of a substance is a fraction of the original intensity  $I$ , such that—

$$\frac{I_x}{I} = \left(\frac{1}{C}\right)^A$$

For purely mathematical reasons the fraction  $\frac{1}{C}$  is usually expressed as a negative power of the base of the hyperbolic logarithms  $e$ , say  $\frac{1}{C} = e^{-k}$ , and we can write—

$$\frac{I_x}{I} = e^{-kA}$$

where  $k$  is called the coefficient of absorption. This form of the law we shall frequently use again. The fraction  $\frac{I_x}{I}$  represents and measures the transparency of the substance.

The inverse of that fraction, or  $\frac{1}{\frac{I_x}{I}} = \frac{I}{I_x} = e^{kA}$  measures the opacity of the substance. It indicates what intensity of light must fall on one side of the substance in order that unit intensity may be transmitted.

In our investigations we use the letter  $T$  to denote transparency, and  $O$  to denote opacity, and the two symbols are related thus:  $O \times T = 1$ .

We must further define what we mean by density as distinct from opacity. By density we mean the number of particles of a substance spread over unit area, multiplied by the coefficient of absorption;  $kA$  is what we term density, and mark by the letter  $D$ .

For our purposes, *i.e.*, in its application to negatives, the density is directly proportional to the amount of silver deposited per unit area, and may be used as a measure of that amount.

The relations between the three terms, transparency, opacity, and density, are the following:—

$$T = e^{-D}$$

$$O = e^D$$

$$D = \log e O = -\log e T$$

The density is the logarithm of the opacity, or the negative logarithm of the transparency.

These relations hold good for some substances with regard to ordinary white light, for others only with regard to monochromatic light, and for others they do not hold good

at all. We have satisfied ourselves that they do hold good for the silver deposited as a black substance in negatives so long as the silver does not assume a metallic lustre, and reflects but a very small amount of light.

By means of these definitions we are now in a position to trace the connexion between the densities of a theoretically perfect negative and the light intensities which produced them.

Since the density is the logarithm of the opacity, and since, in a theoretically perfect negative, the opacities are directly proportional to the intensities of the light which produced them, it follows that each density must be proportional to the logarithm of the light intensity which produced it. (More correctly, the density is a linear function of the logarithm of the intensity of light and time of exposure.)

The result is this:—In a theoretically perfect negative, the amounts of silver deposited in the various parts are proportional to the logarithms of the intensities of light proceeding from the corresponding parts of the object.

The question arises, can such a negative be produced in practice?

In order to answer this question, we had first to find a simple method of measuring the density of the silver deposited in negatives. We had then to study the influence of the developers upon the density of the deposits, and we were then in a position to investigate the action of the light itself.

## II.—INSTRUMENT FOR MEASURING DENSITIES.

We proceed to describe the instrument for measuring the density of the deposit. It is based on the relation existing between density and opacity. We measure the opacity of the plate, and in order to avoid calculations and references to tables of logarithms, the scale of the instrument is so arranged as to read the logarithm of the opacity, which is the density. The reason why we prefer to have the results expressed as density is because the density is a measure of the amount of silver deposited, or of the chemical work done by the light.

The instrument pictured in Figs. 1 and 2 consists essentially of a small Bunsen photometer similar to those used for testing the illuminating power of gas, &c. The paper disc with its grease spot is placed in a small cubical chamber. The chamber carries an eye-piece, through which an image of each side of the disc can be viewed in two small mirrors, and so compared. The chamber can be made to slide in a straight line on a support by turning a key connected to one of two pulleys, over which passes an endless cord attached to the chamber. This arrangement is placed within a larger box, the ends of which have apertures through which light is admitted from two powerful petroleum lamps. Corresponding exactly with these apertures, similar apertures are bored into the sides of the small chamber, which admit the light to either side of the Bunsen disc. The dimensions we have adopted are, for the larger box, 12 in. long, 6 in. high, and 4 in. deep. The small chamber is a cube measuring 2 in. inside. We find it necessary to blacken everything within the box except the scales, and it is also important to exclude all extraneous light by means of a screen. The heat of the lamps also very soon injures the woodwork unless it is covered with asbestos cardboard and sheet metal.

The aperture in the left-hand end of the large box we reduce to about  $\frac{1}{4}$  in. diameter by a diaphragm. At this end is placed the plate to be measured, held in position by springs. The hole at the right-hand end of the box is reduced by a rectangular diaphragm,  $\frac{1}{4}$  in. wide and  $\frac{1}{2}$  in. long, the length being vertical. This diaphragm can be reduced in length by moving a taper diaphragm past it.

The instrument is provided with two scales, one fixed, the other moveable, the use and construction of which we will now explain. The fixed scale indicates the position of the disc chamber, and is constructed as follows:—

Suppose that the lamp on the left-hand side gives light of the intensity  $I_1$ , and that on the right-hand side light of intensity  $I_2$ , and that both lamps are equidistant from the centre of the instrument, and that this distance is  $l$ .

Then, when the disc chamber has been moved to a distance  $x$  from the centre of the instrument, so that—

$$(1) \quad \frac{I_1}{(l-x)^2} = \frac{I_2}{(l+x)^2} \text{ or } \frac{I_1}{I_2} = \left(\frac{l-x}{l+x}\right)^2$$

the two images of the disc will be alike. If a plate is now inserted, which reduces the light from the intensity  $I_1$  to intensity  $i$ , then the disc chamber will have to be moved to another place nearer to the plate before the two images of the disc are alike again. Supposing the distance of the disc from the centre of the instrument is now  $y$ , then—

$$(2) \quad \frac{i}{(l-y)^2} = \frac{I_2}{(l+y)^2} \text{ or } \frac{I_2}{i} = \left(\frac{l+y}{l-y}\right)^2$$

By multiplying the two equations we find the fraction, which measures the opacity—

$$\frac{I_1}{i} = \left(\frac{l-x}{l+x}\right)^2 \left(\frac{l+y}{l-y}\right)^2$$

Taking logarithms on both sides, we have, since  $\log. \frac{I_1}{i}$  is the density  $D$  of the plate,

$$D = \log. \left(\frac{l+y}{l-y}\right)^2 - \log. \left(\frac{l+x}{l-x}\right)^2$$

If therefore at the distances  $x$  and  $y$  we write on the scale the values of  $\log. \left(\frac{l+x}{l-x}\right)^2$  and  $\log. \left(\frac{l+y}{l-y}\right)^2$  we can simply read off these logarithms, subtract one from the other, and the result is the density of the plate. For general convenience we use vulgar and not hyperbolic logarithms. In order to avoid all errors arising from the distance of the lamps, we make the apertures in the box small compared with the luminous portion of the flame of the lamps; it can be shown that in that case the distance  $l$  must be measured between the centre of the instrument and the diaphragm.

The following table gives the relative distances of the various points of the scale from the centre of the instrument, at which the logarithms of  $\left(\frac{l+x}{l-x}\right)^2$  have the values 0.1, 0.2, 0.3, &c.,  $l$  being half the distance between the diaphragms.

TABLE I.—FIXED SCALE OF INSTRUMENT.

$\log. \left(\frac{l+x}{l-x}\right)^2$	Distance from Centre of Instrument.	$\log. \left(\frac{l+x}{l-x}\right)^2$	Distance from Centre of Instrument.
0.000	$l \times 0.000$	0.300	$l \times 0.476$
0.100	$l \times 0.057$	1.000	$l \times 0.519$
0.200	$l \times 0.114$	1.100	$l \times 0.560$
0.300	$l \times 0.171$	1.200	$l \times 0.599$
0.400	$l \times 0.226$	1.300	$l \times 0.634$
0.500	$l \times 0.280$	1.400	$l \times 0.667$
0.600	$l \times 0.332$	1.500	$l \times 0.698$
0.700	$l \times 0.382$	1.600	$l \times 0.726$
0.800	$l \times 0.430$	1.700	$l \times 0.752$

Suppose, as in our case, the box were 12 in. long between the diaphragms, then  $l$  is 6 in. The centre of the instrument is marked with zero, and we find from the table that 0.500 must be placed at  $6 \times 0.280$  in. from the centre on both sides of the centre. Similarly other points of the scale are found by means of the table. The scale on both sides of zero is symmetrical. The distances between the points so found are subdivided into equal parts. This is not absolutely necessary, but it is convenient.

The moveable scale (see Fig. 3) is attached to the upper edge of the taper diaphragm, which is used for reducing the amount of light admitted through the rectangular opening. This taper diaphragm is made of sheet metal about 12 in. long and 2 in. wide, out of which is cut a triangular opening about  $10\frac{1}{2}$  in. in length from base to apex, the width of the



base being  $\frac{1}{2}$  in. It is essential that the sides of this triangle be absolutely straight lines. The scale attached to this taper diaphragm is constructed as follows:—From the apex we measure 10 ins. exactly towards the base, this gives the zero point of the scale. The other points of the scale are marked so as to read directly the densities. At any distance  $x$  from the apex the area of the opening and, with it, the intensity of the light will be reduced as  $10:x$ , and the vulgar logarithm of the fraction  $\frac{10}{x}$  is the corresponding density with which the scale is marked. For convenience we append table showing the distances from the apex, at which the figures 0.1, 0.2, 0.3, &c. are to be placed:—

TABLE II.—MOVEABLE SCALE.

Value of Log. $\frac{10}{x}$ .	Distance from Apex.	Value of Log. $\frac{10}{x}$ .	Distance from Apex.
	Inches.		Inches.
0.0	10	0.50	3.16
0.5	8.91	0.60	2.51
1.0	7.94	0.70	2.00
2.0	6.31	0.80	1.58
3.0	5.01	0.90	1.28
4.0	4.00	1.00	1.00

Intermediate points are obtained by subdivision into equal parts.

An index is fixed to the inside of the box over the centre of the rectangular diaphragm pointing to the number to be read. The Figures 1 and 2 will help to make this description clearer.

Two examples will show how the instrument is used.

1. When measuring a small density we move the sliding scale to zero, and the disc chamber to such a position that the images of the Bunsen disc are alike. We then insert the plate to be measured, and without altering the position of the disc chamber slide the moveable scale until equality is restored. The density will then be indicated by the fixed index on the diaphragm scale.

2. In the case of a high density we place the sliding scale to 0, and by placing a piece of opal glass outside the box, between it and the lamp, we reduce the light on the right-hand side until the disc chamber requires to be moved almost up to the right-hand end of the box in order to secure equality of the images. If necessary, we move the lamp further away. When equality is thus secured, we read the number below the index of the disc chamber on the fixed scale. We then insert the plate to be measured, and move the disc chamber to the left until equality is again restored. If that cannot be done by the movement of the disc chamber alone, it can be obtained by using the moveable scale in addition.

Suppose the index stood at 1.100 on the right, and afterwards at 1.55 on the left of zero, then the density would be  $1.100 + 1.55 = 2.65$ .

If the index stood at 1.10 to the right and afterwards at 1.7 to the left, and equality could then only be restored by using the moveable scale as well, and its index pointed to .75, then the density would be  $1.10 + 1.7 + 0.75 = 3.55$ . Higher numbers than 3.55 do not occur in ordinary negatives. A plate, the density of which is 3.55, only transmits  $\frac{1}{355}$ th part of the light it receives.

The general rule for finding the density is: Consider the numbers to the right of zero as negative numbers, those to the left as positive. Subtract the first reading from the second; the result is the density. If the moveable scale be used as well, the amount it indicates must be added.

It will hardly be necessary to say that a plate of density 1 permits  $\frac{1}{10}$  of the light to pass, and that a plate of density 2 permits  $\frac{1}{100}$  of the light to pass, since 1 is the log. of 10, and 2 that of 100.

With this instrument we have obtained fairly accurate results. Analyses of mixtures of Indian ink and water, indigo solution and water, and of many other substances have been made by it. The following analyses are given to show the capabilities of the instrument:—

1. Experiment with Indian ink.—An Indian ink solution was mixed with water in known proportions, and the density of one solution being known, that of the others was calculated. The following table shows the observed and calculated densities. The calculated density is simply proportional to the amount of Indian ink employed.

TABLE III.—EXPERIMENT WITH INDIAN INK.

Indian Ink employed to 100 cc. of Water.	Density calculated.	Density found.	cc. of Indian Ink found.
cc. 5	.240	.240	5.00
10	.480	.500	10.42
15	.720	.750	15.62
20	.960	.950	19.80
25	1.200	1.245	25.90
30	1.440	1.440	30.0
35	1.680	1.665	34.7
40	1.920	1.885	39.3

The greatest error made does not reach 4 per cent. of the total amount, and even better results can be obtained if more than one reading be taken. But this accuracy is quite sufficient for photographic purposes, where, from other causes, still greater errors are liable to arise, as will presently be shown.

Sometimes, when using the instrument for analysing solutions of coloured salts, a peculiar difficulty arises from the different colours of the two images of the Bunsen disc. This is easily overcome by viewing the disc through appropriately coloured glass, red, green and blue glasses being the most useful. The following experiment with indigo solution is representative of one of the most difficult, since dark blue glass was used to view the disc.

TABLE IV.—INDIGO SOLUTION.

Indigo Solution Employed.	Indigo found.	Density calculated.	Density found.
cc. 100	96.9	1.554	1.487
50	50.6	.777	.787
25	24.1	.388	.375
10	10.0	.155	.155

It will be seen again that the results are only accurate within five per cent. of their value.

With regard to the lamps they should be powerful petroleum lamps with duplex burners. The flames should be in planes, at right angles to the axis of the instrument. Very erroneous results are obtained if Argand burners are used. The lamps should be placed close to the diaphragms, and it is advisable to provide a small stage outside the diaphragm to hold coloured glasses, when a substance requires investigation in light of a particular colour.

Captain Abney has also devised an instrument for measuring transparencies. His instrument consists of a Rumford shadow photometer as indicator, and of a revolving sector, which can be closed or opened whilst revolving, as a measure of the transparency. Apart from the fact that a Bunsen disc is more sensitive than the shadows, there is a fallacy in the assumption that the amount of light, which passes through a revolving sector is proportional to the angle to

which the sector is opened. Experiments made for the purpose show that the amount of light passing through a revolving sector is more correctly represented by a formula—

$$I_s = 1 \frac{\phi}{360} + C.$$

where  $I_s$  is the light transmitted by the sector,  $I$  the intensity falling upon the sector,  $\phi$  the angle of opening and  $C$  a constant, which depends upon the relative position of the lamp, the sector and the screen, and is, in fact, due to the semi-shadow on both edges of the sector openings. The error caused by this constant is small with plates of low density but it rises to over 100 per cent. with plates of high densities, which renders the results utterly untrustworthy.

We have thought it necessary to give this lengthy description of our instrument, since we consider it a very important one; it is for photographic experiments as indispensable as the balance is in analysis. The instrument is capable of other applications; its indications can always be translated into weights by simply multiplying them with a factor. It is, therefore, capable of applications in analysis.

### III.—DEVELOPMENT.

There is a generally accepted belief among photographers that a great amount of control can be exercised in development over the density and the general gradations of a negative, and in this respect alkaline pyrogallol enjoys a special reputation. On this account we have chosen this developer for the following series of experiments, except where otherwise stated. These experiments conclusively show that the only control the photographer has over development lies in securing a greater or less density of image (the former often only at the expense of fog), and that he has no control whatever over the gradations of the negative.

The plan we have adopted in carrying out these experiments is to subject *pieces of one and the same plate* to the varying conditions the influence of which, on the density or the gradation, is the subject of our investigation. A precaution we have always taken, except in our earliest experiments, is never to develop a piece of a plate which has been exposed to the light without simultaneously submitting to the same developer a piece of the same plate which has *not* been exposed, and which we term the "fog strip." The object of this precaution is to ascertain exactly how much of the resulting density is due to the action of the light and how much is due to incidental fog, including therein, fog inherent in the plate or caused by injudicious development, and also the density due to glass and gelatin.

In the following series of experiments, made to ascertain the influence of time, of development, and composition of the developer on the density, we covered up one half of a plate and exposed the other half to a standard light, as will be presently more fully explained. After exposure we cut up the plate in such a way that each piece included a portion of the unexposed and a portion of the exposed plate. Each strip was then developed, such modification in time of development or composition of developer being made as formed the subject of the investigation. The resulting densities were then measured after fixing, washing, and drying,

#### Time of Development.

*Experiment 1.*—Plate: "Wratten Ord." Exposure: ?

Developer, 100 cc. contain  $\begin{cases} 0.085 \text{ g. NH}_3, \\ 0.400 \text{ pyrogallol.} \\ 0.250 \text{ NH}_4\text{Br.} \end{cases}$

#### Results.

Time of development.....Minutes	2.5	5.0	7.5	10.0	12.5	15.0
Densities produced ..	.183	.543	.793	1.160	1.110	1.117
Percentage .....	15.6	46.0	67.8	99.1	94.0	100.0

*Experiment 2.*—Plate: "Wratten Ord." Exposure = 60 C.M.S.

Developer, 100 cc. contain  $\begin{cases} 0.162 \text{ NH}_3, \\ 0.342 \text{ Pyro.*} \\ 0.228 \text{ NH}_4\text{Br.} \end{cases}$

Time of development Minutes..	1.25	2.5	5	10	15
Density exposed plate....	.775	1.175	1.725	2.275	2.475
Density unexposed plate .	.155	.270	.510	.590	.790
Density due to light .....	.620	.905	1.215	1.685	1.685
Percentage developed ....	36.8	53.7	72.1	100	100

*Experiment 3.*—Plate: "Wratten Ord." Exposure = 20 C.M.S.

See Diagram No. 1. Developer, 100 cc. contain:—

$\begin{cases} 0.162 \text{ NH}_3, \\ 0.342 \text{ Pyro.} \\ 0.228 \text{ NH}_4 \text{ Br.} \end{cases}$

Time of development. Minutes	2.5	5	7.5	10	15	20
Density exposed plate .....	.670	.965	1.245	1.420	1.755	1.945
Density unexposed plate ..	.200	.345	.415	.505	.575	.710
Density due to light.....	.470	.620	.830	.915	1.180	1.235
Percentage developed.....	38.0	50.2	67.2	74.1	95.5	100

These experiments show that the total density grows with the time of development, but that the density due to light reaches a limit in about 15 minutes. The continued growth of the total density is due to the action of the developer upon the bromide of silver which had not been affected by the light.

#### Variation of Pyrogallol.

*Experiment 4.*—Plate: "Wratten Ord." Exposure ?

Developed each strip four minutes in a developer, containing in 100 cc =  $\begin{cases} 0.1156 \text{ NH}_3 \\ 0.2000 \text{ NH}_4\text{Br} \end{cases}$

Pyrogallol.....grms..	0.08	0.16	0.32	0.64
Relative amount .....	1	2	4	8
Density.....	1.036	1.506	1.526	1.500

*Experiment 5.*—Plate: "Wratten Ord." Exposure = 60 C.M.S. See diagram No. 2.

Developed 4 minutes in a developer, 100 cc. =

$\begin{cases} 0.162 \text{ NH}_3 \\ 0.228 \text{ NH}_4\text{Br} \end{cases}$

Pyrogallol used.....grms.	.057	0.114	0.228	0.457	0.914	1.828
Relative amount.....	1	2	4	8	16	32
Density exposed plate	.595	.840	1.060	1.215	1.150	1.040
Density unexposed plate	.180	.195	.190	.130	.135	.105
Density due to light...	.415	.645	.870	1.085	1.015	.935

\* Sulpho-pyrogallol equivalent to Pyro.

From these results we gather that an excess of pyrogallol beyond a certain limit tends to retard development and the production of density. This limit appears to be the equivalent of pyrogallol necessary to convert the ammonia into tribasic pyrogallate,  $C_6H_3(O\dot{N}H_4)_3$ .

A similar experiment is here given, made with "sulpho-pyrogallol" compounded with sodium sulphite and citric acid. It will be evident that the presence of the acid, by neutralising the ammonia, is responsible for the much more marked falling off in density.

*Experiment 6.*—Plate: "Manchester Slow." Exposure = 40 C.M.S.

Developed 4 minutes, 100 cc. =  $\begin{cases} 0.162 \text{ NH}_3 \\ 0.228 \text{ NH}_4\text{Br} \end{cases}$  and "sulpho-pyrogallol" corresponding to  $x$  grms. pyrogallol.

Pyro.....grms. $x$ =	0.114	0.228	0.457	0.914	1.371	1.828
Relative amount.....	1	2	4	8	12	16
Density exposed plate	.940	1.710	1.610	1.359	.790	.105
Density unexposed plate.....	.360	.620	.495	.240	.110	.080
Density due to light.	.580	1.050	1.115	1.110	.590	.025

#### Variation of Ammonia.

*Experiment 7.*—Plate: "Wratten Ord." Exposure?

Developed 4 minutes in developer, 100 cc. =

$$\begin{cases} x \text{ grms. NH}_3 \\ 0.40 \text{ Pyro.} \\ 0.20 \text{ NH}_4\text{Br} \end{cases}$$

Ammonia ....grms.	0.0231	.0462	.0925	.185	.277	.370
Density.....	0.00	.613	1.276	1.816	2.136	2.266

*Experiment 8.*—Plate: "Wratten Ord." Exposure 20 C.M.S. See diagram No. 3.

Developed 4 minutes in 100 cc. =  $\begin{cases} x \text{ NH}_3 \\ 0.34 \text{ Pyro.} \\ 0.23 \text{ NH}_4\text{Br.} \end{cases}$

Ammonia.....grms. $x$ =	.103	.207	.414	.828	1.656	3.312
Relative amount .....	1	2	4	8	16	32
Density exposed plate..	.340	.530	.960	1.675	1.710	1.470
Density unexposed plate	.090	.120	.265	.700	1.310	1.390
Density due to light ...	.250	.410	.695	.975	.400	.170

*Experiment 9.*—Plate: "Manchester Slow." Exposure 40 C.M.S. See diagram No. 4.

Developed 4 minutes in 100 cc. =  $\begin{cases} x \text{ NH}_3 \\ 0.34 \text{ Pyro.} \\ 0.23 \text{ NH}_4\text{Br.} \end{cases}$

$x$ grms. $\text{NH}_3$ .....	.207	.414	.828	1.242	1.656	3.726
Relative amount .....	1	2	4	6	8	18
Density exposed plate..	.250	1.530	2.290	2.470	2.470	1.865
Density unexposed plate	.090	.550	1.400	1.880	2.015	1.145
Density due to light....	.160	.980	.890	.590	.455	.420

The general result of these experiments is that the addition of ammonia, up to a certain extent, increases the density in a given time, but that the amount of ammonia which can be added without giving rise to fog, and without simultaneously adding bromide, is very limited. The so-called accelerating action of ammonia being due almost

entirely to its solvent action on bromide of silver, which, if the ammonia is increased sufficiently, results in greatly diminishing the density.

The following table shows the solubility of silver bromide in very dilute ammonia, such as is used for development of plates:—

100 cc. of dilute ammonia containing:—

1.105 grms. $\text{NH}_3$ dissolve	0.0576 AgBr
.555 " "	0.0206 " "
.462 " "	0.0100 " "
.0897 " "	0.0052 " "

The last two solutions represent the extreme concentrations usually employed by photographers for development.

If, to any of these solutions of silver bromide in ammonia bromide of ammonium be added, an immediate precipitate of bromide of silver is the result. The so-called accelerating action of ammonia, and the retarding action of ammonium bromide, are probably due entirely to this solvent action of the one, and the anti-solvent action of the other of these two reagents. The rapid production of fog when ammonia is increased is due to the fact that when pyrogallol solution is added to an ammoniacal solution of bromide of silver, the silver in solution is precipitated immediately in the metallic state.

The following experiments show the influence of

#### Variation of Ammonium Bromide.

*Experiment 10.*—Plate: "Wratten Ord." Exposure?

Developed for 4 minutes, 100 cc. =  $\begin{cases} 0.123 \text{ NH}_3 \\ 0.375 \text{ Pyro.} \end{cases}$

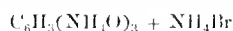
Ammonium bromide grms. in 100 cc.....	0.00	0.10	0.20	0.40	0.80	1.28
Relative amount .....	0	1	2	4	8	12.8
Density .....	1.81	1.73	1.61	1.43	0.34	0.00

*Experiment 11.*—Plate: "Wratten Ord." Exposure = 40 C.M.S. Developed 4 minutes in 100 cc. =  $\begin{cases} 0.162 \text{ NH}_3 \\ 0.312 \text{ Pyro.} \end{cases}$  and various amounts of bromide See Diagram No. 5.

Ammonium bromide	.657	.114	.228	.457	.918	1.828
Relative amount ....	1	2	4	8	16	32
Density exposed plate .....	1.450	1.335	1.235	1.025	.685	.429
Density unexposed plate .....	.560	.455	.315	.255	.130	.090
Density due to light.	.890	.880	.920	.770	.555	.630

It is clear that development in both experiments was entirely prevented in these four minutes when the amount of bromide was about 10 times that of ammonia present.

It also appears from our experiments that a rational developer would consist of a decinormal solution of ammonia, containing so much pyrogallol and ammonium bromide as would correspond with the formula—



We have represented many of these results in the form of diagrams. It will be interesting just to point to diagrams Nos. 1, 3, and 4, to show the great amount of action which the alkaline developer may have on the bromide of silver, although it has never been exposed.

This disagreeable property is common to all alkaline developers, and it renders them unsuitable for scientific investigations. In all our important work we use exclusively the ferrous oxalate developer for the reason that it attacks unexposed bromide of silver so slowly, that within one hour

and even more, no appreciable density can be developed upon a really good plate. Nor does its action vary much with its composition. The addition or omission of bromide from the constitution of this developer does not seem to have any great influence, and a greater or less concentration of the reagents within considerable limits does not affect its action; indeed, we have not found any variation to arise from alterations in its composition, excepting the length of time needed for completion of development.

The following table shows how the density of an exposed plate grows as the time of development is prolonged from five minutes to one hour.

*Experiment 12.—Ferrons Oxalate.*

Time.	Density exclusive of Fog.				
	I.	II.	III.	IV.	V.
Minutes.					
5	.365	.350	..	..	.215
10	.525	.430	..	..	.305
15	.615	.550	.735	.570	.410
20	.615	.575	..	..	.420
25	.700	.650	..	..	..
30	.700	.660	.860	.670	.450
45	..	..	1.000	.715	.545
60	..	..	..	.740	..

Columns I. and II. are the results obtained upon the same plate, one (I.) portion of the strips developed in a developer consisting of 4 parts of a saturated solution of potassium oxalate, mixed with 1 part of a saturated solution of ferrons sulphate, the other (II.) portion of the strips developed in the same developer diluted with an equal volume of water. Columns III. and IV. represent other experiments, the plates being developed with the saturated solution. Whilst I. to IV. were developed with a small amount of bromide of potassium added to the developer, No. V. was developed without bromide. In not one instance did the density of the unexposed portions of the plate amount to more than 0.098, which is the density due to clear glass and gelatin. That ferrons oxalate does not, however used, attack silver bromide which has not been exposed to light is a most valuable and characteristic property of this developer.

An important result of this series of experiments is that the density reached is dependent upon the time of development as well as upon the exposure of the plate. The time it takes to reach a given density varies much with the gelatin employed in making the emulsion and the age of the plate. But with each plate it obeys a certain law, which is more or less clearly visible in every one of the five experiments. The density grows rapidly at first, its growth becoming slower as time advances, and finally tends to a limit. Each experiment is, taken by itself, liable to many errors; but by reducing every experiment to the densities obtained in No. IV., in simple proportion, the following tabulated series of numbers is obtained:—The columns marked I., II., &c., are the reduced densities of the corresponding columns of experiment 12. The column marked "Mean" shows the arithmetical mean for any period of development obtained from the five series. The column "Calculated" is obtained by means of a formula based upon the idea that the number of particles of bromide of silver affected by the light is greatest in the front layer of the film and decreases in geometrical progression as each succeeding layer of the film is reached, an idea which will be better appreciated when we have explained the action of the light upon the film. This idea expressed algebraically leads to the formula—

$$D_t = D(1 - \alpha^t)$$

where  $D_t$  is the density after  $t$  minutes development,  $D$  the limit of density reached by very prolonged development,

and  $t$  the time of development,  $\alpha$  is a fraction depending upon the nature of the film, concentration of developer, temperature, &c. The constants for the series of figures below are  $D = 0.720$ ,  $\alpha = 0.9015$ .

Time.	Recalculated.—Densities.					
	I.	II.	III.	IV.	V.	Mean. Calculated.
Min.						
5	.349	.350	..	..	.298	.332 .290
10	.502	.460	..	..	.423	.462 .464
15	.588	.550	.569	.570	.561	.569 .568
20	.588	.575	..	..	.583	.582 .628
25	.670	.650	..	..	..	.660 .665
30	.670	.660	.615	.670	.625	.645 .687
45	..	..	.715	.715	.715	.715 .713
60	..	..	..	.740	..	.740 .719

The relation of the calculated figures to the experimental data is best seen in diagram No. 6.

A very important conclusion can be shown to proceed from the formula representing the course of development.

If on any one plate two exposures are given, one of which would ultimately yield density  $D_1$  and the other  $D_2$ , and if this plate were developed for a time  $t$ , then two densities,  $d_1$  and  $d_2$ , would result such that:—

$$d_1 = D_1(1 - \alpha^t)$$

$$d_2 = D_2(1 - \alpha^t)$$

and it will be seen that on dividing these equations,

$$\frac{d_1}{d_2} = \frac{D_1}{D_2}$$

the resulting ratio is independent of the time of development, and is equal to the ratio of the ultimate densities which would be reached, so that the gradation of negatives appears to be independent of the time of development.

#### IV.—GRADATION.

The above experiments have shown that with a well-balanced developer there is a limit to density, which depends upon the action of the light, and that, so far, the only control the photographer has lies in deciding whether he will reach that limit or not.

It also became evident that if two different densities be developed upon the same plate to their extreme limits, the ratio existing between these limits must depend solely upon the action of the light. The question we have now to consider is whether it is possible by any modification of development to influence this ratio, and whether this same ratio exists at all stages of development.

In making these experiments, the source of light we have adopted is a standard candle placed at 1 metre distance from the plate. We then produce a number of gradations upon the plate by exposing different portions of it to the light for different periods of time, always leaving one portion of the plate unexposed.

In order to show that the length of time of development does not affect the ratio of densities among themselves, but increases every density by proportional amounts, we give the following experiments, made with various plates, ferrons oxalate being the developer used. The tables show the densities obtained, their ratio among themselves, and the ratios of the two corresponding densities obtained by long and short development.

All densities are given exclusive of fog (the density of unexposed plate being subtracted from density of exposed plate).

## Experiment 13.—Gradations. Ferrous Oxalate.

Plate used.	Exposure, C.M.S.	Short Development, 4 Minutes.		Long Development, 12 Minutes.		Ratio, $\frac{D_2}{D_1}$	Remarks.
		Density $D_1$ .	Ratio.	Density $D_2$ .	Ratio.		
Ilford Red Label. (1.)	10	·775	1·0	1·260	1·0	1·63	Greatest error, $\pm$ 2·4 per cent.
	20	1·000	1·20	1·660	1·31	1·66	
	40	1·180	1·52	1·96	1·55	1·66	
	80	1·250	1·61	2·08	1·65	1·60	
				Mean.....		1·64	
Wratten's Drop shutter. (2.)	10	1·17	1·0	1·74	1·0	1·50	Greatest error, $\pm$ 3·4 per cent.
	20	1·67	1·42	2·37	1·36	1·42	
	40	2·06	1·76	2·91	1·67	1·41	
	80	2·26	1·93	3·33	1·91	1·47	
				Mean.....		1·45	
"United Kingdom." (3.)	10	·160	1·0	·275	1·0	1·70	Greatest error, $\pm$ 5 per cent.
	20	·313	1·95	·485	1·76	1·55	
	40	·518	3·23	·830	3·01	1·60	
	80	·703	4·39	1·145	4·14	1·63	
				Mean.....		1·62	

The greatest errors are comprised within those limits within which our method of measuring densities is only reliable.

The results clearly show that the ratio of densities is given by the light alone, and is not affected by the time of development, a fact quite in accordance with the conclusions previously arrived at.

This ratio, we find, is altogether unalterable. No modifications we have made in developers or in development has

ever seriously disturbed this ratio of the densities. We quote the following few experiments in support of this statement.

*Experiment 14.*—The plate, a "Manchester Slow," having received three different exposures was cut into four portions, two were developed with hydroquinone, and two with eikonogen for different lengths of time. The densities are given exclusive of fog which, with both developers, was very considerable:—

Developer.	Exposure, C.M.S.	Short Develop.		Long Develop.		Ratio $\frac{D_2}{D_1}$	Remarks.
		Density $D_1$ .	Ratio.	Density $D_2$ .	Ratio.		
Hydroquinone.	10	·485	1·0	·785	1·0	1·61	Short time = 2·5 m. Long time = 7·5 m. Mean ratio, 1·55. Greatest error, 4 per cent.
	20	·875	1·80	1·385	1·76	1·58	
	40	1·450	3·0	2·120	2·70	1·47	
Eikonogen.	10	·310	1·0	·580	1·0	1·87	Short time = 4 m. Long time = 12 m. Mean ratio, 1·79. Greatest error, 4·4 per cent.
	20	·560	1·81	·980	1·7	1·75	
	40	·905	2·92	1·600	2·76	1·76	

*Experiment 15* shows that the same result is obtained with pyrogallol development. Plate used: "Manchester Slow." Densities exclusive of fog.

Exposure, C.M.S.	Developed, Three Minutes. Ammonia added at once.		Developed, 18 Minutes. Ammonia added in Six Doses every Three Minutes.		Ratio $\frac{D_2}{D_1}$	Remarks.
	Density $D_1$ .	Ratio.	Density $D_2$ .	Ratio.		
10	·385	1·0	·420	1·0	1·09	Mean Ratio, 1·15.
20	·770	2·0	·850	2·0	1·10	Greatest error, 5 per cent.
40	1·095	2·84	1·315	3·1	1·19	
80	1·455	3·7	1·735	4·0	1·21	

*Experiment 16* is important because it contradicts emphatically the belief that gradations of an over-exposed negative can be altered by using greater amounts of bromide. Plate: "Manchester Slow."

Exposure. C.M.S.	Developed, Four Minutes, 100 cc. = 0.22 NH <sub>4</sub> Br.		Developed, 12 Minutes, 100 cc. = 0.66 g. NH <sub>4</sub> Br.		Ratio $\frac{D_2}{D_1}$	Remarks.
	Density $D_1$ .	Ratio.	Density $D_2$ .	Ratio.		
10	.440	1.0	.485	1.0	1.10	Mean ratio, 1.15.
20	.840	1.91	.955	1.98	1.15	Greatest error, 4.3 per cent.
40	1.200	2.73	1.440	2.97	1.20	
80	1.625	3.70	1.900	3.90	1.16	

*Experiment 17.*—For this experiment a "Wratten Ordinary" plate received four different exposures. It was then cut into four portions, and each portion was developed with a different developer. The result is extremely interesting and important, since it shows that the ratio between the various densities is identically the same whatever developer is employed, except in the case of eikonogen, in which the ratios are a little different. We shall recur to this difference in another place.

#### TIME OF DEVELOPMENT DIFFERENT FOR EACH DEVELOPER.

Exposure.	Ferrous Oxalate.		Pyrogallol.		Hydroquinone.		Eikonogen.	
	Density	Ratio.	Density	Ratio.	Density	Ratio.	Density	Ratio.
C.M.S.								
10	.310	1.0	.320	1.0	.440	1.0	.300	1.0
20	.535	1.7	.550	1.7	.695	1.7	.470	1.6
40	.810	2.6	.805	2.5	1.000	2.4	.645	2.2
80	1.080	3.5	1.005	3.1	1.400	3.4	.820	2.7

These experiments all confirm the statement that the gradations of a negative, as expressed by the ratios of the densities, are independent of time of development, cannot be affected by alterations in the composition of the developers, and are almost identically the same whatever developer is employed. We are thus driven to the conclusion that the photographer has no control over the gradations of the negative, the ratios of the amount of silver deposited on the film being solely dependent upon the exposure. The photographer has the power to increase the total density by prolonged development, but by no means at his disposal can he alter the ratios existing between the amounts of silver reduced in the various parts of the negative; they are regulated entirely by the exposure.

These ratios are not even altered by intensification after development, as is shown by the following results:—

#### INTENSIFICATION.

*Experiment 18.*—Plate "Wratten Ordinary" exposed and developed with ferrous oxalate, measured and afterwards intensified and measured again.

Exposure.	Before Intensification.		After Intensification.		$\frac{D_2}{D_1}$
	Density $D_1$ .	Ratio.	Density $D_2$ .	Ratio.	
10	.31	1.0	.30	1.0	1.33
14	.50	1.61	.91	1.5	1.82
20.5	.67	2.16	1.30	2.16	1.94
29.3	.86	2.77	1.71	2.85	1.98
41.9	1.03	3.32	2.15	3.5	2.08
60	1.30	4.19	2.54	4.2	1.96
					Mean, . . . . 1.95

It will be seen that the process of intensification has almost exactly doubled the amount of silver on the plate. Almost the same result was found in the following experiment:—

*Experiment 19.*—Similar to last experiment.

Exposure.	Before Intensification.		After Intensification.		Ratio $\frac{D_2}{D_1}$
	Density $D_1$ .	Ratio.	Density $D_2$ .	Ratio.	
10	.260	1.0	.475	1.0	1.82
20	.460	1.8	.850	1.8	1.85
40	.700	2.7	1.270	2.6	1.81
80	.950	3.6	1.700	3.6	1.79
					Mean, . . . . 1.82

In this case intensification did not quite, but very nearly, double the amount of silver in each density, but the ratio existing between the several gradations is again not affected at all.

We see therefore, that, whatever may have been the mode of development employed, and whether intensified or not, the ratios of densities are characteristic of the action of the light, and can be alone relied on in investigations respecting the action of the light on the sensitive film.

#### REDUCTION.

There is only one process known to us so far, which will totally alter the ratios existing between the deposits of silver on a negative, viz., the process of reduction, that process consisting in immersing the developed plate into a solution of potassium ferrieyanide and sodium thiosulphate (hyposulphate). This process so alters the ratios that photographers ought to use it very cautiously.

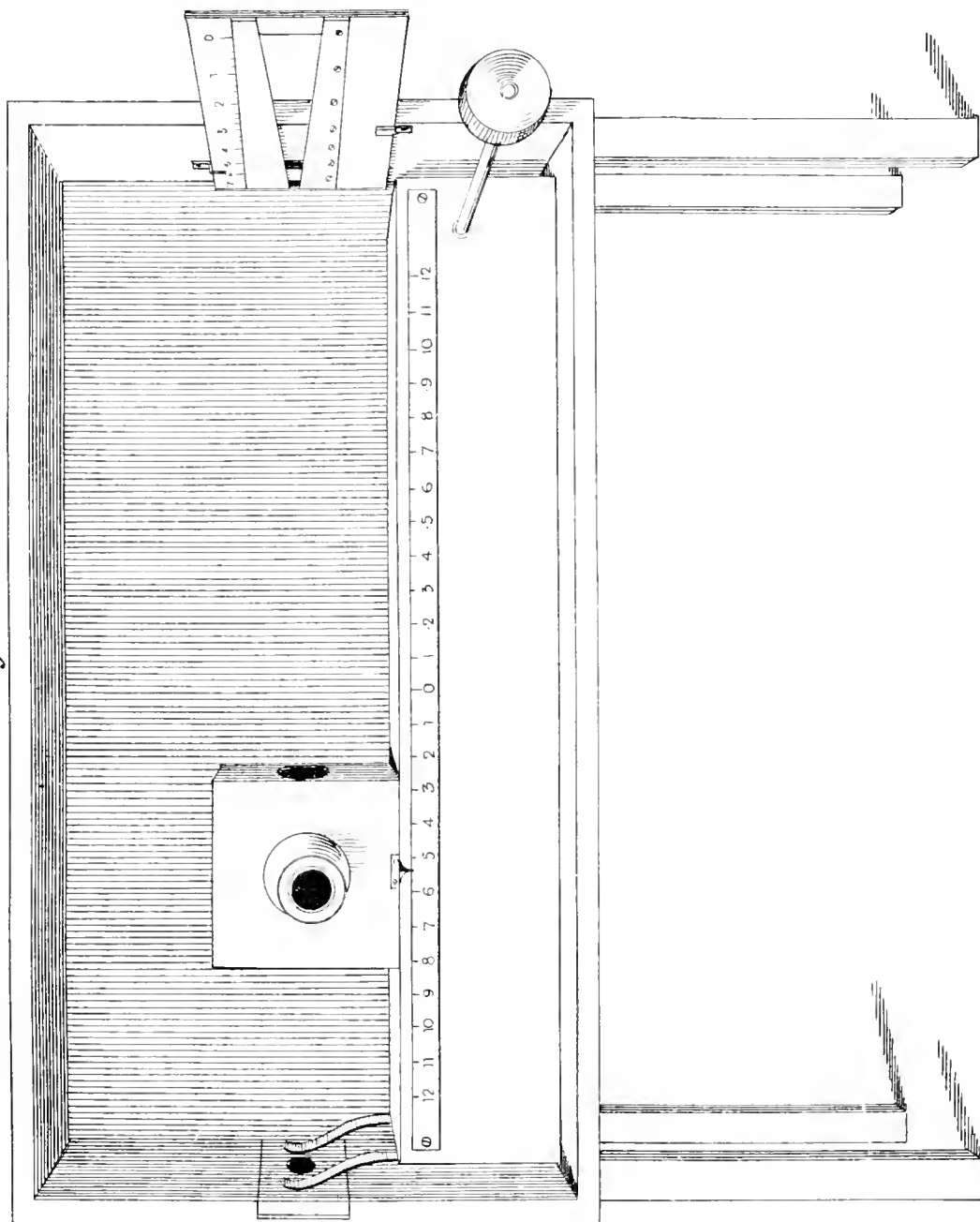
*Experiment 20.*—Plate exposed, measured, and reduced by immersion in potassium ferrieyanide and sodium thiosulphate.

Exposure.	Before Reduction.		After Reduction.		Ratio $\frac{D_1}{D_2}$
	Density $D_1$ .	Ratio.	Density $D_2$ .	Ratio.	
10	.410	1.0	.620	1.0	20.5
20	.655	1.6	.130	6.5	5.0
40	1.010	2.46	.365	18.2	2.7
80	1.450	3.5	.680	34.0	2.1

#### V.—ACTION OF LIGHT ON SENSITIVE FILM.

Our investigations have not only revealed the fact that one single density taken by itself is not characteristic of the exposure which the sensitive film received, since the density may be partially due to "fog," or may not be developed to its extreme limit, but the experiments have

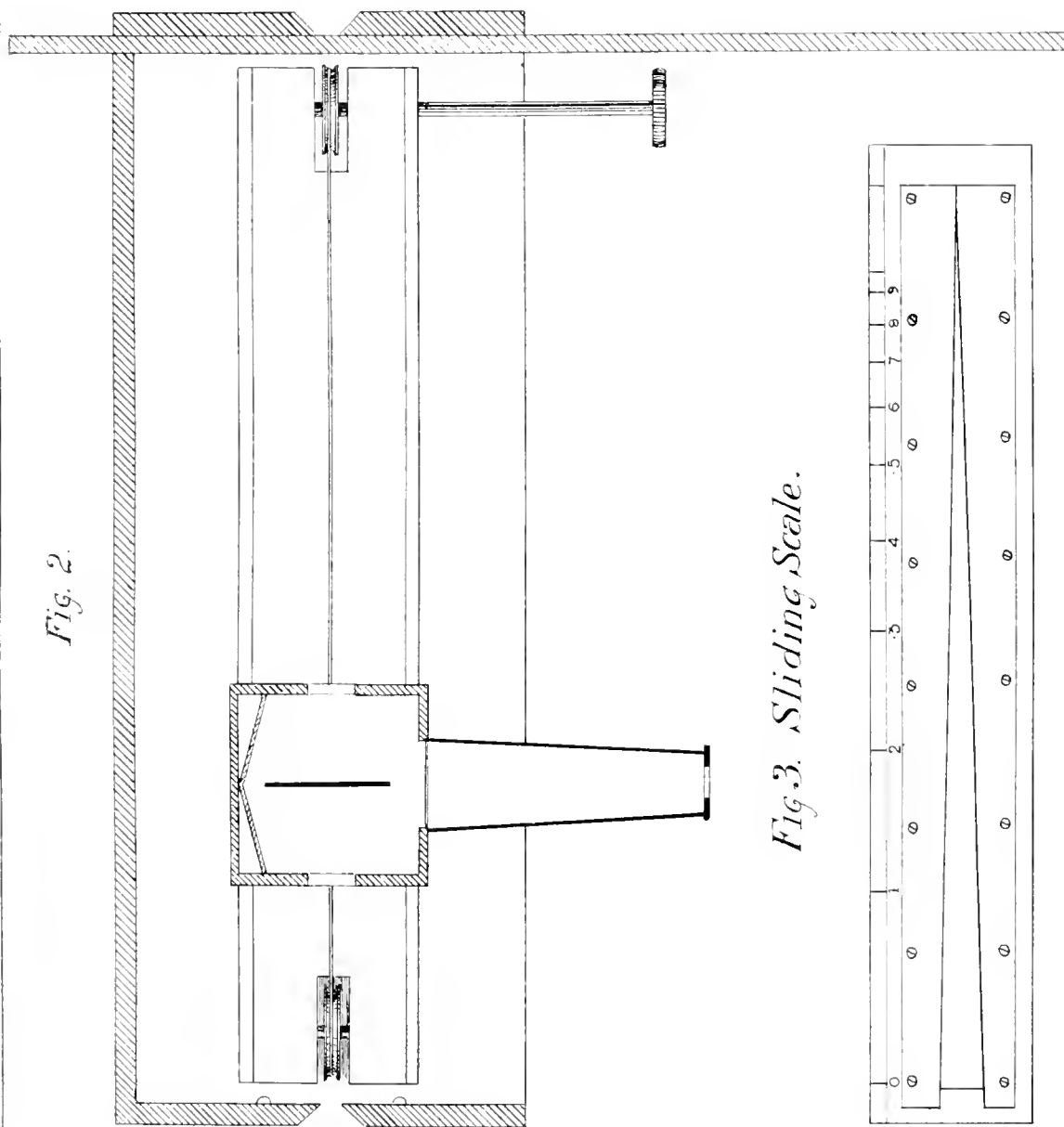
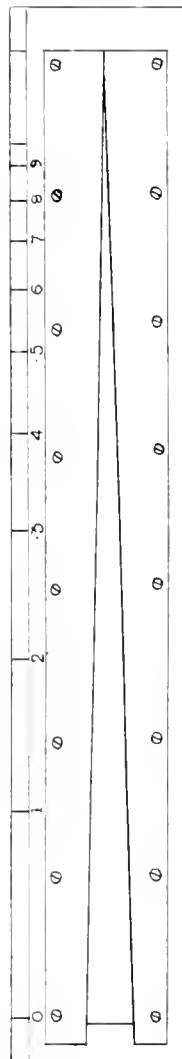
Fig. 1



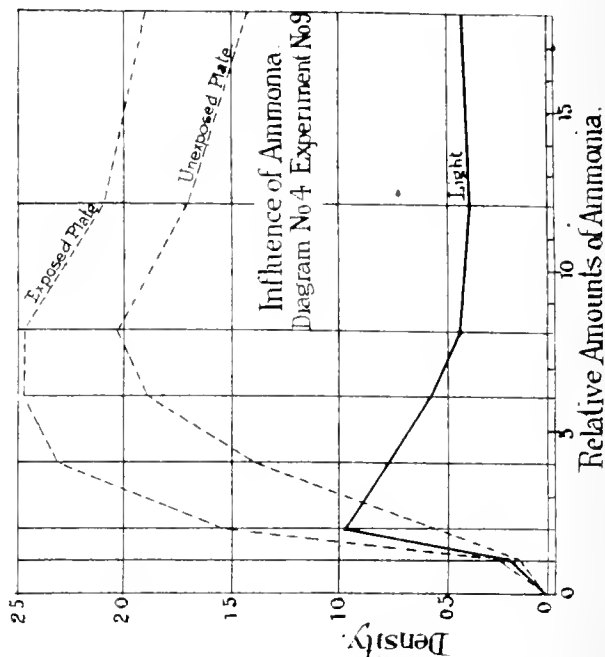
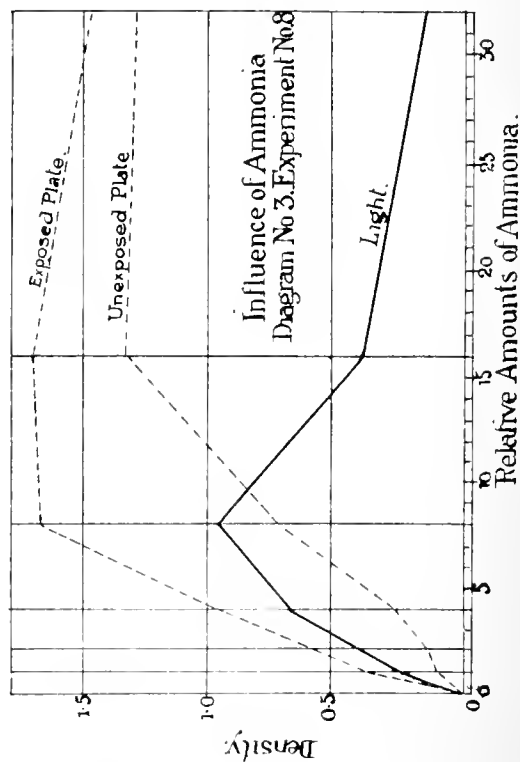
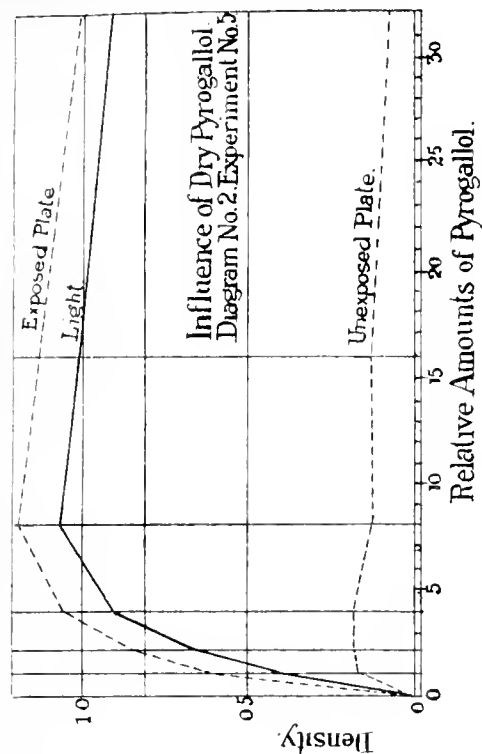
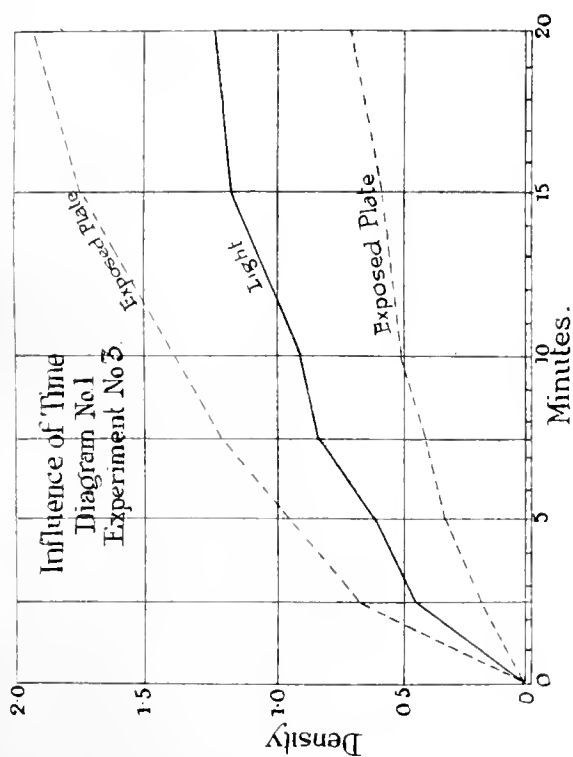
*Hurter & Driffield's Apparatus for Measuring Density of Negatives.*



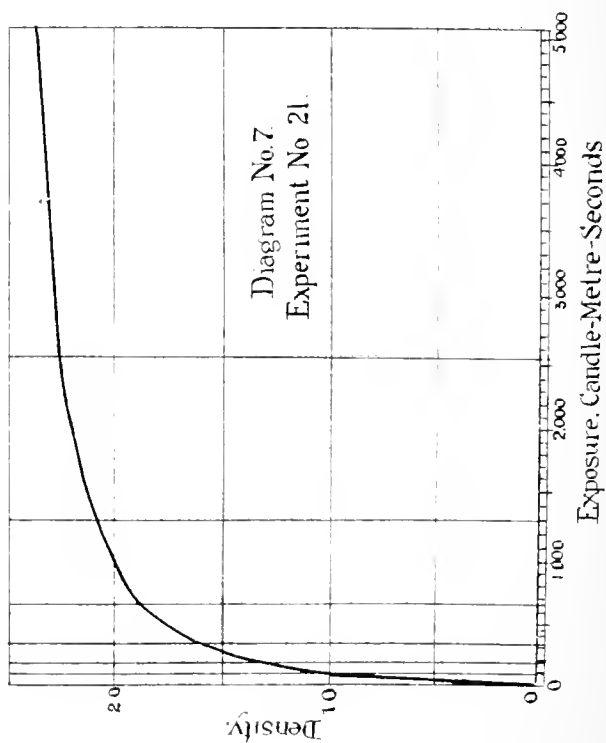
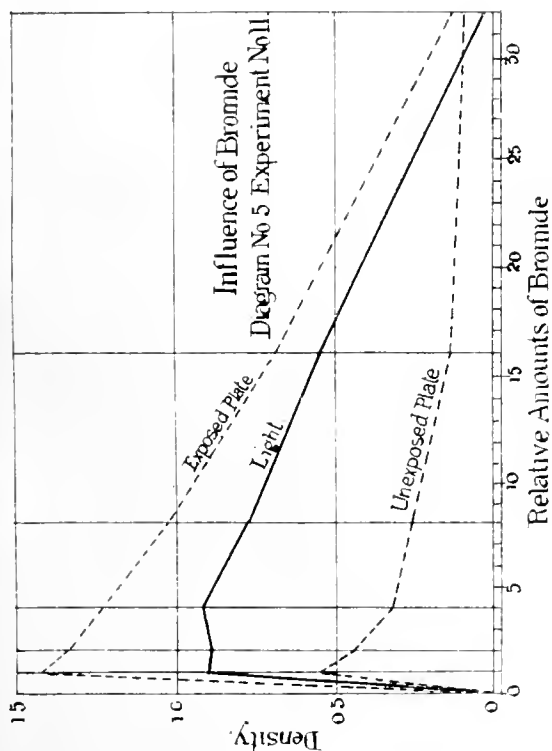
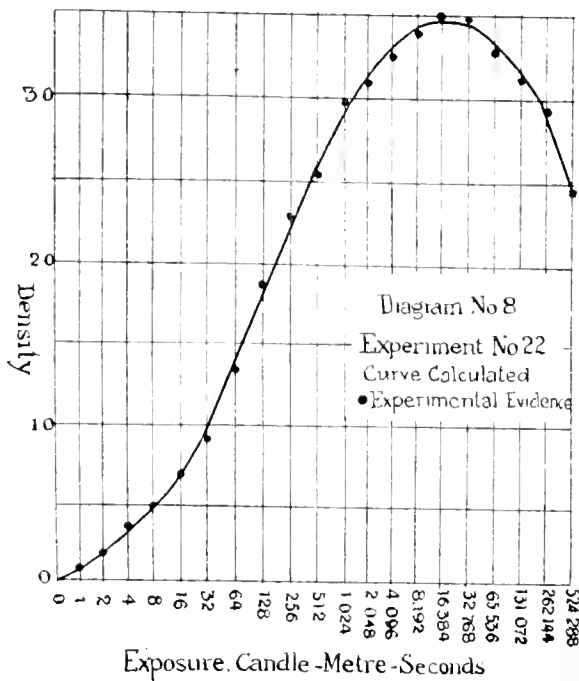
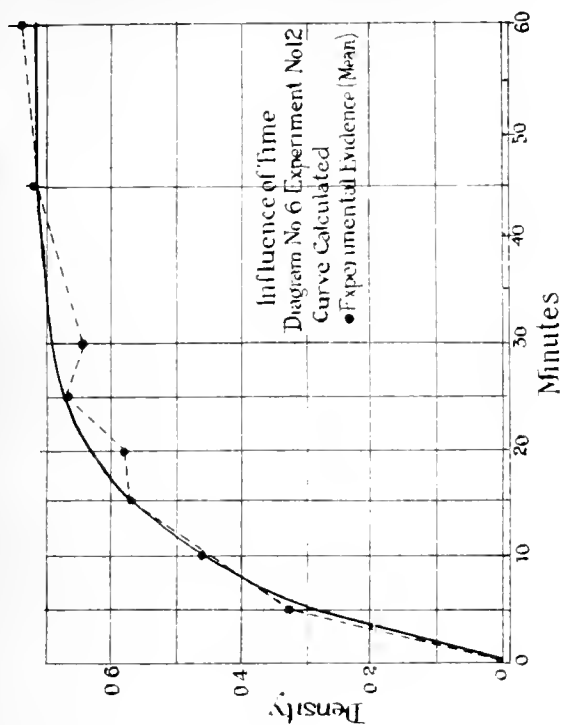


*Fig. 2.**Fig. 3. Sliding Scale.*



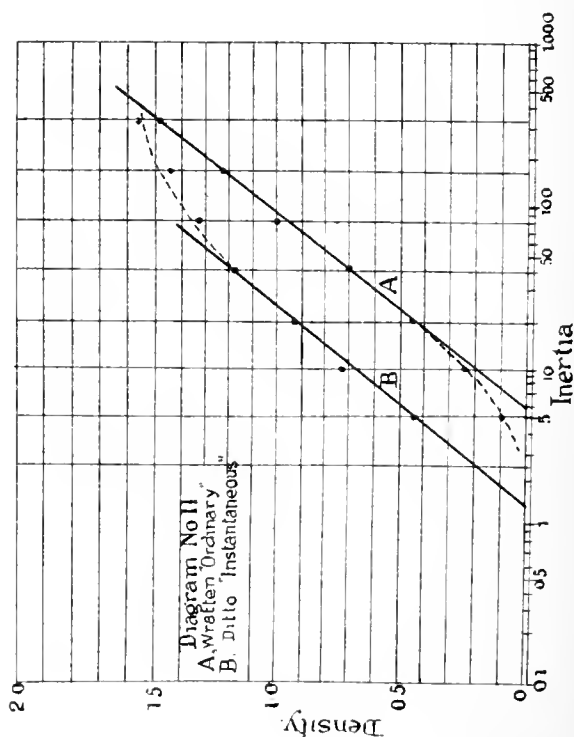
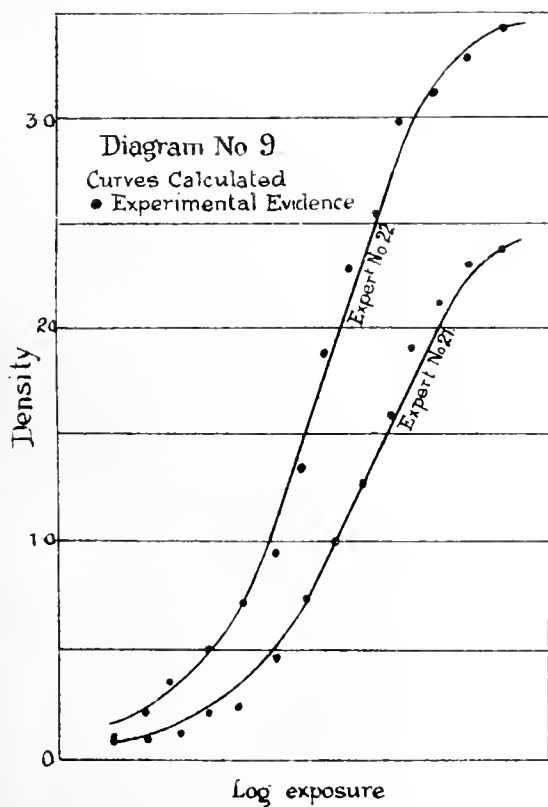
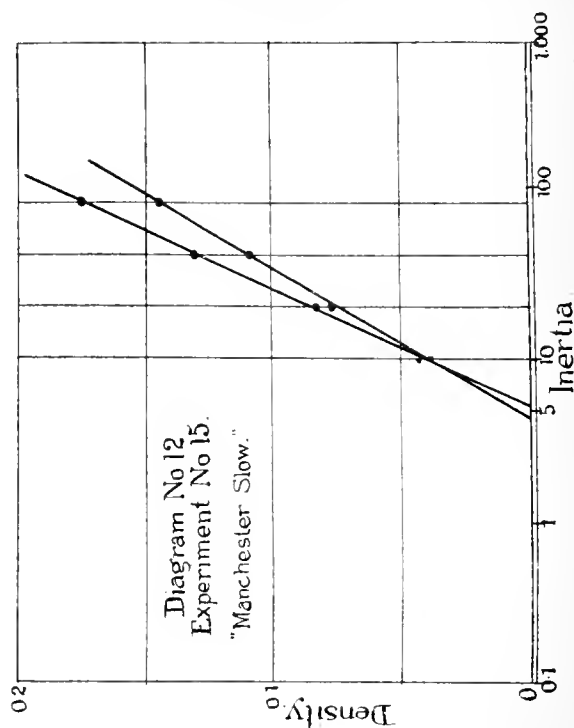
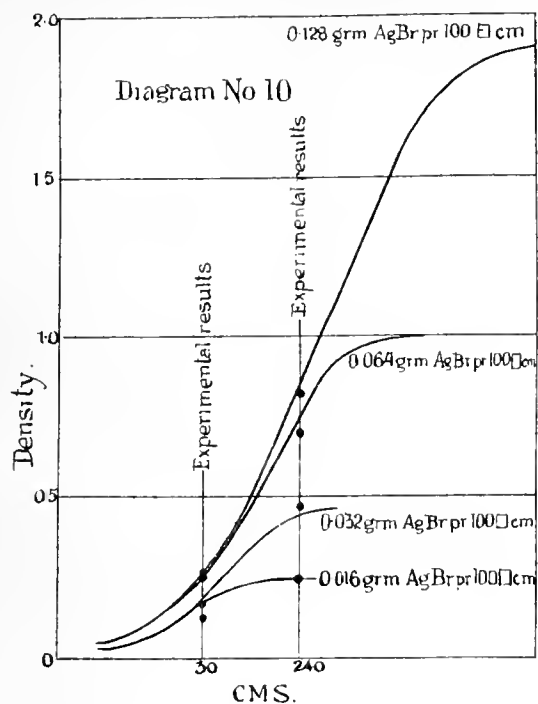




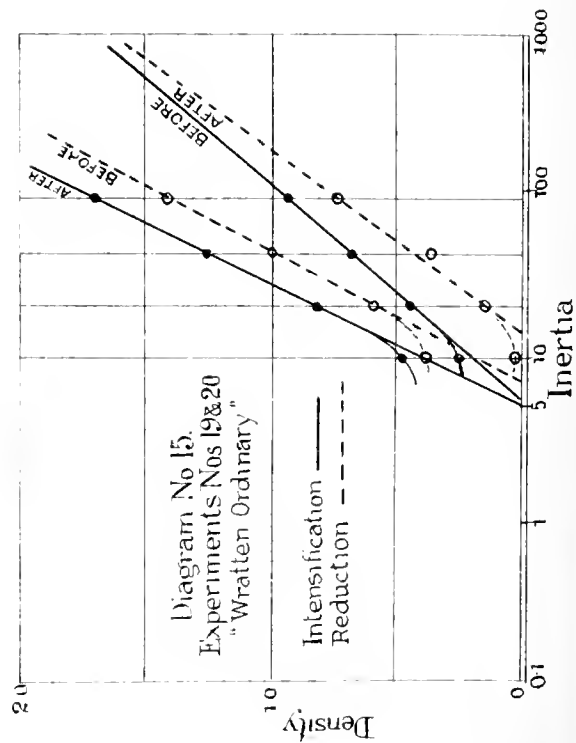
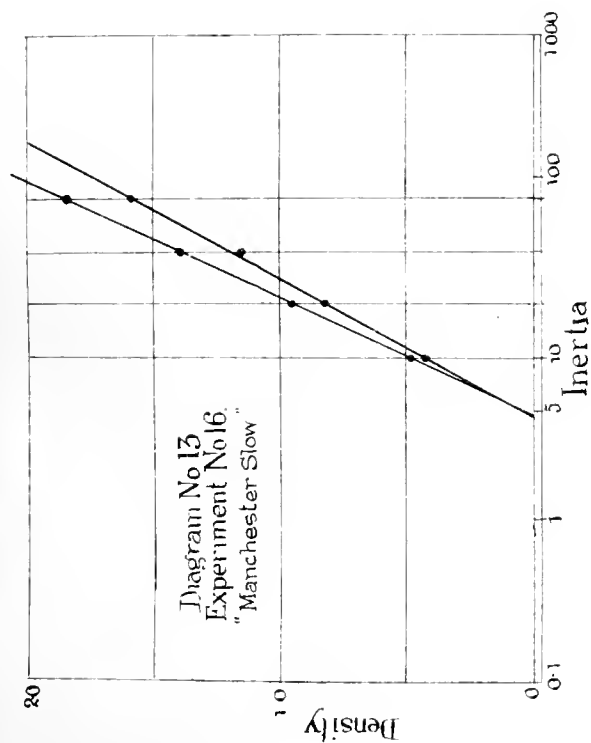
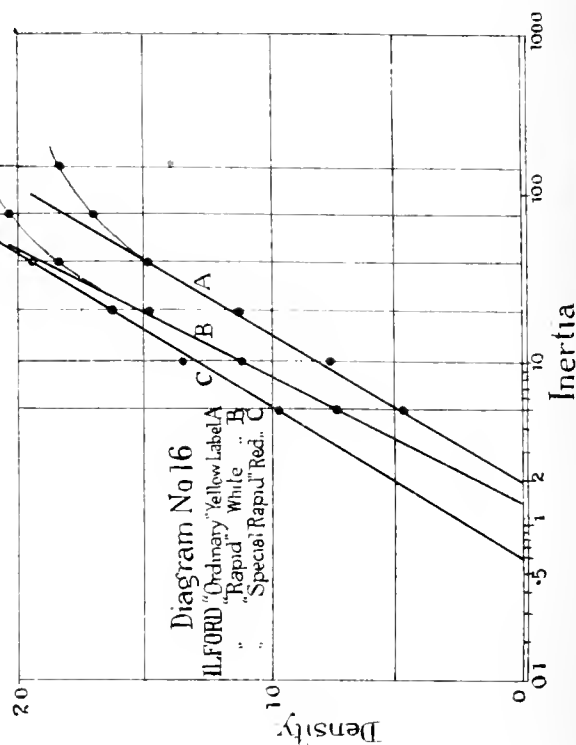
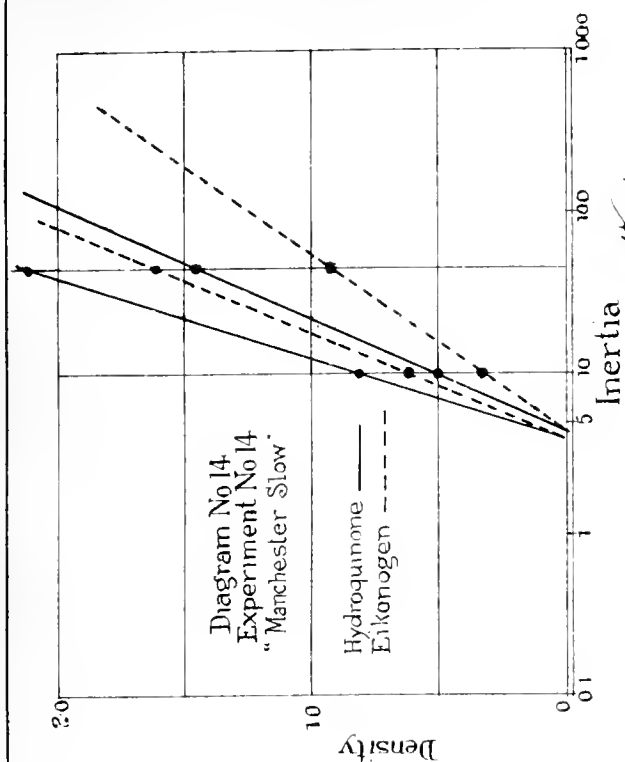


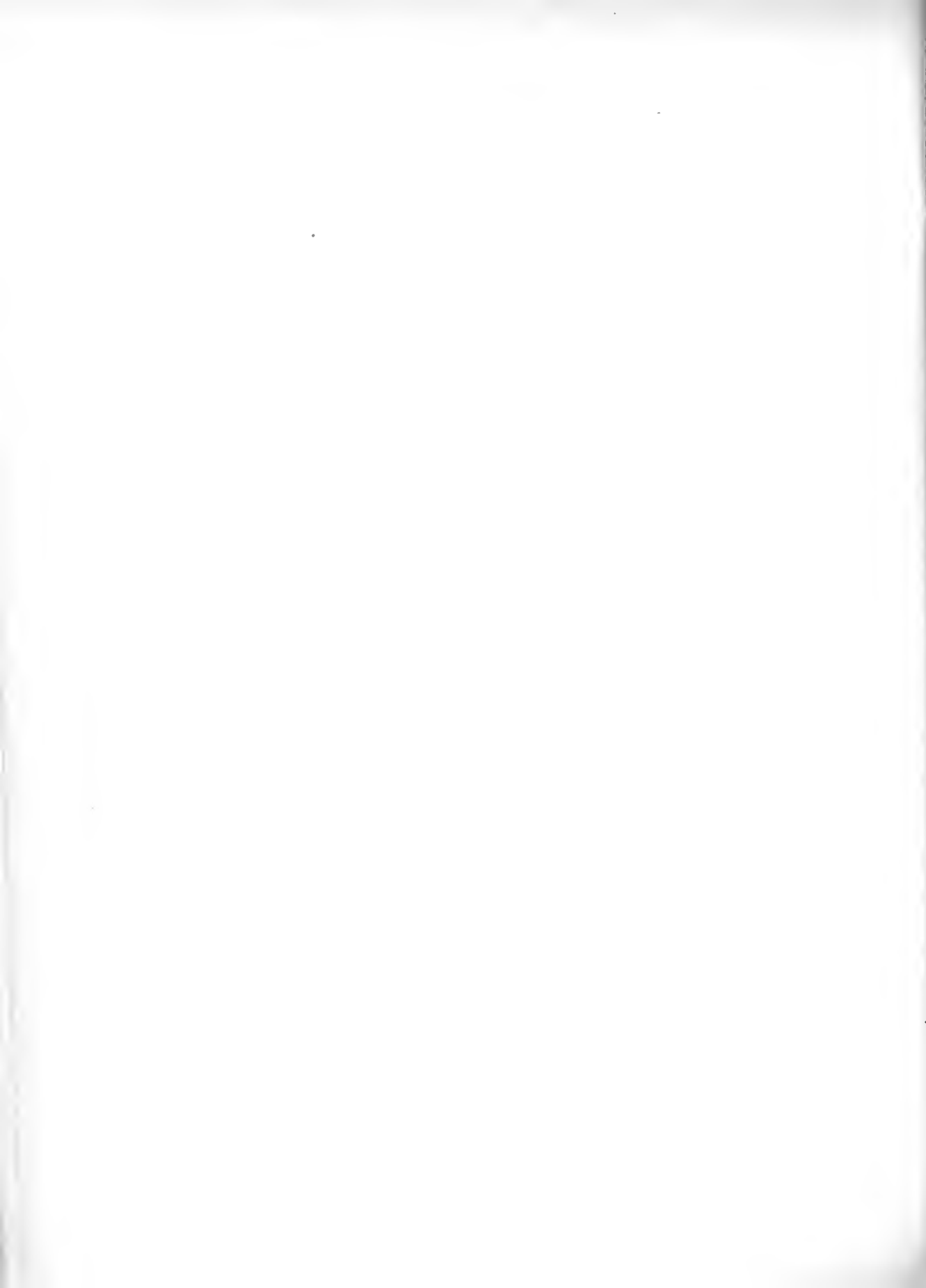












also clearly shown that the ratio of two densities, exclusive of fog, is a function of the action of the light on the plate. It will be noticed that in all these experiments the exposures given varied between 10 seconds and 80 seconds, and the source of light was always a standard candle placed exactly one metre off the plate. If we tabulate the ratios found between the 10 seconds and 80 seconds exposures in these experiments we see at once that the ratio, though constant for one particular plate, is very different for different plates.

Name of Plate.	Ratios of Densities for		Experiment No.
	Exposures 10'' = 4.	Exposures 80'' = 8.	
Hford "Red Label" .....	1.53	1.63	13
Wratten "Dropshutter" ..	1.71	1.92	13
"United Kingdom" .....	3.12	4.27	13
"Manchester Slow" .....	2.97	3.82	15
Do. Do. ....	2.85	3.80	16
Do. Do. ....	2.84	..	14

Own make .....	Batch A. ....	Exposures	160'' .....	8.00
	" A. ....	"	20'' .....	"
	" A. ....	"	240'' .....	5.30
	" A. ....	"	360'' .....	"
			1440'' .....	3.00
			180'' .....	"

The ratio is for the same exposures, smaller for rapid plates than for slow plates, but even with the same plate, the ratio between two densities varies for exposures which bear the same ratio to each other, but are different in absolute value as is seen from the experiments given in the above table, and made with plates prepared by ourselves.

It is certain, therefore, that the ratio between two densities depends not only on the ratio of the exposures, but also on the sensitiveness of the plate and the absolute values of the exposures. The following investigations were made to discover the connexion existing between exposure, sensitiveness, and density produced.

**Unit of Exposure.**—For these investigations it was necessary to adopt a standard unit of exposure. As unit of light we have chosen the intensity of a standard candle at one meter distance, and as unit of time the second, so that our unit of exposure is the product of the intensity of the standard candle at one meter distance and the second, and we call this unit of exposure one "candlemeter-second." We find for experimental purposes, with plates of average speed it is an excellent unit, easily procured, and of sufficient constancy to permit of satisfactory repetitions of experiments. There are a few precautions necessary to ensure uniform results. The flame of the candle cannot be relied on until it has settled to a height of nearly 45 mm. measured from the top of the spermaceti to the top of the flame, as shown in Fig. 4. The candle must be protected against draughts, and this is best done by placing it within a black box having one side open. This also prevents the illumination of bright objects on the working table and consequent reflections. The candle should be extinguished by an extinguisher and kept covered up while not in use.

As regards measuring time of exposures we use a chronograph watch, or a metronome for short exposures, but we find that errors of exposure become too great if less than 10 seconds are measured. If we wish to give shorter exposures than 10 candlemeter-seconds (C.M.S.) we place the standard candle two meters off, thus reducing its intensity to one quarter.

It is scarcely necessary to say that we have carefully ascertained that within such limits of exposures as our experiments embrace it is immaterial whether an exposure be made with a light of  $\frac{1}{4}$  candlemeter for 40 seconds or a light of one candlemeter for 10 seconds. We have also

proved by experiment that, as far as the ratios of densities are concerned, they remain constant, whether the exposure be made with a candle, with a petroleum lamp or with daylight, so long as the product of intensity of light and time of exposure be the same. The intensities of such different sources of light cannot however for this purpose be compared by the ordinary Bunsen photometer, but must be compared by photographic experiments. But with careful work even single densities can be reproduced with tolerable accuracy. For instance, on three separate days we obtained on three separate plates of the same batch by carefully measuring the time, both of exposure and of development, densities 0.750, 0.730, and 0.720 respectively. Four different standard candles gave upon one plate in 10 seconds the following densities: 0.490, 0.490, 0.500 0.480.

With the standard candle we investigated, in the first place, the general effect of prolonged exposure on the density, *i.e.*, we ascertained how much silver was reduced by different exposures.

**Experiment 21.**—A "Manchester Slow" plate exposed, developed with ferrous oxalate and measured, gave:—

Exposure C.M.S.	Density.	Difference.	Exposure C.M.S.	Density.	Difference.
0.625	.045	..	80	1.010	.255
1.25	.055	0.010	160	1.270	.260
2.50	.085	0.030	320	1.555	.285
5.00	.175	.090	640	1.885	.330
10.0	.250	.075	1280	2.088	.203
20	.460	.210	2560	2.262	.174
40	.755	.295	5120	2.352	.090

It will be seen that, every time the exposure is doubled, the density increases, at first slowly, then considerably, and (disregarding errors of experiment) from 40 C.M.S. up to 1280 C.M.S. every time the exposure is doubled, nearly an equal addition to density is the result, the addition to the density being on an average 0.266, but after an exposure of 1280 C.M.S. further doubling produces less and less increase in density. The first few densities are too small to admit of accurate measuring.

This series of results is represented graphically on diagram No. 7, the exposures being chosen as abscissae, the densities as ordinates; from this diagram it will be seen at once how rapidly densities grow at first as exposure is increased, and how slowly at last the densities tend towards a limit.

The following series of exposures is carried still further in order to ascertain the character of the curve representing the action of the light on the silver bromide, and to learn, if possible, something of the limit towards which the curve tends.

**Experiment 22.**—

Exposures C.M.S.	Densities.	Difference.	Exposures C.M.S.	Densities.	Difference.
1	.060		1024	2.085	+ 450
2	.160	+ 100	2048	3.115	+ 130
4	.340	+ 180	4096	3.280	+ 165
8	.500	+ 160	8192	3.405	+ 125
16	.715	+ 215	16384	3.508	+ 103
32	.910	+ 225	32768	3.471	- 0.034
64	1.345	+ 405	65536	3.280	- 0.194
128	1.875	+ 530	131072	3.128	- 0.162
256	2.200	+ 415	262144	2.920	- 0.208
512	2.535	+ 245	524288	2.444	- 0.456

This series of results could not be graphically represented to advantage by choosing exposures as abscissæ since they vary from one candlemeter-second to over half a million. But it is evident that prolonged exposure gradually reduces the density attainable after development.

The graphic representation of experiment 22 on the same scale as experiment 21 would require a diagram about 500 times as long as diagram No. 7, and nothing of any value would be learnt from such a diagram.

What we really wish to ascertain is whether it is possible to produce a theoretically perfect negative, such as was defined, and what the connexion is between the densities and the exposures.

If in any part of the curve of densities, as represented in diagram No. 7, the densities were proportional to the logarithm of the exposures, we should discover that portion of the curve if, instead of choosing the exposures as abscissæ, we used the logarithms of the exposures as abscissæ. This is easily done when the exposures progress, as they always do in our experiments, in a geometric series. We have only to mark every new exposure equi-distant from the previous one as abscissæ. In this manner the results of experiment 22 are plotted in diagram No. 8.

It will be perceived that the curve now consists of four distinct branches. It proceeds from exposure 1 in almost horizontal direction, ascends slowly to exposure 16, from thence it proceeds almost in a straight line to exposure 2048, when the growth of densities becomes slow. The densities reach their maximum at exposure 16,384, and from thence the curve returns, the densities diminishing slowly with increased exposures.

We accordingly distinguish four different periods of exposures. The first period we term the period of "under exposure;" it is comprised in the first curved portion. The second period, that during which the curve is almost a straight line, we call the "period of correct representation." The third period is that during which the curve is again strongly bent as far as its maximum; this is the period of "over-exposure," and the last portion of the curve we term the period of "reversal."

1. *Period of Under-exposure.*—During this period the ratios between two densities are at first accurately equal to the ratio of the corresponding exposures. It is very difficult to study this portion of the curve accurately, owing to the short exposures which it demands, but still more owing to the small densities which this period yields, and which are difficult to measure. By making very slow emulsions, we have, however, succeeded in showing clearly that at first the amount of silver reduced is proportional simply to the exposure. Thus, a plate made by ourselves, with pure bromide of silver, gave the following results:—

Exposure, 20 C.M.S.	Density, .125 or 1.
" 160 "	" 1.055 or 8.4.

The ratio between the densities being very nearly 8, the ratio of exposures.

Again, a "United Kingdom" plate gave the following results:—

Exposure, 2.5 C.M.S.	0.160.	Ratio, 1.
5.0 C.M.S.	0.313.	1.95.

A very slow "Manchester" plate gave the following results:—

1 C.M.S.	0.260.	Ratio, 1.
2 C.M.S.	0.550.	2.1.

But of course there is no definite point which marks the end of this period and the beginning of the next. But it is from this period that we learn that, for short exposures, the amount of silver reduced is directly proportional to the exposure.

*Period of Correct Representation.*—The second period of exposures we have thus named because during this period a plate is capable of giving a negative differing as little as possible from that which, at the beginning, we defined as theoretically perfect. That definition demanded that the densities of the negative should be proportional to the logarithms of the exposures which produced them. It is

characteristic of this period that the densities are proportional to the logarithms of the exposures. This is shown on diagram No. 8, where the densities are the ordinates, the logarithms of exposures are abscissæ, and the period of correct representation a straight line. We have measured densities of dozens of plates falling within this period, and we find them all to conform to this very simple linear equation—

$$D = \gamma [\log. I.t \pm C],$$

D being the density,  $\gamma$  a constant depending on time of development,  $I.t$  the product of intensity of light and time, i.e., the "exposure," and  $C$  a constant depending upon the speed of the plate. As we shall give further detailed proof of this, we will here merely insert one example of a plate, the constant  $C$  of which is zero, it is Plate 2 of experiment No. 13. That plate gives the following results with equation:—

$$D = 1.75 \times \log. \text{exposure.}$$

Exposure.	Density found by Experiment.	Density Calculated.
10	1.74	1.75
20	2.37	2.27
40	2.91	2.75
80	3.33	3.30

Many similar examples will be quoted presently.

We have thus arrived at the answer to the question, can negatives be produced such as we defined to be theoretically perfect? And this answer is, they *can* be produced, but only by so carefully adjusting the time to the intensity of the light, that the exposures may fall within that period of correct representation. No variations whatever in development will correct an under- or over-exposure.

*Period of Over-exposure.*—Little need be said about this period. As the curve tends to become parallel to the axis of abscissæ it is clear that when exposures fall within this period, shadows and high lights will all be represented by densities which are almost equal. There will be no contrasts. In the first period of under-exposure the contrasts are too great; here they are too small.

*Period of Reversal.*—This period we have named the period of reversal because within this period happens that peculiar phenomenon, the transformation of the negative into the positive, the "solarisation," "reversal," &c. It is easy to understand how the negative becomes a positive. Whilst the deep shadows still act upon the plate increasing the density the high lights have passed their maximum and their densities grow less and less. The more the exposure is prolonged the less dense the high lights become, the shadows exceeding them in density. It is quite easy to observe this phenomenon of reversal with a powerful petroleum lamp or gas burner, or to produce by direct contact printing a secondary negative, instead of a positive, from the original negative, by about 15 to 20 minutes exposure at 6 in. distance from the light. When, in the camera, exposure is prolonged, it is well known that a positive is obtained instead of a negative. It has been stated by Jansen that a secondary negative and a secondary positive, can be obtained by prolonging the exposure still further. We have not, however, been able to verify this statement, and we believe it to be erroneous. Our investigations show that the density tends to a limit, and a picture produced by prolonged exposure in the camera is gradually lost in a uniform veil of fog, though it is still visible even after a three days' exposure.

A "United Kingdom" plate received various prolonged exposures, with the following results:—

		Difference.
75,000 C.M.S. gave density	1.415	
150,000 " "	1.111	.304
300,000 " "	.970	.141
600,000 " "	.925	.045

A piece of the same plate exposed to direct daylight for 90 minutes (about six million C.M.S.) gave a density 1.200. From this it appears to us that there is an equilibrium established between the action of the refrangible and less refrangible rays.

The period of reversal is, theoretically, exceedingly interesting, and deserves further careful study, but the reversing action is so slow, and requires such enormous exposures, that it does not need to be considered from a practical point of view, and we shall disregard it entirely for the present. The three first periods, that of under-exposure, that of correct representation, and that of over-exposure are the only practically interesting portions of the curve. We have already stated that, during the first period, the ratio of densities is equal to the ratio of exposures, *i.e.*, the amount of bromide of silver reduced is proportional to the exposure, whilst, during the second period, the density only grows in proportion to the logarithm of the exposure. It almost ceases to grow during the third. Of course, these assertions are only two approximate statements of one single law connecting the densities with the exposures.

All photo-chemical investigations which have hitherto been made have proved that the amount of chemical action is proportional to the "exposure" (*i.e.*, the product of intensity of light and time). The sensitive film of the photographic plate forms no exception to this general law, and we take it as a fundamental truth that the amount of action upon the plate is, at any moment of the exposure, proportional to the energy which the plate receives at that moment. During the first period, when the surface, or chiefly the surface of the film, is acted upon, the results of the investigations have shown this to be true accurately. But when the action of the light upon particles of bromide of silver below the surface has to be considered, the question arises, how much of the light which impinges on the surface really reaches those particles?

Of the rays of light which impinge upon the surface of the sensitive plate, some are reflected and some pass right through the plate. If one sensitive plate be exposed to light behind another, it will be found that it also is affected.

The energy of the reflected and transmitted light cannot, obviously, play any part in the molecular work to be done in the film. It is useless photographically.

The light absorbed in the film is the only light which contributes towards the formation of the "latent image," but not even the whole of the light which is absorbed does useful work. It can be proved experimentally that a plate which has received such an exposure as to yield maximum density on development, absorbs exactly as much light as a plate which has not been exposed at all, yet the light absorbed by a plate already so exposed obviously contributes nothing towards increase of density.

From this it is clear that the light absorbed by a particle of silver bromide, which has already received sufficient energy to bring it into that condition in which it is capable of development, is useless.

It will, therefore, be evident that, of the light impinging upon the plate, there is only one portion useful, *viz.*, that which is absorbed by unaltered silver bromide, the light reflected, the light transmitted, and the light absorbed by particles of silver bromide already changed, being altogether useless.

The amount of work done at any moment of the exposure is therefore proportional to the amount of energy received by the unaltered silver bromide only.

It is very easy to state this proposition mathematically, and thus find the law which connects the densities with the exposures.

If the intensity of the light (with respect to chemically active rays) is  $I$ , and the fraction of the light reflected from the surface of the film is  $\alpha$ , then the amount  $(1-\alpha) I$  enters the film. If the film contains, at the moment we are considering,  $x$  particles of silver bromide per unit area, which are already changed, then the transparency of the plate with respect to the changed particles is  $\epsilon^{-kx}$ , *i.e.*, this is the amount of light which passes the particles already changed. If from this amount we deduct the amount of light which passes *all* the particles of silver, changed or unchanged, the difference represents the amount of light

absorbed by the silver bromide not yet affected. Now the light which passes all the particles of silver, if there are  $a$  of them per unit area, will be measured by the transparency of the plate *viz.*,  $\epsilon^{-ka}$ . Deducting this from  $\epsilon^{-kx}$  and multiplying the difference with the total amount of light entering the film will give the mathematical expression for the amount of light which, at the moment we are considering, can do useful work; this amount is:  $(1-\alpha) I (\epsilon^{-kx} - \epsilon^{-ka})$ . If this expression is multiplied by the short time of exposure  $dt$ , it will represent the amount of useful energy conveyed to the plate during that time.

Suppose it requires an amount of energy  $e$  to change one particle of silver bromide into the condition capable of development, then the number of particles  $dx$  so changed during the time  $dt$  will be

$$(1.) \quad dx = \frac{1}{e} (1-\alpha) [\epsilon^{-kx} - \epsilon^{-ka}] dt.$$

This is the complete mathematical expression of the idea that it is only that portion of the light which is absorbed by unchanged silver bromide, which contributes to the growth of density.

By integration of equation (1), and by substitution of the symbol  $O$  for  $\epsilon^{-ka}$ , we find that the density of the "latent image" (before development) is

$$D = \log_e [O - (O-1) \beta^{k(1-\alpha)} \frac{It}{e}]$$

where  $\beta$  is a fraction, the hyperbolic logarithm of which is  $-\frac{1}{O}$ .  $O$  is simply the opacity of the plate to the chemically active rays before exposure.

In this derivation of the connection between the density  $D$  and the exposure  $It$ , two assumptions have been silently made which need explanation. The coefficient of absorption,  $k$ , has been assumed to have the same value both for the altered and the unaltered silver bromide. We have, however, experimentally ascertained that this is a fact. It can be easily proved photographically. If, behind a plate, one portion of which has been already exposed so as to yield maximum density, the other portion having received no exposure at all, a very sensitive plate is placed, and if now a suitable exposure be given, it will, on development, be found that the shielded plate has uniform density all over. This proves that  $k$  is the same as regards blue light both for the altered and for the unaltered silver bromide.

The second assumption is that the sensitive film obeys the laws of absorption, as explained at the beginning of this paper. It would prolong this paper very much if we had to furnish here the proof that, as far as the chemically active rays are concerned, and as far as the light *not reflected* is concerned, the law of absorption does hold good. Suffice it to state that, to the more refrangible portion of the spectrum, the sensitive film is as black as Indian ink is to white light.

To recur to our formula, it requires still more alteration to complete it. The density as given by the formula is the maximum density, and expressed as regards the behaviour of white altered silver bromide towards the blue rays of the spectrum. As we know already, we can develop of that maximum density as much as we please, and the change from white to black during development makes the density more or less equal for all rays of the spectrum. We therefore simply multiply the equation by a constant to express this change, and we call this the development constant. The formula then stands—

$$D = \gamma \log_e [O - (O-1) \beta^{k(1-\alpha)} \frac{It}{e}]$$

$k$ ,  $\alpha$ , and  $e$  represent physical and chemical properties of the bromide or silver, which together constitute its sensitiveness to light. We combine them into one single symbol, and write  $i = \frac{e}{k(1-\alpha)}$ , so that we have finally—

$$(2.) \quad D = \gamma \log_e [O - (O-1) \beta^i \frac{It}{e}].$$

This formula represents the density after development as a function of the opacity of the unexposed plate, of the



exposure, and of the symbol  $i$ , which is a measure of the slowness of the silver bromide, and which symbol we shall call the "inertia" of the silver bromide.

To show the approximation of densities calculated by this formula to those obtained in experiments 21 and 22, we append here the calculated and the observed densities. For this purpose the plates used for experiments No. 21 and 22 were investigated for their opacity to the rays of the spectrum from F to H, and this opacity was found to be 332.

*Experiment 21 compared with theory.*

Exposure. C.M.S.	Density Found.	Density Calculated.	Exposure. C.M.S.	Density Found.	Density Calculated.
0.625	.045	.035	80	1.010	.992
1.25	.055	.065	160	1.270	1.272
2.5	.085	.121	320	1.555	1.531
5	.175	.214	640	1.885	1.780
10	.250	.339	1,280	2.088	2.022
20	.460	.520	2,560	2.262	2.218
40	.755	.743	5,120	2.352	2.352

*Experiment 22 compared with theory.*

Exposure. C.M.S.	Density Found.	Density Calculated.	Exposure. C.M.S.	Density Found.	Density Calculated.
1	.060	.092	128	1.875	1.800
2	.160	.172	256	2.200	2.165
4	.340	.302	512	2.535	2.518
8	.590	.482	1,024	2.985	2.860
16	.715	.735	2,048	3.115	3.138
32	.940	1.059	4,096	3.280	3.328
64	1.345	1.405	8,192	3.405	3.405

On examining the "calculated" series of results it will be found that they have exactly the same characteristic properties as those we pointed out as appertaining to the three periods. For the short exposures the calculated densities are nearly proportional to the exposures, whilst from 16 C.M.S. to 1200 C.M.S. the densities increase by nearly equal amounts for every successive double exposure, and differ very little from densities calculated by the simple formula—

$$D = \gamma [\log. It - C].$$

In order that this may be very clearly seen we append another table comparing in column 1 the densities obtained by the correct formula (2), with densities in column 2 calculated by the approximate formula—

$$D = 1.176 [\log. It - 0.579].$$

Exposure, C.M.S.	(1.) Density by Correct Formula.	(2.) Density by Approximation.	Exposure, C.M.S.	(1.) Density by Correct Formula.	(2.) Density by Approximation.
16	.735	.735	256	2.165	2.151
32	1.050	1.089	512	2.518	2.505
64	1.405	1.443	1024	2.860	2.859
128	1.800	1.797	2048	3.138	3.213

We think it necessary to draw attention to this agreement, because the approximate formula is extremely easily applied, whilst the correct formula requires very tedious calculations, and we shall make a very important practical application of such calculations.

Although there is a close general agreement between the experimental results and the numbers calculated by means of the equations, yet in individual cases there are discrepancies. Diagram No. 9 shows the theoretical curves in full lines, the actual observations being indicated by dots. This diagram leaves little doubt that the action of the light on the sensitive film is fairly represented by our equation, and consequently it may be assumed as proved that the action of the light at any moment is proportional to the amount of light absorbed by unaltered silver bromide.

Nevertheless we felt that more experimental proof was wanted to support our equation, which represents the resulting density as a function of the opacity of the unexposed plate to blue light. When it is remembered that the density of the unexposed plate is proportional to the silver bromide spread over its area, it will be perceived that this statement means, in fact, that the density of the image depends upon the amount of silver on the plate in some way, and this is almost a self-evident proposition.

We prepared sensitive plates of different opacities by spreading on equal areas different amounts of silver bromide. These plates were measured to ascertain their opacity to blue light, and the following results obtained:—

Plates No. 1.	Amount of Silver Bromide per 100 Sq. cm.	Opacity to Blue Light.
1	Grms. 0.016	1.738
2	0.031	3.09
3	0.062	10.0
4	0.124	83.0

By means of these opacities four curves were calculated, which are represented in diagram No. 10. To ascertain whether the relative distances between those curves were true, points in each curve belonging to the same exposure (or abscissa) had to be determined in at least two different portions. For this purpose four plates, one of each opacity, were simultaneously exposed, and two different exposures were given of 30 and 240 C.M.S. respectively. The plates were then developed and the ratios of the densities were taken alone for comparison.

The following results were thus obtained:—

Exposure.	Plate I.	Plate II.	Plate III.	Plate IV.
	Density.	Density.	Density.	Density.
30 C.M.S. ....	.065	.095	.260	.272
240 C.M.S. ....	.120	.275	.700	.852
Ratio .....	1.84	2.83	2.69	3.31

The ratios which are obtained for the theoretical densities, calculated by the equation (2), are the following ones:—

—	Plate 1.	Plate 2.	Plate 3.	Plate 4.
Ratio .....	1.70	2.59	2.83	3.22

It will be seen that the theoretical ratios agree as well with the observed ratios as could possibly be expected from so difficult an experiment. In diagram No. 10 the relative distances of the curves, as calculated from the ratios obtained by the experiment, are marked by dots.

Diagram No. 10 is worthy of some remarks. It will be at once perceived that the more thinly the plates are coated the shorter is that portion of the curve which is a straight line. This means that the period of correct representation is very short, and great contrasts cannot be truly rendered by a thinly-coated plate. It will also be found on closer inspection that the centre of the straight portion is in each curve in a different place, and that the thinner the plate the

shorter is the exposure necessary to reach the centre portion. This means that a thinly-coated plate is somewhat faster than a thickly-coated one, though they are made of the same emulsion. A thinly-covered plate, however, appears very much faster than it is in reality. It is ineapable of rendering wide contrasts, and hence the negative always looks flat, and thereby gives to the eye the impression of over-exposure.

We have now learnt the great influence which the opacity of the unexposed plate has on the density of the resultant image, and we must now point out a most important source of error in photographic experiments such as we have described.

If a plate be not perfectly evenly coated, the densities, after development, arising from equal exposures will be different on different parts of that plate. We give here an example of a plate, not a bad one either, on which, in different parts, different exposures were given. The table shows the densities which the same exposure produced on the one half and on the other half of the plate:—

Exposure.	I.	II.	Ratio.
	Density.	Density.	
10	·275	·240	1·14
20	·535	·480	1·12
40	·825	·775	1·06
80	1·185	1·080	1·10

The errors on this plate amount to from 6 to 14 per cent., owing to unequal thickness of the film. It is needless to say that in the dark room, in ruby light, such differences in the thickness of the film cannot be observed. The only remedy for this is to use only very thickly coated plates. We may here say that for our most important experimental work we used slow plates specially prepared for us by Mr. Chapman, of Manchester, every care being taken to secure a thick and even film.

Thickly-coated plates give also very much greater latitude in exposure. The plates used in experiments 21 and 22 would have given good pictures of subjects with contrasts varying from 1:80, though the exposures had varied from 1:2, so that an exposure of 10 seconds, or one of 20, would have resulted in but little difference in the negatives, excepting that the one would have been much slower in printing, because generally denser. Thinly-coated plates, on the other hand, need very accurately-timed exposures.

#### VI.—SPEED OF SENSITIVE PLATES.

We gave two formulæ as the result of our investigations; one of them, the approximate one, is the direct result of our experimental work, the other is the mathematical expression of the idea that a certain definite amount of energy is needed to bring a particle of silver bromide into the condition in which it can afterwards be developed, and that it is only to the light absorbed by unaltered silver bromide that increase of density consequent on increased exposure is due.

Whilst the approximate formula is never strictly true, and can be used only for exposures which fall within the period of correct representation, it is extremely simple, and we are about to describe an important application of it, viz., the determination of the speed of the sensitive plate.

The more correct formula cannot be used for practical applications, owing to its complication, but it serves to indicate the limits within which the approximate formula may safely be used.

In the formula—

$$D = \gamma \cdot \log. (O - (O - 1) \beta^{\frac{1}{i}})$$

we may replace  $(O - 1)$  by the symbol  $O$  when that represents a large number, that is, when the plate is richly coated with silver bromide. If, in addition, we remember

that  $\log_e \beta$  is  $-\frac{1}{\gamma}$ , the equation can be transformed into another, viz.:—

$$D = \gamma \cdot \log. \left( \frac{It}{i} \right)$$

which equation holds good only when the numerical value of  $\frac{It}{i}$  is greater than 1 and less than the opacity  $O$ . It is between these two limits only that this equation gives tolerably correct results. Comparing this last equation with the approximation we gave before, it will be seen that the constant  $C$  of that approximate formula is the logarithm of  $i$ , the symbol measuring those properties of the film which together constitute its sensitiveness, and which we termed the inertia of the plate.

Supposing we had two richly-coated plates, with different inertias,  $i$  and  $i_1$ , and we wished to impress the same density upon them by a given intensity of light  $I$ . They would require different exposures, and the exposures would have to be such that—

$$\frac{It}{i} = \frac{It_1}{i_1}$$

or the times would have to be chosen so that—

$$\frac{t}{i} = \frac{t_1}{i_1}$$

This means that if the values of  $i$  are known for different plates, the exposures required to obtain the same results are also known for those plates, if the exposure is known for any one of them.

The determination of the numerical value of the symbol  $i$  is therefore an important problem.

Since the density of the image is an abstract number, it follows that the ratio  $\frac{It}{i}$  is an abstract number also, and that  $i$  is therefore an exposure. We termed this symbol the inertia, and it really measures that exposure which will suffice to change a particle of silver bromide into the developable condition. But for its practical application it has another meaning. It measures the least exposure which will just mark the beginning of the period of correct representation.

The speed of the plate is the inverse value; the longer the exposure needed to bring the plate just to the beginning of the period of correct representation, the slower is the plate. Therefore we measure the speed of the plate by the value  $\frac{1}{i}$ .

The method we adopt for measuring the value of  $i$  is briefly as follows:—We give to the plate at least two exposures falling within the period of correct representation, and develop. We then measure the densities exclusive of fog. We thus obtain two equations connecting the two densities  $D_1$  &  $D_2$  with the two known exposures  $E_1$  &  $E_2$  viz.:—

$$D_1 = \gamma \cdot \log. \frac{E_1}{i} \text{ and } D_2 = \gamma \cdot \log. \frac{E_2}{i}$$

from which we obtain by elimination—

$$\log. i = \frac{D_2 \log. E_1 - D_1 \log. E_2}{D_2 - D_1}$$

and—

$$\gamma = \frac{\log. E_2 - \log. E_1}{D_2 - D_1}$$

The value of  $i$  is expressed in candlemeter-seconds, and can be found by reference to ordinary tables of logarithms.

We will now describe our practice. For the determination of the inertia, only the central portion of the plate should be used; the margin should be avoided, as it is liable to be irregular in thickness of film. In order to insure at least two exposures falling within the period of correct representation we give to the plate eight different exposures of 2·5, 5, 10, 20, 40, 80, 160, and 320 C.M.S., leaving a portion of the plate unexposed. We develop this plate with ferrous oxalate, and, after properly washing, fix in a perfectly clean bath of thiosulphate. We then wash and dry spontaneously,

or by means of alcohol. The length of time for development is judged by the density of the image. We avoid too great density, but develop sufficiently long to obtain a decided deposit for the lower exposures. When all the densities have been measured we subtract from every one of them the density of the fog strip in order to obtain densities "exclusive of fog."

From this series of densities we may calculate the value of  $i$ . For that purpose we find the differences between the consecutive densities, and we choose from the series those points which give differences most nearly alike. As an example we quote the series of results obtained with the Manchester slow plate of experiment No. 21:—

Exposures ...	2'5"	5"	10"	20"	40"	80"	160"
Densities ....	·085	·175	·250	·460	·755	1·010	1·270
Differences	·09	·075	·210	·295	·255	·260	

We should take the results of exposures from 20 C.M.S. to 160 C.M.S., as those falling within the period of correct exposure. Choosing the exposures 20 and 160 for the calculations, we should obtain, in accordance with the formula given—

$$\log. i = \frac{1.270 \times \log. 20 - 0.460 \times \log. 160}{1.270 - 0.460}$$

or  $\log. i = 0.787$ , and from an ordinary logarithm table we should find  $i = 6.12$  candlemeter-seconds.

In another experiment with a plate of the same make the following results were obtained:—

Exposures .....	10"	26"	40"	80"
Densities .....	·300	·590	·910	1·260
Differences ...		·290	·320	·350

Choosing only the 20" and 80" points, we have—

$$\log. i = \frac{1.260 \times \log. 20 - 0.590 \times \log. 80}{1.260 - 0.590} = 0.771$$

or  $i = 5.90$  candlemeter-seconds.

It will be seen that these values for the inertia of the "Manchester Slow" plate are almost alike. With faster plates it is not so easy to obtain quite such concordant values, but they are always sufficiently accurate for practical purposes, for whether an exposure in practice be four or five seconds it will not appreciably alter the resulting negative, so that in the determination of the inertia an error of 10 per cent. is fortunately not of very great consequence, and in most cases two determinations carefully made will not differ more than 10 per cent.

We prefer, however, to obtain the result by a graphic method, by means of which we avoid all calculations and all references to tables of logarithms. We scratch on an ordinary plate a horizontal scale of inertias similar to the scale of an ordinary slide rule, but we repeat the scale four times instead of twice, as in the case of the slide rule. Diagram No. 11 shows this arrangement. We scratch at points 2.5, 5, 10, 20, 40, 80, 160, and 320 of this scale vertical lines (exposure lines), and divide them each into 20 equal parts, marking the highest as density 2.0, the lowest as 0. Having measured the densities we mark them on the scales of the corresponding exposure lines, and draw a straight line through those points which appear to fall most accurately within such a line. It is better to stretch a white thread across these points, as the portion of the line can thus be more easily determined. Where the thread or the straight line intersects the inertia scale we can at once read off the inertia of the plate.

The solutions of the problem of ascertaining the inertia or its inverse the speed of the plate have hitherto been unsatisfactory, and always depended finally upon the judgment of the comparative visibility of letters or numbers printed upon a sensitive plate.

Our method, by referring the speed to a standard candle as unit will enable different operators to obtain almost identical and definite numerical results. Should at any time a better practical unit of light be found, the method is at once applicable with it also. The fact is, we have based our method on the measured effects produced by a given unit of light, excluding the influence of alterations in development, whilst the present method, by means of Warnerke's sensitometer, depends entirely upon development. We could so alter the composition of the developer as to make a rapid plate give most misleading results.

Such a proceeding is impossible with our method.

There is a theoretical possibility that a plate may be rapid to one developer and slow to another, so as to require different exposures according to the developer used. If silver bromide be reduced to metallic silver, 22,700 units of heat must be supplied to replace the heat of combination. Of this amount of heat the developer in the act of development supplies a portion. Ferrous oxalate, for instance, would probably supply 12,900 units, so that the light need only supply the difference, viz., 9,800 units. But if another developer could supply more than 12,900 units, then the light, clearly, need not supply quite so much, and in that case the plate would be faster to one developer than to another.

We have not paid such close attention to this question yet as to enable us to decide it finally, but as far as our experiments have gone we have found very little difference, if any, between the various developers, and we do not feel justified in assigning to the small differences we have observed any great importance.

If a developer could be found which would render a plate materially faster, that developer would strike a serious blow at the hypothesis that the latent image consists of sub-bromide of silver.

We append a number of interesting diagrams representing our graphic method of determining the inertia of a plate.

*Diagram No. 11* shows the general arrangement we adopt for finding graphically the value of the inertia. An ordinary "slide rule" furnishes the mode of subdividing the scale (the distances of the numbers being proportional to their logarithms). The two curves are the curves of Wratten and Wainwright "ordinary" and "instantaneous" plates. It will be noticed that the "instantaneous" plate shows, within the given exposures, a portion of the period of over-exposure, whilst the "ordinary" shows a portion of the period of "under-exposure." The inertia of the one is 1.4, that of the other 5.5, and in round numbers the one plate is four times as fast as the other.

*Diagram No. 12* shows the results of experiment 15 graphically, for the purpose of showing that variations in the mode of development do not influence the determination of the inertia; the densities of the two modes of development being different, yet the straight lines practically converge to the same point.

*Diagram No. 13.*—This diagram represents another variation in pyro development, viz., in the amount of bromide (experiment 16). It will be again seen that the values of the inertia are almost identical, in spite of a considerable difference in the composition of the developer and the time of development.

*Diagram No. 14* shows four determinations of the inertia of one plate (see experiment 14), two determinations being made with eikonogen and two with hydroquinone. The duration of development being different for each determination, yet the results of all four determinations are practically identical.

*Diagram No. 15* shows that the inertia of a plate can be determined after intensification, but not after reduction. It is, therefore, better to develop too little rather than too much. We clearly must not resort to reduction, but we may intensify if the trial plates have been under-developed. (Experiments 19 and 20.)

*Diagram No. 16* shows the determination of inertia of the Ilford plates, "ordinary," "rapid," and "special rapid" (red label). Their inertias are respectively 2.0, 1.4, and 0.56, and their speeds are relatively as 1 : 1.41 : 3.5. The

"red label" plates are the most rapid plates we have so far investigated, but we found it very difficult to ascertain their true speed in the camera, on account of the difficulty in securing adequate density.

When the inertia of the plate is known, it is possible to time the exposures in the camera so that the densities of the gradations are almost exactly proportional to the logarithms of the light intensities which produced them. By this means negatives can be produced which satisfy very nearly the definition we gave of a theoretically perfect negative. It must be borne in mind, however, that such a negative is not necessarily true to nature. The optical density after development differs from the density of the latent image. If the negative is to be true to nature, a plate must be used which is richly coated, the exposure must be carefully timed, and the development must be carried only so far that the value of the development constant  $\gamma$  is numerically equal to 1. Experiments which we have made indicate that for the production of artistic effects on ordinary silver chloride paper, it is necessary to exaggerate the densities, *i.e.*, to prolong the development until  $\gamma$  is greater than 1, and nearly reaches the value 2. This requires further investigation; suffice it to have pointed out that what Captain Abney terms "photographic untruth" is not necessarily inherent in photography, since the photographer has it in his power to decide the degree of exaggeration.

The exposure to be given in the camera can be found by means of the actinograph,\* when the inertia of the plate is known.

The "actinograph speed" of the plate is found by means of the formula  $S = 34 i$ , where  $S$  is the speed and  $i$  the inertia in candlemeter seconds. We find, for instance, the speed of the Ilford plates from their inertia (as shown on Diagram 16).

	Speed.
Ilford "ordinary" = $\frac{34}{2}$	= 17
Ilford "rapid" = $\frac{34}{1.4}$	= 24
Ilford "special rapid" = $\frac{34}{0.56}$	= 60

This method of referring the sensitiveness of the plate to the candlemeter-second as unit will, we believe, greatly promote progress both in the preparation of the plates and in their application. We flatter ourselves that we have supplied one of the greatest needs of plate makers and photographers in general, by enabling them, for the first time, to ascertain accurately the sensitiveness of different plates, and by means of the actinograph to apply this information in practice.

The above is the main practical result which has accrued from our investigations, but incidentally we have shown the fallacy of many popular views on the subject of development, and the paramount importance of correct exposure.

From a purely scientific point of view, perhaps, the most interesting result of our labour is the elucidation of the numerical relation between the exposure and its effect on the sensitive film, and the simple explanation of these relations, based upon the optical properties of the unexposed sensitive film. It would not have been difficult to extend these considerations so as to include in them the reversing action of the less refrangible rays. This would not, however, have served any practical end at present, and it would have complicated the formula very considerably. We reserve for a future communication this extension of the law which we have discovered.

\* See this Journal, 1890, 370, or the Photographic Societies Reporter for April 30th, 1889.

## A NEW ACTINOMETER.

BY EDWARD GEO. BALLARD, A.R.S.M.

A READY method of measuring the amount of actinic rays proceeding from any landscape or object, has for a long time been a thing much desired by photographers both amateur and professional. Many instruments with this view have been from time to time brought out, some depending upon the darkening of prepared paper to a certain tint, others upon the facility with which a series of numbers can be perceived through screens of certain graduated opacities or darknesses. Mr. Leon Warnerke, some 10 years ago, introduced an instrument in which he employed a plate of luminous calcium sulphide, which was excited by exposure to the object about to be photographed, and thus received a certain degree of luminosity, according to the quality of the light proceeding therefrom. After waiting a certain interval of time, the luminosity was measured by viewing the plate through a series of screens of various graduated degrees of translucency and having numbers or letters upon them; the last number or letter visible to the eye was taken as the indicator of the intensity of the light. So it will be perceived that the intensity or degree of the luminosity exhibited by the plate of luminous sulphide, was, in Warnerke's instrument, taken as the measure of the light which had been acting upon it.

Now it is well known (1) that after the sulphide of calcium is excited by exposure to the light and the source of light is suddenly removed, the bluish phosphorescence emitted rapidly fades, especially when the sulphide is excited to its maximum degree; (2) the amount of light necessary to do this is not so very great; for if two halves of a card covered with luminous sulphide be exposed to light, the one half to a northern sky and the other to bright sunshine, the difference of luminosity exhibited by the two halves of the card is not appreciable when they are examined in the dark.

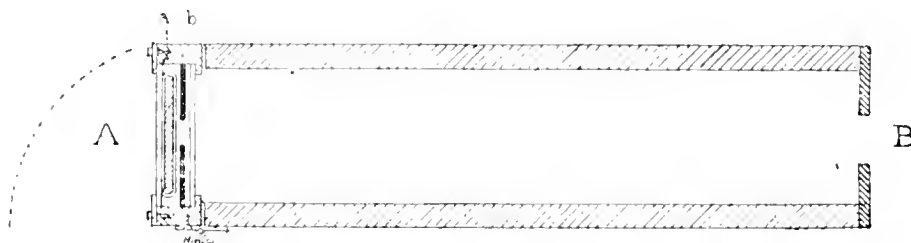
In the instrument about to be described, advantage is taken of these two facts, and of the fact that luminous calcium sulphide or Balmain's luminous paint is excited by, and emits afterwards, *only* those rays which usually act on the photographic plate.

The rate and manner in which the fading occurs are employed in my actinometer as a means and a standard of comparison for measuring the intensity of the light proceeding from any object, landscape, or sea view. This is arrived at by noting the number of seconds it takes for the luminous sulphide (after having been excited to its maximum) to acquire the same degree of intensity as a portion of the light proceeding from the object, landscape, or sea view in question, and then by referring for the corresponding plate-exposure period to a specially constructed table.

Before explaining how the table of exposures mentioned above is arrived at, it will perhaps be as well to describe the actinometer itself. It consists of a wooden or other opaque tube, coloured dull black inside, open at both ends, and of size and shape at one end convenient for adjustment to the eye. The other end is provided with a second, and similar, but shorter, tube hinged to it, so as to form a sort of lid. This lid is glazed next the tube with a piece of clear, or better, ground glass. Over this is placed a piece of card (or glass) coated with Balmain's luminous paint. A portion of this card is removed (a circular portion is found convenient, for many reasons), and behind this is placed a piece of blue glass wrapped in several layers of tissue paper or other translucent material, and, finally, over all is placed a piece of ordinary glass. The blue glass is capable of cutting off all rays except those corresponding to the colour of the light proceeding from the luminous paint. The use of the tissue paper is to lessen the intensity of the light which passes through the circular opening in the card covered with the luminous paint, to the degree requisite to give correct results with the instrument. This once found is constant for any given make of photographic plate.

To use the instrument, allow the hinged portion A to (Fig. 1.) fall, so that the side containing the card with the luminous paint upon it shall be fully exposed to daylight for 30 seconds. By this time the paint will be excited

Fig. 1.



*a* — Blue glass wrapped in tissue paper.

*b* — Card covered with luminous paint with circular hole in centre.

Fig. 2.

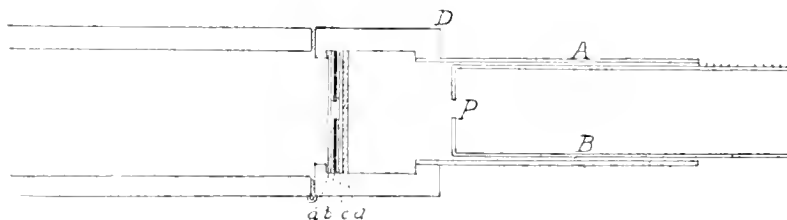
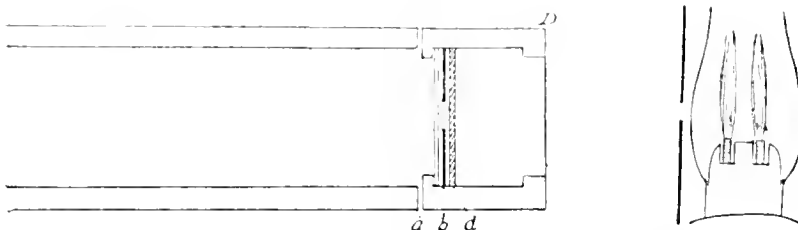


Fig. 3.



*a* — Ground glass.

*b* — Card with luminous paint.

*c* — Tissue paper.

*d* — Blue glass.

to its maximum degree. Place the eye at the end B. Then quickly close the hinged portion A. A blue luminous ground with a dark spot upon it will be seen.

Count the number of seconds it takes for the spot to disappear, *i.e.*, to acquire the same tint as the surrounding luminous sulphide. Then refer to the table, and opposite the number of seconds required for this to take place will be found the correct exposure to be given to the particular make of photographic plate to which the instrument is adjusted. The actinometer itself can easily be adjusted to a plate of any degree of speed by removing or adding to the layers of tissue paper behind the card or glass carrying the luminous paint. Any good table of exposures may be employed for this purpose, or better still that excellent little machine for calculating photographic exposures, called the "actinograph," invented, after much labour and study, by Dr. Ferdinand Hurter and Mr. Driffield, of Widnes.

*The Table of Exposures.*—In order to construct this, it was of course necessary to ascertain how and in what manner the luminous paint faded, in other words to determine the relative intensity of the luminosity of the paint after the lapse of various intervals of time. Three methods were employed for this purpose, and the results obtained by all the methods were found to agree. First, an instrument was made similar in construction to the actinometer already described, except that in addition the hinged portion was provided with a tube, A, inside which another tube, B, was made to slide light-tight, as shown in the drawing, Fig. 2. The inner tube was closed at the end nearest the

tissue paper screen, with the exception of a small circular opening, P, forming a diaphragm which, when presented to a clear northern sky, constituted the standard of light for the experiments. The tube B was graduated in tenths of an inch. In other respects the instrument resembles the actinometer already described, containing in the hinged lid the luminous sulphide with the circular central portion removed, and backed with tissue paper and blue glass.

The luminous surface, having been excited by allowing the hinged portion to fall for 30 seconds and closed again, was observed to acquire the same tint as the central spot at the end of 10 seconds, when the instrument was directed to a clear northern sky, the distance from the tissue paper C to the diaphragm P being noted. The tube B was then drawn out until the diaphragm P was twice the distance from the tissue paper that it was at the first observation. The spot, on repeating the experiment, was found to disappear in 40 seconds. At the next experiment this last distance from the screen was doubled, by drawing out the tube B still further, *i.e.*, to a distance four times what it was in the first experiment. It required 160 seconds now for the spot to vanish.

The next experiments were made with a duplex paraffin lamp. The wicks of the lamp employed were a full inch in width, giving a flame from  $1\frac{1}{2}$  to  $1\frac{3}{4}$  inches wide. At a distance of  $1\frac{1}{4}$  in. from the centre of the lamp between the two wicks a diaphragm was placed  $\frac{1}{2}$  in. in diameter. The light passing through this diaphragm was that employed for the experiments which were conducted with the same

instrument as that already described, with this difference, viz., that the tubes A and B were removed, also the tissue or oiled paper screen c, which latter was placed at the end of the instrument as shown in figure 2 at D. The first observation was taken at a distance of 5 inches from the diaphragm to the tissue paper screen D, when the spot vanished in 10 seconds. The following observations were then made, taking 5 in. as the unit of distance:—

	Intensity.
At a distance .50 from the diaphragm the spot vanished in 2.5 seconds .....	$\frac{1}{4}$
At a distance .75 from the diaphragm the spot vanished in 3.0 seconds .....	$\frac{1}{3}$
At a distance .85 from the diaphragm the spot vanished in 7.0 seconds .....	$\frac{1}{7}$
At a distance 1.00 from the diaphragm the spot vanished in 10.0 seconds .....	1
At a distance 2.00 from the diaphragm the spot vanished in 40.0 seconds .....	$\frac{1}{4}$
At a distance 3.00 from the diaphragm the spot vanished in 90.0 seconds .....	$\frac{1}{9}$
At a distance 4.00 from the diaphragm the spot vanished in 160.0 seconds .....	$\frac{1}{16}$

The intensities of the light emitted by the paint at the end of the various periods of time will be inversely as the square of the distances 1, 2, 3, 4, &c. So by comparing the light passing through the tissue paper and blue glass with the fading luminous paint, its intensity can easily be ascertained; the number of seconds required for the spot to vanish being indicative of the intensity of the light under examination.

In order to check the above, a series of experiments were made with carefully cut diaphragms, placed at a fixed distance from the tissue-paper screen. These diaphragms had the following diameters,  $1, \frac{1}{2}, \frac{1}{4}$ , and the results of the experiments are given below:—

Diameter of Diaphragm.	Intensity of Light admitted.	Seconds for Spot to vanish.			
		Exp. 1.	Exp. 2.	Exp. 3.	Exp. 4.
1	1	10	2.5	7.5	3
$\frac{1}{2}$	$\frac{1}{4}$	40	10	30	12
$\frac{1}{4}$	$\frac{1}{16}$	160	40	120	48

It will be seen that the results are relatively the same.

The following is an example of the application of the foregoing facts. Suppose that upon a certain day, with an F<sub>16</sub>-stop, a plate requires 1 second exposure to obtain a good picture, and that the dark spot of the actinometer vanishes in 10 seconds. Again, suppose that on making an observation of the light on another occasion it is perceived that the spot vanishes in 40 seconds. The exposure needed in the latter case would be four seconds, because the light emitted by the paint at the end of 40 seconds is only  $\frac{1}{4}$  the intensity it was at the end of 10 seconds. This is the same as saying that the light transmitted by the circular opening in the card covered with the luminous paint is only  $\frac{1}{4}$  what it was in the first case: this is precisely what it is desired to measure. When the instrument is thus set to any given plate, the exposure is  $\frac{1}{16}$  of the number of seconds required for the spot to vanish after the luminous surface has been excited. For plates of other degrees of speed special tables can easily be constructed. The instrument when once adjusted to a particular make of plate will indicate the correct exposure under any condition of light and this *with rapidity*, the element of judgment being dispensed with altogether. The difference between the actinometer described and that invented by Warnerke is quite evident. I simply employ the fading sulphide, after being fully excited by exposure to light, as a standard or sliding scale with which to compare a portion of the light proceeding from the object about to be photographed. I have found the instrument extremely useful, and I never have a failure now due to over or under exposure.

## Manchester Section.

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Committee:

J. Angell.	J. Grossmann.
G. H. Bailey.	P. Hart.
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G. E. Davis.	Sir H. E. Roscoe, M.P.
H. Grimshaw.	C. Truby.
Harold B. Dixon.	D. Watson.

Hon. Local Secretary:

J. Carter Bell, Bankfield, The Cliff, Higher Broughton, Manchester.

SESSION 1890.

Notices of Papers and Communications for the Meetings to be sent to the Local Secretary.

Meeting held at Victoria Hotel, Manchester, on Tuesday, 1st April 1890.

MR. IVAN LEVINSTEIN IN THE CHAIR.

### A CONSTANT AIR-BATH.

BY H. GRIMSHAW, F.C.S.

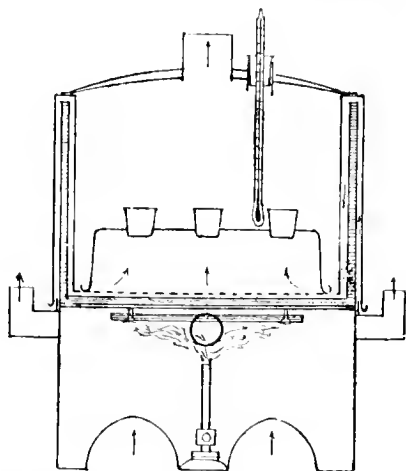
AN air-bath, considered as an apparatus for the quantitative dessication of all kinds of material, should aim at the envelopment of the substance in a moving body of pure air, which is supplied at as near a constant temperature as possible. This is the object, and the more simple and inexpensive the appliance can be made the better. The ordinary form of air-bath, constructed like a cupboard of sheet copper, violates nearly every condition required to attain the objects in view. It is simple enough certainly, but its square form makes it more expensive of construction than a cylindrical one, and its construction makes it absolutely impossible to maintain anything like a constant temperature. In addition to this, the products of combustion from the burner used to heat it corrode the exterior and fill the interior with moisture and carbonic and sulphurous acids.

Adams, in a paper published in the Analyst, XIV., 222, after pointing out the conditions which the ordinary air-bath violates, describes a new form of air-bath which he had constructed, which fulfilled the conditions of being constant to a remarkable degree and entirely prevented the access of the products of combustion to the interior of the bath. On careful consideration of the construction of this bath, which is illustrated by a diagram, I considered that a much simpler, cheaper, and practically as efficient an air-bath might be made, and the result of my attempt in this direction having been, so far as I can judge, very satisfactory, I considered that it would be of interest to the Society.

The construction of the bath will, I think, be seen at a glance from the diagram appended, which is one-eighth the real size. It consists of four parts—(1), the body; (2), the vessel forming the air envelope or casing; (3), the base all of which are cylindrical; (4), the lid or cover. The body is made of cast iron or brass, and is merely a cylinder 11½ in. in diameter externally and 8 in. deep, closed at the bottom, and of a uniform thickness of ½ in. The object in making this so thick is to provide a mass of metal which will distribute and to a large extent control the heat. This part might be made of sheet copper or even stout sheet iron, but would not be so effective. Also the body and the base might be cast in one piece, but this would add unnecessary weight.

The base is made of stout sheet iron or sheet copper, and is, in the model I have made and used 11½ in. in diameter and 7 in. deep. It is cylindrical, and closed at the top. It is

provided at the bottom with semicircular apertures for the admission of air to the Bunsen lamp, and close to the top are four short chimneys of sheet iron at least 1 in. in diameter turned upwards at a right angle. The object of these is to carry away the products of combustion and deliver them at a level above that of the intake of air to the drying chamber, thus avoiding the presence of these gases in the interior of the air-bath. Attached to the top of this part of the apparatus is a kind of baffle-plate of  $\frac{1}{2}$ -in. iron to receive the flame of



the Bunsen instead of allowing it to play directly upon the top of the base cylinder, by which means radiation of heat from the bottom of the bath is lessened, and the sheet metal of the base is not burned away. I need scarcely say that all the joints of this heat chamber or base should be made tight without the use of solder, which would be melted out. A rose burner is better than the ordinary Bunsen as the source of heat. In the next bath which I make I intend to make the base of a slightly greater diameter, so that in packing the air-bath for transit or storage the other portions may be placed inside it to save room.

The third portion of the bath, which I will call the air-case, is merely a double cylinder of sheet iron or copper open top and bottom, but with the space between the two cylinders closed at the top and open at the bottom, so as to slide over the body of the bath, leaving an air space of about  $\frac{1}{4}$  of an inch on each side of the body, the thick metal of which imparts its heat to the air as it travels up the outside and down the inside. The air-case is of course cylindrical, the internal cylinder being  $10\frac{1}{2}$  in. in diameter, and the outer cylinder about 1 in. wider, whilst the height of each is about  $8\frac{1}{4}$  in. I propose to try the effect of adding still another enveloping cylinder externally to this part of the bath, through which the heated air from the base will circulate, which will give an economy of heat and possibly still greater regularity. A series of "notches" taken out of the bottom edge of the interior cylinder admit the hot air uniformly all round the cover of the circumference of the air-bath, and this is caused to rise very uniformly into the whole of the chamber by means of a finely perforated plate, shown by the dotted line.

The fourth part of the air-bath is the cover which is made double to prevent radiation of heat, and has a chimney 2 ins. in diameter and about 2 ins. or 3 ins. high. The chimney may have a circular damper fixed on a swivel or a series of perforated caps to regulate the draft of air through the drying chamber. Through the lid there is a 1-in. aperture for a cork carrying the thermometer. Another similar aperture may be made to carry a maximum thermometer to indicate whether, between the intervals of reading, the heat of the bath has been any time too high.

The diagram shows a stand inside the bath of about the right height to bring the vessels containing the substance into the most constant zone of heat. The outside of the bath may be of course japanned or coloured as desired.

The air-bath of Mr. Adams, previously alluded to, contains a closed cylindrical chamber encircling the air chamber, the expansion of the air in which, by the usual contrivance of a mercury lute cuts off the supply of gas more or less according to the temperature. This introduces an expensive complication into the construction of the bath, which, I fear, will always be liable to go wrong and render the addition useless. If necessary for very refined work of course an expansion apparatus could be added to this air-bath, but I should prefer it to be entirely separate in construction from the bath itself, and its connexion with the gas supply pipe to be through an aperture in the cover, such as the one for the thermometer.

In practice I have found that with the Manchester gas supply there is no real need for a regulator, if the gas supply is looked to in the morning and evening when the extra pressure is put off and on the gas mains, the bath keeps within about one degree as a rule.

In working further with the air-bath I shall obtain a record of its performance which, if of sufficient interest, I will take the liberty of placing before the Society.

#### NOTE ON THE DECOMPOSITION BY HEAT OF THE CHLORIDES OF MAGNESIUM AND CALCIUM.

BY H. GRIMSHAW, F.C.S.

It is not a new observation that when the chlorides of calcium and magnesium are heated in contact with air, a portion of this chlorine is given off.

In view of the very large quantities of both these substances used in the sizing and finishing of cotton and other goods, it is evident that it is of considerable interest and importance to define at what temperature, at how low a temperature in fact, and to what extent the decomposition of these salts proceeds, because if the chlorine is liberated at temperatures to which it is at all likely that the fabrics containing them may be subjected, under the ordinary conditions of their use and manufacture, then the chlorine or resulting hydrochloric acid will be certain to cause more or less deterioration of the fabrics.

We know that at a red heat the chloride of calcium becomes alkaline to litmus, and that at temperatures considerably lower than this the chloride of magnesium parts with an appreciable amount of its chlorine. Recently several cases of deterioration of the strength of cotton fabrics have been traced to the action of chloride of magnesium, and we may take it, I think, as an undoubted fact that this "tendering" of the cotton fibre in such fabrics is due to the action of the hydrochloric acid formed by the decomposition of the chloride.

I am making an attempt to define with accuracy the lowest limit of temperature at which the decomposition of the chlorides of calcium and magnesium, and incidentally the chloride of zinc, takes place, the extent of the decomposition, and the influence of time, and the presence of moisture, have upon this, and I am able to give some figures which though only of a preliminary nature are of interest on some of these points.

The method I have adopted in the experiments is as follows:—

I prepare a solution in distilled water of the chlorides of calcium and magnesium respectively, of which I take so many cubic centimetres for each trial, so avoiding a good deal of time and trouble in weighing. I also prepare a decinormal solution of silver nitrate, which serves to estimate, by the usual volumetric method the chlorine either before or after heating.

(1.) Twenty-five cc. of the calcium chloride solution took  $18.3$  cc. of the decinormal silver nitrate, and  $25$  cc. of the magnesium chloride solution took  $16$  cc. of the silver nitrate.

(2.) Twenty-five cc. of the calcium chloride and magnesium chloride solutions respectively were evaporated to



dryness on the water-bath, and then subjected for five hours to a temperature of  $150^{\circ}\text{C}$ . in the air-bath which I describe in another paper. The residue was in each case taken up with water, and after filtration, titrated with the standard silver solution. The calcium chloride took  $18.3\text{ cc.}$  as before, and the magnesium chloride only  $9.7\text{ cc.}$ , showing a very considerable loss of chlorine.

(3.) Twenty-five cc. of the calcium and magnesium chlorides solutions were evaporated to dryness as before and subjected to a temperature of  $165^{\circ}\text{C}$ . for a period of two hours. On titration as before, the calcium chloride residue took again  $18.3\text{ cc.}$  of standard silver, whilst the magnesium chloride residue took  $10.7\text{ cc.}$  The temperature was higher, and the time of exposure less than in experiment No. 2.

(4.) Twenty-five cc. of the magnesium chloride was taken, evaporated, and heated as before, but with a temperature of  $117^{\circ}\text{C}$ . and an exposure of three hours. On solution and titration as before,  $15.2\text{ cc.}$  of the standard silver were consumed.

These few preliminary experiments would appear to establish the fact, that at any temperatures which can possibly prevail in the manufacture or use of goods containing the calcium chloride, this salt is perfectly stable, and no fear of deleterious action need be entertained, whilst the reverse is the case with the magnesium chloride. The figures for the latter show that this salt is decomposed comparatively readily at a low temperature; a temperature of  $117^{\circ}\text{C}$ . is one which in some operations the fabric may easily attain, and it would appear likely that an even lower temperature may effect this.

I hope shortly to complete an extensive series of experiments to define these points more definitely still with regard to the magnesium and calcium, and also directed to show whether the chloride of zinc is more or less stable than the chloride of calcium, as it is a salt which is also very largely used in the same connexion.

I should like here to acknowledge the co-operation of my laboratory assistant, Mr. W. Wilson, in the carrying out of the method of experimenting.

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The names in *Italics* are those of members of Committee who retire at the end of the current Session. The following have been selected to fill the vacancies, and will take office in July next:—*Chairman: E. C. C. Stanford; Vice-Chairman: A. Crum Brown; Secretary: G. G. Henderson; Treasurer: W. J. Chrystal; Committee: W. J. A. Donald, C. A. Fawcett, J. Falconer King, T. P. Miller, J. Pattison, and R. R. Tatlock.*

### SESSION 1890.

June 3rd (Glasgow):—

Dr. J. Clark and Mr. McGhie. "A new Cobalt Mineral."

Notices of Papers and Communications for the Meetings to be sent to the Local Secretary.

*Meeting held 6th May 1890, in the Rooms of the Philosophical Society, Queen Street, Edinburgh.*

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## AN ACCOUNT OF THE MANUFACTURE OF PHOSPHORUS.—PART II.

BY J. B. READMAN, DSC., F.R.S.E., F.C.S., EDINBURGH.

THE first part of this paper on the manufacture of phosphorus described in general terms the present method of preparing that element on the industrial scale. The process so described is open to many objections, and is one which results in much loss of material.

In the first place it involves, for its economical accomplishment, what may be considered independent manufactures which require to be conducted conjointly with it, each one involving its distinct plant and apparatus. For example, to supply the acid necessary for the decomposition of the phosphate of lime, vitriol chambers, with their accessories, require to be erected.

An extensive breaking and grinding plant for the raw material must also be available, and further, in order to provide retorts, which ought certainly to be made on the spot, commodious premises for moulding, drying, storing, and ultimately firing them, have to be conveniently arranged. Besides all this, the extensive filtering service required, the disposal of the waste sludge (hydrated sulphate of lime), the numerous store and stock tanks for acid liquors of different strengths. The series of lead evaporating and drying pans, and the distillation at a high temperature of the desiccated mixture of phosphoric acid and carbon in what are practically small pipes or crucibles, each one yielding only a pound or two of phosphorus involve a large plant, and the manipulation of much material contrasted with the small amount, comparatively, of phosphorus obtainable.

Moreover, the process I described is wasteful on account of the loss of material entailed at its various stages; even with the most careful washing of the sulphate of lime in the filters it is impracticable to remove all the free phosphoric acid from it, for to effect this completely the effluent washings would be so weak and so charged with sulphate of lime in solution as to render their introduction into the process objectionable rather than helpful.

Loss of phosphoric acid is also incurred—as was pointed out—by the presence of bases, such as iron and alumina in the concentrated acid liquor and the ultimate re-formation of insoluble phosphates; but the most serious source of loss, and that of phosphorus in its free condition, occurs in the distillation of the desiccated mixture of acid and carbon in clay retorts. Whatever amount of care is bestowed on the manufacture of retorts, frequent cracking and breaking, or pinholes, accompanied by escape of phosphorus, are inevitable at the high temperature required for the operation. This is abundantly proved in the case of zinc, the distillation of which bears a strong analogy to that of phosphorus, but in the former case the retorts are subject to no internal pressure whatsoever, whereas the phosphorus retorts have a slight water seal, and this undoubtedly is a factor when accounting for the loss in distilling in fireclay vessels so inflammable a substance as phosphorus.

Furthermore, the wear and tear throughout the process is excessive, and the cost for upkeep of plant and the renewal of retorts and furnaces very heavy.

With these facts before me I initiated some years ago a series of experiments to ascertain if a more simple and cheaper process of producing phosphorus could be found, dispensing with the use of sulphuric acid and also with the employment of retorts.

For the last three years or more, as circumstances permitted, I have carried out many experiments in this direction, involving upwards of 250 separate fusions, many of them requiring a continuation of the heat for several hours' duration.

The materials used in the first instance were phosphate of lime, silica in the form of sand, and carbon in various conditions, and the apparatus consisted of Morgan's plumbago crucibles "Salamander" size No. 8, and a Sefström's blast furnace.

The object of the preliminary experiments was to ascertain first if silica at high temperatures, and in presence of carbon is capable of expelling, practically, all the phosphorus contained in an intimate mixture of these two substances along with phosphate of lime; and, second, if the silica after, or during, the expulsion of the phosphorus, formed with the lime of the phosphate of lime a fusible slag which would remain in a liquid condition so as to admit of its ready withdrawal.

Specimens, showing the results of some of the experiments, are lying on the table, and will be severally referred to.

In the first of the series of experiments the following proportions were employed, viz. :—

	Parts.
Charleston phosphate of lime .....	100
Sand .....	40
Coal .....	15

These ingredients—all in a fine state of division—were intimately mixed, then slightly damped to promote cohesion, and finally introduced into a plumbago crucible previously raised to a full red heat in the Sefström's blast furnace. The crucible remained in the furnace for one hour and 15 minutes, and during part of that time was subject to a bright red heat approaching whiteness; it was then removed, allowed to cool, and examined.

It was found that fusion had taken place to a small extent round the sides of the crucible, but that the interior was practically unchanged. The resulting slag (part of which is contained in the specimen bottle), was so unsatisfactory that no analysis was made of it—very little phosphorus appeared to be evolved.

The next trial was precisely the same as the one just described, with the one exception that more sand was used in the mixture. The result was a rather increased fusion with partial decomposition of the phosphate of lime, but still incomplete and unsatisfactory.

Having failed in these two experiments to effect complete fusion and decomposition of the mineral phosphate of lime, it was resolved in the next experiment to substitute for the latter basic slag from the Bessemer steel process. The proportions used were—

	Parts.
Basic slag (ground) .....	100
Sand .....	67
Coal (ground) .....	10

These ingredients were carefully mixed and treated as before described.

The mixture fused completely in 15 minutes, and on pouring out the contents of the crucible and cooling, the slag was found to be vitreous in character and of a dark greenish-brown colour (see specimen). Besides the slag a large metallic, very brittle, button was obtained, which on examination was found to consist of phosphide of iron possessing the following composition—

Iron .....	71.63
Phosphorus .....	23.23
Silicon, manganese, &c. ....	5.00
	100.00

This corresponds approximately to the formula  $\text{Fe}_2\text{P}$ .

It was found in this experiment, and the result was amply confirmed in others which followed, that when basic slag is heated with the proper proportion of silica and carbon—though perfect fusion may result—a very small proportion of the phosphorus is expelled from the crucible, the great affinity that iron possesses for phosphorus preventing the expulsion of the latter, which if liberated at all at once combines with the large quantity of iron present in these basic slags, with the formation of phosphide of that metal while the lime, manganese, magnesia, &c., combine with silica to form a complex silicate, which, as is well known, is fusible at temperatures that silicate of lime withstands.

(I shall have occasion to refer once again to some further experiments with basic slag, the only phosphorus-bearing

material present in the mixture, during which a very much increased temperature was employed.)

Failing to expel phosphorus at the temperature employed from either native phosphate of lime or from basic slag, experiments were next made using a mixture of the two. The first of the series was performed with an intimate mixture of the following substances—

	Parts.
Charleston phosphate of lime (11.86 per cent.) ..	100
Basic slag .....	50
Sand .....	75
Coal .....	41
	270

This mixture, after one hour in the crucible, produced a very fluid slag (see specimen), and during the operation a large quantity of phosphorus was disengaged, which blazed at the mouth of the crucible with characteristic brightness. The contents of the crucible after the operation weighed 167 parts, and the clean slag on analysis was found to contain 1.37 per cent. of phosphorus only. Proving that a large quantity of phosphorus had been removed during the fusion, shots of phosphate of iron from the basic slag were found dispersed through the residue.

The next experiment was made with the same materials, using slightly different proportions, viz. :—

	Parts.
Charleston phosphate of lime (11.86 per cent. P.) ..	100
Basic slag .....	33
Sand .....	75
Coal .....	33
	241

This mixture fused well, and the resulting residue weighing 175 parts had the following composition :—

	Per Cent.
Phosphorus .....	2.23
Silica .....	55.60
Lime .....	27.37
Undetermined .....	14.80
	100.00

In this case the total phosphorus existing in the original mixture amounted to 1036.75 grains—the phosphorus remaining in the slag and buttons 292.3 grains—the difference, 744.72, being phosphorus expelled. This is equivalent to 71.8 per cent. of the whole phosphorus contained in the mixture.

The escape of phosphorus as before was sufficiently evidenced by the bright flame of that element proceeding from the crucible during the decomposition. Seven supplementary experiments were next made, varying the proportions of the ingredients slightly in each case.

The next material used was pure bone ash, intimately mixed with basic slag, sand, and coal. The proportions were—

	Parts.
Bone ash (17.66 per cent. P.) .....	100
Basic slag .....	33
Sand .....	80
Coal .....	33
	246

This mixture also fused well and yielded a residue which weighed 176 parts and contained—

	Per Cent.
Phosphorus .....	2.82
Silica .....	49.12
Lime .....	34.13
Undetermined .....	13.93
	100.00

In this mixture, inclusive of the basic slag, there were 1324.5 grains of phosphorus, and in the residue 372.8 grains, showing that 951.7 grains had been expelled or 71.1 per cent. of the whole.

This slag was extremely fluid and highly vitreous (see specimen). Having now proved the possibility of expelling phosphorus from the ordinary phosphates of lime met with

in commerce, the next endeavour was to condense it, and with this object a charge was made up as follows:—

	Parts.
Charleston phosphate of lime.....	100
Basic slag.....	33
Sand.....	71
Coke.....	27
	<u>231</u>

The ingredients were carefully ground and mixed as before and were then introduced into a tabulated plumbago retort made specially for these experiments by the Morgan Crucible Company, Battersea; the retort was placed in the Sefstrom furnace and was then connected to a series of condensing vessels. The heat was carefully and gradually raised until a high temperature was attained. Phosphorus soon began to distil over, and when the distillation appeared to be at an end, the retort was disconnected and the condensed phosphorus collected. The result gave—

Total phosphorus in charge.....	968.25 grains.
Phosphorus condensed.....	534.00
Do. escaped uncondensed.....	264.25
Do. left in residue.....	170.00
	<u>968.25 grains.</u>

This is equivalent to a yield of 55.1 per cent. of the whole phosphorus present in the mixture, and includes 104.2 grains of phosphorus (=10.7 per cent. of total present) contained in basic slag, which, as we have seen, yields practically no free phosphorus when treated in this way.

The next endeavour was to dispense with the employment of basic slag and to imitate, as far as possible, the very fluid slags obtained in the reduction of iron ores in the blast furnaces.

A typical slag from one of the furnaces in the West of Scotland was obtained and its composition determined. The following is the analysis:—

	Per Cent.
Silica.....	38.60
Alumina.....	12.85
Lime.....	32.54
Oxide of manganese.....	4.98
Oxide of iron.....	.64
Magnesia.....	6.49
Sulphide of calcium.....	3.58
Phosphoric acid.....	Trace.
	<u>99.68</u>

This slag, when introduced into the crucible, fused readily in 10 minutes and remained fluid for three minutes after pouring into the small ladle.

A mixture was now made with—

	Parts.
Charleston phosphate of lime.....	100
China clay.....	50
Charcoal.....	17

This mixture fused well and yielded a slag (see specimen) which contained 1.86 per cent. of phosphorus.

Another trial was made with the addition of 20 parts of sand. This mixture also fused readily and gave a slag of the following analysis. (See specimen.)

	Per Cent.
Phosphorus.....	1.74
Silica.....	40.92
Lime.....	37.70
Alumina.....	17.08
Undetermined.....	2.56
	<u>100.00</u>

On the table are slags from six additional experiments with different proportions of these ingredients.

Further trials were next made, using in addition black oxide of manganese. The slag resulting (see specimen) has the characteristic manganese colour, and its chemical composition closely approximates to the iron slag before referred to. The phosphorus remaining in this case amounts to only 0.86 per cent.

### Analysis of Slag.

	Per Cent.
Phosphorus.....	0.86
Silica.....	42.44
Lime.....	32.41
Alumina.....	15.61
Manganese, &c.....	8.69
	<u>100.00</u>

Specimens of the results of further experiments are before you.

Having succeeded both in obtaining a readily fusible slag and also in expelling phosphorus from these mixtures, repeated attempts were again made to obtain phosphorus from basic slag as being the cheapest source of phosphorus at present in the market. A mixture was made of—

	Parts.
Basic slag.....	100
Fire-clay.....	50
Charcoal.....	50

A high temperature was used and the mixture became perfectly fluid in the short space of 10 minutes, but as before, it was found that practically all the phosphorus combined with the iron present in the slag, forming the brittle phosphide (see specimen) described previously.

Arrangements were now made to employ a higher temperature in future experiments; this was accomplished by increasing the pressure of the air blast and by the employment of a superior quality of coke in the furnace. The object of the increased temperature was to endeavour to effect the decomposition of phosphate of lime by silica and carbon alone—as in the first experiment of all—without the use of extraneous fluxes. A trial was made with an intimate mixture containing—

	Parts.
Charleston phosphate.....	100
Sand.....	50
Coke.....	20

This was introduced into a plumbago crucible previously heated to whiteness in the Sefstrom furnace, and this temperature was maintained during the experiment.

The result was a very fluid slate-coloured slag of the following composition:—

Phosphorus.....	1.35
Silica.....	49.88
Lime.....	33.04
Undetermined.....	15.73
	<u>100.00</u>

Similar results were obtained using other mineral phosphates, such as "Canadian," "Somme," "Spanish," thus proving that if temperature employed be sufficiently high fluxes are not required to form fusible slags with these substances. Having established this point the next and principal one was to ascertain the yield of phosphorus from these simple mixtures, and for this purpose a charge weighing about 5 lb. was prepared and distilled in a plumbago retort, precisely similar to the one on the table. The details of this experiment are as under:—

WEIGHT OF ORIGINAL MIXTURE 34,000 GRAINS, CONTAINING 2,372 GRAINS P.

	Grains P.
Weight of slag 22,760 grains, containing 1.3 per cent. P, equal to.....	295.8
Weight of phosphide of iron 320 grains, containing 23.2 per cent. P, equal to.....	74.2
Total P left in residues.....	369.0
Yield of crude phosphorus obtained in condensers.....	1,730.0
Phosphorus which escaped condensation, by difference.....	273.0
Total P in original mixture.....	<u>2,372.0</u>

(This is a yield of 72.2 per cent. of crude phosphorus.)

Upwards of 20 confirmatory experiments were made, which fully proved the correctness of the figures here tabulated.

Having now given sufficient evidence of the possibility of obtaining phosphorus by the action of silica and carbon on mineral phosphates forming as a by-product a fusible slag,

my next endeavour was to devise an apparatus to carry out the reaction, as both from the nature of the slag and the very high temperature required retorts were not capable of being employed for any length of time. Patents had been taken out by several investigators (Aubertin, Brison, and Serve), all Frenchmen, claiming among other things the employment of the blast furnace for the manufacture of phosphorus. These patents have all lapsed, and do not appear to have ever been prosecuted on a technical scale for any length of time. The specifications lie on the table, and may be of interest. The following is a short summary of each. Aubertin and Boblique, of Paris, took out in 1867 provisional protection for "A new or improved process for the direct production of phosphorus for industrial purposes," in which phosphate of lime is mixed with 200 per cent. of silica and 25 per cent. of charcoal or coke dust, and introduced in a pulverised state in a closed vessel, and heated to a temperature between orange red and white heat, the reaction (it is added) taking place without it being necessary to melt the mixture?

In 1868, Brisson, of Chalons-sur-Saône, took out a complete patent for "Improvements in the manufacture of phosphorus and amorphous phosphorus, and in furnaces and apparatus employed for that purpose." He makes two claims: first, the manufacture of phosphorus by means of a blast furnace combined with a blowing engine; second, the direct admixture in the blast furnace of the fuel with the materials for producing the phosphorus.

In 1870, Aubertin again appears, having taken out provisional protection only, for "Improvements in the manufacture of phosphorus," in which he employs the same materials as before, adopting what he describes as the "calcination" or the "fusion" process. In the former case he uses furnaces or retorts and excess of silica, to prevent fusion, and in the latter he adds just sufficient silica to form a fusible slag, and employs for the reaction "that description of blast furnace known as 'Fluss-Ofen,' employed in Styria and Carinthia."

Lastly, in 1879, Serve, of Givors, France, took out a complete patent for "Improvements in the manufacture of phosphorus," in which the following claims are made: First, the preparation in the form of blocks or bricks of an intimate mixture in suitable proportions of phosphates of lime, baryta, or other earthy phosphates, and of silica or other silicious body, bound or held together by a carburetted body, such as tar, pitch, or coal. Second, The extraction of the phosphorus from above by the employment of ordinary retorts or other suitable apparatus, but by preference a blast furnace similar to those used for smelting iron. Third, The construction of apparatus shown in the drawing, consisting of a furnace and condenser connected therewith.

The other patents that have been taken out in this country in connexion with the manufacture of phosphorus will be fully entered upon in the concluding paper on this subject.

The blast furnace as a substitute for retorts seemed an apparatus likely to be successful, especially in view of the fusible mixtures obtainable; and with the object of proving it I erected several successive eupola furnaces in the neighbourhood of Edinburgh at a cost of over 1,200*l*. The first one was soon demonstrated to be too small, and a water-jacketed eupola of 18 in. internal diameter was substituted. This was used for some time, and was then modified, and finally a brick-lined furnace, about 3 ft. 6 in. internal diameter, was erected. The finely-ground and incorporated ingredients were made into bricks or balls by various binding agents, and presented the appearance of the specimens before you. The blast was obtained by a Roots' pressure blower, and both the hot and the cold blast were employed.

I have tracings of these furnaces and blowers here, but the description of the experiments will form part of the final paper, as perhaps I have entered already too much into the details of the preliminary work.

## BLASTING-GELATIN, AND SOME OTHER EXPLOSIVE MIXTURES.

BY GEORGE MCROBERTS, F.R.S.E., F.C.S., F.L.C.

IN a former paper I dealt with the method of manufacturing blasting-gelatin, but in this paper I intend to speak more of the material itself than of the way of preparing it. Blasting-gelatin, properly so called, consists of from 92 per cent. to 93 per cent. of nitro-glycerin and 7 per cent. or 8 per cent. of nitro-cotton. The kind of nitro-cotton used is not the ordinary gun-cotton, or tri-nitro cellulose, but is a mixture of mono- and bi-nitro-cellulose. The latter is soluble in a mixture of alcohol and ether, and forms the well-known colloid; and it is also soluble in nitro-glycerin, especially when the latter is heated. With nitro-glycerin it forms a jelly thick or thin according to the proportion of nitro-cotton used. Blasting-gelatin, consisting of 93 per cent. of nitro-glycerin and 7 per cent. of nitro-cotton, is the strongest form of it, and so far as experience has yet shown, it is the most powerful explosive in existence. It has a specific gravity of 1.55; is of a yellowish colour, semi-transparent, and is elastic to the touch. It is insoluble in water, in which it can remain any length of time without injury to its explosive properties; and, when exploded, it is wholly resolved into gas at a high temperature; and the ballistic result is found by the mortar to be about 50 per cent. greater than that of the best dynamite. For some purposes, this form of blasting-gelatin is the best. It can be used under water with great facility. There are, however, some kinds of blasting for which milder forms of gelatin are more suitable; and, accordingly, the manufacturers have endeavoured to supply the wants of customers by modifying in various ways the energy of the explosive. This is done by mixing with thin gelatin, consisting of 95 per cent. or thereby of nitro-glycerin and 5 per cent. of nitro-cotton, a mixture of wood-meal and nitrate of potash, or of charcoal and nitrate of potash; in fact, by mixing with the gelatin two of the ingredients of ordinary gunpowder. Various names have been given to these mixtures, but whatever name may be given to the mixture it is in every case less powerful in blasting than the original gelatin. One good mixture consists of 80 per cent. of thin gelatin (95 per cent. of nitro-glycerin and 5 per cent. of nitro-cotton) and 20 per cent. of a mixture of wood-meal and nitrate of potash. The latter mixture consists of 16 parts of nitrate of potash and four parts of wood-meal or charcoal. The usual name given to this form of gelatin is gelatin-dynamite. Another useful mixture consists of 60 per cent. of thin gelatin (96 per cent. of nitro-glycerin and 4 per cent. of nitro-cotton) and 40 per cent. of the mixture of wood-meal and nitrate of potash (32 parts nitrate of potash and 8 parts of wood-meal or charcoal). The mixture with wood-meal and nitrate of potash, as I have explained above, lessens the energy of the explosive, and modifies its effects in blasting, so that the rock is broken into larger fragments than is the case when pure gelatin is used. At the same time, wood-meal and nitrate of potash, being cheaper than nitro-glycerin, render the cartridges less costly to the miner.

A little consideration will show that it would be comparatively easy to make many varieties of blasting-gelatin by mixtures, more or less effective, of thin gelatin, with ingredients either explosive or non-explosive; and, in fact, many attempts have been made by inventors at home and abroad to improve on blasting-gelatin by mixing it.

In making mixtures of blasting gelatin or nitro-glycerin, or nitro-cotton, with other ingredients, great attention must be paid to the chemical constituents of the mixtures. Blasting gelatin is, without doubt, the most perfect of explosives in this sense—that the carbon, hydrogen, and oxygen present are just in the exact proportions required to form carbonic acid and water, and thus develop, at the moment of explosion, the maximum of heat and expansion. In mixing this form of explosive, therefore, with other ingredients, the greatest care must be taken that the latter contain within themselves enough of oxygen for the carbon present to produce perfect combustion, otherwise there will be a loss of power—neither the mixture nor the gelatin producing good effect.

A great many inventors are in the field with smokeless powder. Up till now there are several hundred patents in connexion with this matter, but hitherto the success of the inventors has not been great. No powder that has yet been tried is absolutely smokeless; but what is meant generally, by a smokeless powder, is one in which the oxygen of the explosive combines perfectly with the carbon, to form carbonic acid, and with the hydrogen, to form water. If the ingredients are mixed, in chemically rational proportions, then the amount of oxygen present will just be such that the whole of the carbon and the hydrogen, when the explosion takes place, will combine with it, and form carbonic anhydride ( $\text{CO}_2$ ) and water ( $\text{H}_2\text{O}$ ). There will then be no smoke—for what we usually understand by smoke is the presence of small particles of uncombined carbon in the air. A smokeless powder that fulfils those conditions possesses great energy, seeing that the heat developed at the instant of explosion is the highest possible; and the gases formed, therefore, have the maximum of expansion. One of the best smokeless powders consists of a modification of blasting-gelatin, made by mixing it either with the ordinary gun-cotton, or with collodion cotton, to such an extent that the finished substance is dry and elastic.

Nobel's Patent Smokeless Powder, which he calls Balistite, is formed in that way. It usually consists of about 50 per cent. of nitro-glycerin and 50 per cent. of nitro-cotton. This substance is not powder in the ordinary sense—that is, a substance in a state of fine division. It is in the form of cakes, or squares, or cubes, or cords. It presents the appearance of elastic rubber, or vulcanite when rolled into a sheet. When set fire to it burns slowly with a yellow flame. When kindled in a confined space the gas developed does not escape, until it attains a certain pressure; and as it does this much more slowly than ordinary gunpowder, it can be used in guns with much effect, and without danger.

Another variety of smokeless powder, patented by Sir Frederick Abel and Professor Dewar, consists of nitro-glycerin and ordinary gun-cotton or nitro-cotton, with or without other ingredients—the combination of which is assisted by the use of acetone or other solvents. This material can be made in any form, but it is specially good in the form of thin cords or threads, which can be rolled up into cartridges of any size and any form, for small arms or for cannon. This smokeless powder has been adopted by Her Majesty's Government for use in the British Army and Navy, and I have no doubt it will, by-and-bye, become well known.

In making a smokeless powder, consisting of about 50 per cent. of nitro-glycerin, and 50 per cent. of nitro-cotton, the combination of the two explosives is assisted by the use of solvents. Various solvents, such as ether, acetone, &c. may be used, and the sensitiveness of the explosive mixture modified to any extent by adding a small portion of camphor to it—say, 2 to 5 per cent.; but in any case, the combination cannot be satisfactorily effected without the aid of heat. The heat may be obtained with safety from hot water.

As in the case of Nobel's patent for blasting gelatin, the inventor describes no machinery in the Balistite patent by which that substance can be made, but it is evident at a glance that to manufacture the material on a commercial scale, it will be impossible to avoid using machinery; and the manufacturer who attempts to work Nobel's patent balistite will have to devise machinery and plant for the purpose. That itself will demand considerable inventive talent. Machines to make this substance must be arranged to guard against the application of too high a temperature, and to avoid undue friction or pressure of the explosive—otherwise dangerous accidents may result.

For the manufacture of Cordite, which is the name given to the smokeless powder patented by Sir Frederick Abel and Professor Dewar, excellent machinery has been, or is being made at Woolwich Arsenal, which appears to answer all the conditions required for turning out readily and rapidly a supply of explosive cords which, from that form, may be made into cartridges of any shape and size.

From trials made on guns at Woolwich, it appears that this new smokeless powder, Cordite, gives, in rifled muskets, an initial velocity of about 2,000 ft. per second, while the

pressure within the barrel of the gun is less than that produced by the ordinary black powder. If these results can be maintained, there is a great future for this smokeless powder, and there can be little doubt that it will, by-and-bye, almost entirely supersede black powder for guns, both for the purposes of war and sport. It has the great advantage over ordinary gunpowder of not being spoiled by water, and that is a circumstance of much importance, because armies in the field using black powder for cartridges have to take great care to keep their ammunition dry. Such precaution will be unnecessary in the case of the new smokeless powder, which burns as readily under water as in the open air.

The following table of mortar trials is given to show the ballistic effect of various explosive mixtures. The mortar in which the trials have been made is suspended by arms 10 ft. in length, from a rigid framework. It is free to swing from the effect of the shot, and the length of its flight is registered automatically by an arrangement fixed to the mortar itself, so that in every case the angle through which it swings from the shot is accurately recorded. The weight of the mortar is known, and when the angle through which it swings is ascertained, sufficient data have been obtained to calculate the work done by the shot. The weight of the explosive, in every case, is 10 grms., and the work done is reduced to foot pounds. The 10 grm. charge of explosive is always ignited by a fuse and detonator. The latter contains from 7 to 8 grains of a mixture of chlorate of potash and fulminate of mercury.

#### ENERGY OF EXPLOSIVES.

Quantity of Explosive used in mortar — 10 grms.

Name of Explosive.	Ingredients.	Per-centage.	Energy in Foot Pounds.
1. No. 1 dynamite	Kieselguhr .....	25	920
	Nitro-glycerin .....	75	
2. No. 1 blasting gelatin.....	Nitro-glycerin .....	92	1,410
	Nitro-cotton .....	8	
3. Nitro-glycerin.	Nitro-glycerin .....	100	1,266
4. Gelatin dynamite .....	Nitro-glycerin, 92·3 per cent.....	80	1,213
	Nitro-cotton, 7·7 per cent.		
	Nitrate of potash .....	14	
	Wood meal .....	6	
5. Gelignite.....	Nitro-glycerin, 91·85 per cent.....	60	1,013
	Nitro-cotton, 5·15 per cent.....		
	Nitrate of potash .....	28	
	Wood meal .....	12	
6. Detonator mixture .....	Chlorate of potash .....	20	536
	Fulminate of mercury ..	80	
7. Fulminate of mercury .....	Fulminate of mercury ...	100	327
8. Ammonia powder .....	Nitrate of ammonia.....	91·28	790
	Wood meal .....	5·72	
	Sulphur .....	3·00	
9. Nitrate of ammonia powder .	Nitrate of ammonia.....	60·13	889
	Picric acid.....	30·57	
10. Nitrate of potash and picric powder ..	Nitrate of potash .....	53·55	650
	Picric acid.....	46·55	
11. Nitrate of soda and picric powder .....	Nitrate of soda .....	72·13	560
	Picric acid .....	27·87	

ENERGY OF EXPLOSIVES—*continued*.

Name of Explosive.	Ingredients.	Per-centage.	Energy in Foot Pounds.
12. Nitrate of soda and pierie powder .....	{ Nitrate of soda..... Pierie acid.....	{ 49'12" 50'88"	680
13. Nitrate of soda and pierie powder .....	{ Nitrate of soda..... Pierie acid.....	{ 41'59" 55'41"	670
14. Nitrate of ammonia and ferrocyanide of potassium .....	{ Nitrate of ammonia..... Ferrocyanide of potassium	{ 72'29" 27'71"	350
15. "Securite" .....	{ Nitrate of ammonia..... Dinitrobenzol .....	{ 82'64" 17'36"	970
16. Nitrate of potash and dinitrobenzol .....	{ Nitrate of potash .....	66'79"	550
	{ Dinitrobenzol.....	33'21"	
17. Chlorate of potash and dinitrobenzol .....	{ Chlorate of potash .....	70'86"	815
	{ Dinitrobenzol.....	29'14"	
18. Chlorate of potash and paraffin.....	{ Chlorate of potash .....	89'40"	670
	{ Paraffin.....	20'60"	
19. Chlorate of potash and nitro-cotton .....	{ Chlorate of potash .....	53'16"	920
	{ Nitro-cotton .....	46'84"	
20. Dinitro-cotton.....	Dinitro-cotton .....	100	550
21. Maxim's powder .....	{ Nitrate of potash .....	78'18"	200
	{ Sulphur .....	10'40"	
	{ Paraffin.....	11'42"	
22. Gunpowder ...	Gunpowder $\text{RLG}^2$ A .....	..	335
23. Gunpowder ...	Gunpowder $\text{RLG}^2$ D .....	..	347
24. Gunpowder ...	Gunpowder $\text{RLG}^2$ F .....	..	362
25. Gunpowder ...	Gunpowder, Brown, C II .....	..	174
26. Vril powder (blasting).	{ Ferrocyanide of potassium } Nitrate of potash .....	{ 82'06" 1'78"	496
	Solid paraffin .....	1'14"	
	Peroxide of iron.....	12'02"	
	Charcoal.....	82'00"	
27. "Roburite" .....	{ Dinitrobenzol..... Moisture, &c. ....	{ 16'7" 1'3"	950
	Gun-cotton .....	50'20"	
28. Tonite .....	{ Nitrate of barium..... Carbonate of soda..... Moisture.....	{ 47'40" 72" 1'54"	820
	Gun-cotton .....	59'60"	
29. "Potentite" ..	{ Nitrate of potash .....	56'80"	840
	{ Moisture .....	2'75"	
30. Gunpowder (for cannon) .....	Gunpowder.....	..	440

The mortar is by no means a scientifically accurate instrument, and does not measure or record the total work done by the explosive; but in that respect the trials are alike for every explosive, and the comparative results may be taken as fairly representing, in each case, the blasting value of the explosive tested.

Some of the mixtures given in this table are still in use as blasting agents, while others, although well known for a time to workers in mines and quarries, have, owing to various faults, either been given up entirely, or are now very little used in blasting.

An examination of the foregoing table will show the principle on which inventors of explosives go in making their mixtures. An explosive mixture usually consists of (1) a substance of itself explosive, such as nitro-glycerin or gun-cotton; (2) an oxidising agent; and (3) a carbonaceous substance to be oxidised. Sometimes the mixture consists merely of (1) an oxidising agent; and (2) of a carbonaceous substance. Such a mixture, some years ago, would not have been considered an explosive at all; indeed it can be exploded only by means of a powerful detonator, and so long as the detonators are kept apart from the mixture, it is quite safe. Even when set fire to, such mixtures will not explode. It will be observed from the table that the energy of blasting-gelatin is greater than that of nitro-glycerin. That is accounted for by the circumstance that nitro-glycerin contains a little more oxygen than is necessary for the complete combustion of itself, and that this oxygen is utilised by the carbon of the nitro-cotton contained in the blasting-gelatin. It will be observed also that the more rational mixtures are the more powerful. By rational is meant here that the ingredients of the mixture are taken in such proportions that the carbon and oxygen they contain will suit exactly to form carbonic acid at the instant of explosion. Generally speaking it may be asserted that nitro-glycerin mixtures are the best and most powerful for blasting, but they are liable to this objection, that in cold weather they become hard and frozen, and to be effective in blasting it is usually considered necessary to thaw them. For thawing them hot water pans or ovens are used, and as nitro-glycerin mixtures freeze at about 40° F., and do not readily thaw at even 10 degrees above that temperature, it may be stated that in this climate at least nitro-glycerin preparations are in a frozen state during six months of the year. However, nitro-glycerin preparations can be used in blasting as readily when frozen as when thawed, provided the miner has the necessary knowledge. Frozen dynamite is more difficult to explode than unfrozen dynamite, consequently a more powerful detonator is required to effect its explosion. That is all. But in blasting, frozen cartridges do not adapt themselves to the bore-hole so readily as the soft cartridges; but if the miner fills the hole with frozen cartridges, and then puts an ordinary detonator into a soft one, on the top of all, the explosion of the whole train will be readily effected. Frozen gelatin can be exploded easily enough by means of such a detonator as will explode the thawed material. A rifle bullet may be fired through frozen dynamite without exploding it; but a bullet from the same rifle, fired through unfrozen dynamite invariably sets it off. With blasting-gelatin the direct converse is the case. The rifle bullet passes through unfrozen gelatin without exploding it, but through frozen gelatin I have never found a bullet fail to set it off.

It will be noticed that the blasting energy of roburite and securite are nearly the same as that of No. 1 dynamite, and that the energy of tonite and potentite, which are gun-cotton mixtures, are also nearly the same. Tonite and potentite are not liable to freeze, and can, therefore, be used in winter as in summer. Tonite and potentite have a less specific gravity than dynamite and blasting-gelatin, so that a larger bulk of the former is required to do the same work as is done by a smaller bulk of the latter. In using tonite and potentite for blasting, therefore larger bore-holes are required than where nitro-glycerin preparations are used.

Securite and roburite have lately come into use as blasting agents. Those mixtures are perfectly safe when kept apart from the detonators, as they will not explode, even when set fire to, without the detonators.

To produce full explosive effect with roburite and securite, very powerful detonators are necessary.

## Communications.

## ON THE ESTIMATION OF GLYCEROL IN CRUDE GLYCERIN.

BY J. LEWKOWITSCH, PH.D., F.L.C., F.C.S.

IN a paper published by F. Filsinger, an abstract of which is to be found in this Journal, 1890, 421, objection is taken by the author to the correctness of Benedikt and Cantor's acetin method for the estimation of glycerol in commercial crude glycerin. About a year ago Filsinger published (in the Zeits. f. angew. Chemie, 1889, 3) eight analyses of a crude glycerin with the intention to show that the acetin method is far from giving concordant analyses, whereupon I proved (this Journal, 1889, 574), for various crude glycerins of different origin, that the results obtained by this method agree very closely. It is gratifying to note that in his last paper Filsinger has dropped this objection, and that his estimations of glycerol by the acetin method show a satisfactory agreement, but he still adheres to his previous opinion that by the permanganate method alone accurate results are arrived at, while the acetin method yields too high percentages for glycerol. A reason for this assertion is not given, for I for one, being practically acquainted with glycerin distilling, cannot consider the one adduced by Filsinger as a satisfactory one, viz., "that he was in a position to testify to the correctness of the permanganate method in several instances by the practical yields from the crude material employed." Fancy practical results in a works confirming the correctness of analytical methods!

As to the acetin method, it may be imagined, from a theoretical point of view, that the results could only be too low, as, judging by analogy, the process of etherification may reach a limit without being brought to an end. An acting of acetic anhydride on fatty acids (which I have recently shown takes place, Proceedings of the Chemical Society, 1890, 72) did not occur in the tests published by me, as the soap ley glycerins examined by me did not contain any. Even the "polyglycerols," although they will hardly be met with in ordinary (unadulterated) commercial glycerins, are not readily acetylated, as I convinced myself by preparing them; in fact, the acetin method allows of an estimation of any glycerol retained in these polyglycerols.

It will therefore be incumbent on Filsinger to show some other reason why the permanganate method should give more correct results, and especially to prove that no oxidation to carbon dioxide has taken place. As Filsinger takes only 0.3 gm. of crude glycerin in the permanganate test, minute errors are more likely to influence the result than in the acetin test, where 1.5—2 gm. are used.

## TABLE OF SPECIFIC GRAVITIES OF STRONG SULPHURIC ACID.

BY H. DROOP RICHMOND

(Second Chemist to the Egyptian Government).

IN reviewing the different tables of densities of strong sulphuric acid, one is struck with the want of agreement with each other that they show, amounting in some cases to .0045 in the density and nearly 1 per cent. in the strength of acid indicated.

The following table of specific gravities at 15° illustrates this well:—

Percentage.	Pickering.	Lunge and Naef.	Winkler.	Archbutt.
100	1.8384	1.8384	..	..
99.5	1.8404	1.8395	..	..
99	1.8417	1.8403	1.8400	..
98.5	1.8426	1.8407	1.8420	..
98	1.8430	1.8412	1.8427	..
97.5	1.8432	1.8413	..	1.8411
97	1.8430	1.8410	1.8430	1.8440
96	1.8420	1.8406	1.8428	1.8431
95	1.8401	1.8390	1.8427	1.8412
94	1.8374	1.8372	1.8418	1.8386
93	1.8341	1.8339	..	1.8354
92	1.8301	1.8294	..	1.8317
91	1.8255	1.8241	..	1.8268
90	1.8202	1.8185	..	1.8216

The recent work of Pickering (J. Chem. Soc. 128, 64—184) has placed at our disposal a series of determinations which is without doubt the most complete ever published, and from them I have calculated a table giving for each .0001 of specific gravity the corresponding percentages of sulphuric acid. My reasons for preferring the determinations of Pickering to all other observers are the precautions taken to observe accuracy, and the large number of determinations made, he having made nearly as many determinations as all other observers put together.

Pickering's determinations were made at four different temperatures, but it is from those at 17.925° that I have calculated my table, and I have reduced them to a temperature of 15° on the assumption that the expansion is regular between 8° and 18°, an assumption which Pickering's determinations show to be sensibly correct, and would not introduce any appreciable error into the table; I have calculated my table by the same method as used by Pickering, i.e., plotting out the densities against the percentages, taking the figures given in his paper (*loc. cit.*) and correcting them to 15°, drawing a curve and taking the figures from the curve; I have preferred to do this to taking my figures from the table of densities at 15° given by him, as I considered that all chance of error in the calculations would be eliminated; my figures on comparison agreed almost absolutely with those in his table, in no case was there a difference of more than .00002 in the density; the accuracy of the table then is beyond doubt; nowhere is there an error in the density affecting the fourth place of decimals, while except from 97 per cent. to 98 per cent. the error in the percentage will not exceed .02 per cent. at the outside.

The practical utility of the table is somewhat marred by the fact that the density reaches its maximum at 97.5 per cent., and after that slightly diminishes; but if an acid of whose strength, as indicated by the density, any doubt is entertained is diluted with about 4 or 5 per cent. of water, the table becomes as useful as before, and I have no doubt that taking the density will be the best method of determining the strength of strong sulphuric acid.

## TABLE OF SPECIFIC GRAVITIES AT 15° OF SULPHURIC ACID, 100 PER CENT. TO 90 PER CENT.

1.8384	100.00	1.8388	99.92	1.8392	99.84	1.8396	99.73
5	99.98	9	.90	3	.81	7	.70
6	.96	1.8390	.88	4	.78	8	.67
7	.94	1	.80	5	.76		.64



TABLE OF SPECIFIC GRAVITIES, ETC.—continued.

1'8400	99'61	1'8414	95'61	1'8364	93'68	1'8314	92'32
1	'58	3	'55	3	'65	3	'29
2	'55	2	'50	2	'62	2	'27
3	'52	1	'45	1	'59	1	'24
4	'49	0	'40	0	'56	0	'22
5	'46	1'8400	'35	1'8359	'53	1'8309	'19
6	'43	8	'30	8	'50	8	'17
7	'40	7	'25	7	'47	7	'15
8	'37	6	'21	6	'44	6	'12
9	'33	5	'16	5	'41	5	'10
1'8410	'29	4	'12	4	'38	4	'07
1	'25	3	'08	3	'35	3	'05
2	'22	2	'04	2	'32	2	'02
3	'19	1	'00	1	'29	1	'00
4	'16	0	91'36	0	'26	0	91'98
5	'11	1'8399	'02	1'8349	'23	1'8299	'05
6	'08	8	'88	8	'20	8	'03
7	'02	7	'84	7	'17	7	'01
8	98'98	6	'81	6	'14	6	'88
9	'94	5	'77	5	'12	5	'86
1'8420	'89	4	'73	4	'09	4	'84
1	'84	3	'69	3	'06	3	'81
2	'78	2	'65	2	'03	2	'78
3	'71	1	'61	1	'00	1	'76
4	'68	0	'57	0	92'98	0	'74
5	'56	1'8389	'53	1'8339	'05	1'8289	'72
6	'48	8	'49	8	'03	8	'70
7	'40	7	'46	7	'00	7	'68
8	'32	6	'42	6	'87	6	'65
9	'22	5	'38	5	'84	5	'63
1'8430	'08	4	'34	4	'82	4	'61
1	97'85	3	'31	3	'79	3	'59
2	'50	2	'27	2	'77	2	'56
3	'40	1	'24	1	'73	1	'54
4	96'93	0	'20	0	'71	0	'52
1'8429	'76	1'8379	'17	1'8329	'69	1'8279	'50
8	'65	8	'13	8	'66	8	'47
7	'55	7	'10	7	'63	7	'45
6	'46	6	'07	6	'61	6	'43
5	'39	5	'03	5	'59	5	'41
4	'31	4	'00	4	'56	4	'39
3	'24	3	95'97	3	'54	3	'37
2	'16	2	'93	2	'52	2	'35
1	'09	1	'90	1	'49	1	'32
0	'02	0	'87	0	'46	0	'30
1'8419	95'95	1'8369	'83	1'8319	'44	1'8269	'28
8	'88	8	'80	8	'41	8	'26
7	'81	7	'77	7	'39	7	'24
6	'74	6	'74	6	'37	6	'22
5	'67	5	'71	5	'34	5	'20

TABLE OF SPECIFIC GRAVITIES, ETC.—continued.

1'8264	91'18	1'8247	90'84	1'8231	90'53	1'8215	90'24
3	'16	6	'82	0	'51	4	'23
2	'14	5	'80	1'8229	'49	3	'20
1	'12	4	'78	8	'47	2	'18
0	'10	3	'76	7	'46	1	'17
1'8259	'08	2	'74	6	'44	0	'15
8	'06	1	'72	5	'42	1'8209	'13
7	'04	0	'70	4	'40	8	'11
6	'02	1'8239	'68	3	'38	7	'10
5	'00	8	'66	2	'37	6	'08
4	90'98	7	'64	1	'35	5	'06
3	'96	6	'62	0	'33	4	'04
2	'94	5	'60	1'8219	'31	3	'02
1	'92	4	'59	8	'29	2	'01
0	'90	3	'57	7	'28	1	89'99
1'8249	'88	2	'55	6	'26	0	'97
8	'86						

In this table all specific gravities are referred to water at 15°.

## Journal and Patent\* Literature.

### I.—GENERAL PLANT, APPARATUS, AND MACHINERY.

#### PATENTS.

*Improvements in Beaters employed in Machinery for Grinding and Pulverising Myrobalauns, Bark, Bone, Locust Beans, and other Animal, Vegetable, and Mineral Substances.* F. Firth and J. Whitehead, Dewsbury. Eng. Pat. 4151, March 14, 1889. 6d.

The improvement consists in forging the heads of revolving beaters for vegetable, and other substances, distinct from the arms, and then attaching them thereto in any suitable manner. By this method the cost is reduced, and the heads when worn can be reversed, or new ones can be substituted at a great saving of time.—E. S.

*Method of and Apparatus for Purifying Sea Water for Use in Steam Generators.* V. B. Lewes, Greenwich. Eng. Pat. 5339, March 28, 1889. 8d.

The object of the invention is to make sea water capable of being directly used for boiler feeding without the necessity of previous distillation, thereby obviating the injurious action

\* Any of these specifications may be obtained by post, by remitting the cost price, plus postage, to Mr. H. Reader Lack, Comptroller of the Patent Office, Southampton Buildings, Chancery Lane, London, W.C. The amount of postage may be calculated as follows:—

If the price does not exceed 8d. .... ½d.  
Above 8d., and not exceeding 1s. 6d. .... 1d.  
" 1s. 6d., " " 2s. 4d. .... 1½d.  
" 2s. 4d., " " 3s. 4d. .... 2d.

which distilled water has on the plates of steam generators. The inventor mixes with the sea-water a salt or compound, such as carbonate of soda or potash, capable of precipitating the calcium and magnesium salts contained in the water. He then treats the mixture to boiling in a closed vessel in which the precipitate can separate, whilst the liquid is passed through filters before it reaches the boiler. The claim is for the process or method as above indicated, and a drawing is added showing one arrangement of suitable apparatus.—B.

*Improvements in the Manufacture of Clear Ice and in Apparatus Employed therein.* W. L. Wise, London. From Escher, Wyss, and Company, Zurich, Switzerland. Eng. Pat. 5592, April 2, 1889. 8d.

THE usual means employed for producing clear ice consist in agitating the water to be frozen by means of internal contrivances, such as pegs, chains, rakes, and the like, which work within stationary freezing cells. These contrivances must of necessity be removed before the coagulation of the water commences, with the result that particles of air remain occluded in the water and cause the ice to become more or less opaque. The inventors overcome this difficulty by enclosing the water in moveable cells which are themselves caused to rock, to rotate or be otherwise agitated whilst immersed in the freezing mixture. The cells may be of cylindrical shape and may be made to turn on their axes either concentrically, eccentrically, or at an angle, and may be put in motion by any convenient gearing. There are three claims and two sheets of drawings.—B.

*Improvements in the Manufacture of Metallic Hampers or Skips for Carboys and other Articles.* H. Finch, Marple, and J. Mackenzie, Yarm-on-Tees. Eng. Pat. 6770, April 23, 1889. 8d.

THE hoop-iron, or steel forming the standards, instead of being flat, is fluted or corrugated, preferably in the form U, their heads only being kept flat; and are fixed in position by binding their flat ends over, under, and round the top and bottom hoops. To increase their strength the hoops are preferably curved both laterally and longitudinally in rolling, or they may be tubular. The invention can be adapted to skips and receptacles of various shapes, and for other purposes.—E. S.

*Improvements in Presses for Compressing Semi-Fluids into Solids, commonly known as Filter-Presses.* J. Hill, Stoke-on-Trent. Eng. Pat. 7087, April 29, 1889. 8d.

THIS invention refers to filter presses used for making potter's clay, or for compressing yeast, paraffin, or other semi-fluids, in which porous slabs are used as the filtering medium. The filter press is made in several sections, each section being formed of two or more porous slabs of suitable thickness, fixed in frames and bolted together side by side, so as to leave spaces between each set. The faces of the slabs are grooved to form passages for the liquid. The semi-fluid is forced into the spaces, the liquid passing through the slabs, whilst the solid matter is retained in the space. When the spaces are filled, the solid matter is removed, and the slabs are taken out and turned half round, so that the liquid in flowing through now clears the pores and prevents them being clogged. There are four claims and three sheets of drawings.—E. S.

*Improvements in and relating to Apparatus for Evaporating Sea and other Water for Obtaining a Supply of Fresh Water.* J. Newton, London, and D. A. Quiggin, Liverpool. Eng. Pat. 8139, May 16, 1889. 8d.

THIS is an improvement on Eng. Pat. 538 of 1889 (this Journal, 1890, 43) granted to the same inventors, the

object being to reduce "priming" in the evaporator, to facilitate the removal of scale on the heating surfaces of the feed-water heater, and to more easily keep a constant level of water in the evaporator. The first object is accomplished by inserting in the evaporating liquid vertical circulating tubes, open at both ends, and entirely immersed, thereby causing the hot water and vapour to ascend and the cold water to flow downwards with regularity. The removal of the scale is facilitated by attaching the feed-heating tubes to the cover of the heater box, and thus making them removable with the cover, for cleaning. A fixed level of the water is maintained in the evaporator by means of adjustable and self-acting floats and valves, for details of which, and of the other contrivances, the two sheets of drawings must be consulted. There are six claims.—B.

*Improvements in Brewers' and other Thermometers.* J. J. Hicks, London. Eng. Pat. 8184, May 16, 1889. 8d.

THESE improvements in brewers' thermometers refer to the shape of the surrounding case used for the retention of some of the liquor of which the temperature is to be taken, to self-acting means for clearing the scale of froth, to means for facilitating the reading of the temperature, for cleaning all parts after use, and to other details of construction or arrangement. The specification contains eight claims, and is accompanied with one sheet of drawings.—B.

*Certain Improvements in Filters.* E. M. Knight, San Mateo, U.S.A. Eng. Pat. 648, January 14, 1890. 6d.

THIS invention relates to those filters in which a fibrous or porous material is used as the filtering medium, and has for its object, (1), to prevent the leakage of the unfiltered into the filtered liquid; (2), to render the percolation of the liquid through the filter free and uniform; (3), to simplify and expedite the process of filtration; and (4), the cleansing of the filter and renewal of the filtering medium.

The filtering frame is made, preferably, of iron, and has a number of transverse bars to support the filtering medium, and is of such a thickness that when the sheets of asbestos cloth are stretched over each side there is a considerable space between them, which forms a chamber into which the liquid to be filtered flows. The asbestos cloth is covered with a paste of filtering medium, rubbed into the fibre of the cloth, and is further retained there by a wire netting, the whole being held together at the edges by clamping-plates and bolts, thus preventing the excess of any liquid to the interior chamber without first passing through the filter. The filter is cleaned by removing it from the frame and plunging it into the fire.—E. S.

*Improvements in Refrigerating Machinery.* T. B. Lightfoot, London. From C. Linde, Wiesbaden, Germany. Eng. Pat. 1875, February 4, 1890. 8d.

THESE improvements apply to refrigerating machinery in which cold is produced by evaporation of a volatile liquid, the vapours being then compressed by machinery and subsequently cooled in condensers, in order to act again by evaporation. The improvements mainly refer to means for counteracting the prejudicial effect of "dead spaces" in the compressing cylinders, and of want of tightness in valves and stuffing-boxes, which defects are more specially noticeable in cases of great differences of pressures and of temperatures existing between the evaporator and condenser.

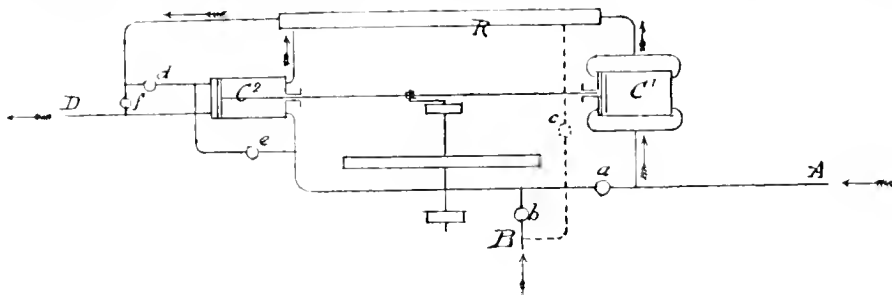
The method recommended is that of compressing and cooling by stages, the compressors being arranged in such a way that the vapours subjected to the higher stages of compression are operated upon in those ends of the cylinders only in which there are no stuffing-boxes.

One such compressing arrangement is shown in the accompanying figure, which also illustrates a method of combining the vapours from a second evaporator, that may

or may not be of higher pressure and temperature than those from the first.

In the figure  $C_1$  and  $C_2$  are double-acting compressing cylinders, both ends of  $C_1$  and the stuffing-box end of  $C_2$  being coupled by pipes to draw from the evaporator at A

and deliver into the receiver R, from which the other end of compressor  $C_2$  draws and finally delivers at increased pressure into the condenser at D. The low-stage compressors may also be made to draw from the second evaporator at B, or the vapours from the latter, if of higher pressure and



temperature than those from A, can pass directly into the receiver R, as shown by dotted lines. Other combinations may be made by a change in the pipes and valves. The specification shows other arrangements of compressors working by stages, also of cooling apparatus acting on the same system; and there are five claims.—B.

This shows a gain of 6 per cent. with hot air. The quantities of water evaporated during a week's trial with three boilers were, using hot air, 170,220 litres, and, using cold air, 151,868 litres, the consumption of coal being 21,900 kilos. in the former case and 20,700 kilos. in the latter. The average temperature of the air leaving the chamber was 53.7°, but as it afterwards passes under the bottom-flue its temperature on arrival at the fire-bars must be much higher, but as yet it has not been determined; the average temperature of the cold air was 20.9°; the average pressure in the boilers was 4.25 kilos. using hot air, and 4.30 kilos. using cold air, and in the former case 1.8 per cent. less cinders were produced. In a second experiment, made with the same coal and under similar conditions, but with three other boilers, the following average results were obtained:—

## II.—FUEL, GAS, AND LIGHT.

*The Influence of the Temperature of the Air supplied to Boilers in the Yield of Steam.* Poupardin. Bull. Soc. Ind. Mulhouse, 1889, 615—619.

THE results obtained by Meunier and Scheurer-Kestner showing the different yield of steam in summer and winter from the same coal, led the author to think that besides the cooling of the mass and loss by radiation, the temperature of the air-supply was a factor of importance. A preliminary experiment, made by conducting air through a wooden tube from the top of the boiler to the furnace, showed that more steam was produced than when the air was taken through a similar tube from the front of the boiler. The erection of a set of six new boilers at their Mer-Rouge works by the firm of *Schlumberger Fils* gave the author an opportunity of making a thorough trial with heated air. The flues and masonry were constructed to allow a system of air-pipes to be fixed somewhat after the fashion of a Green's economiser. The air-pipes were placed upright and left open at the top for the admission of air, communicating below with a chamber, with which was connected a pipe running at the back of the boilers, which in turn supplied pipes leading under the boilers to the furnaces. Arrangements were made for shutting off the hot air when desired, and using cold air in the ordinary way. The boilers have been tested by a year's working, and an increased yield of steam has invariably been obtained when hot air was used, as may be seen in the following example:—

	Gross Yield of Steam per Kilo. of Coal consumed.	Yield of Steam, Weight of Cinders deducted.
With hot air.....	Kilos. 7.77	Kilos. 8.95
With cold air.....	7.33	8.63
Gain with hot air.....	0.44	0.32

	Gross Yield of Steam per Kilo. of Coal consumed.	Yield of Steam, Weight of Cinders deducted.
With hot air.....	Kilos. 8.70	Kilos. 10.98
With cold air.....	8.09	9.34
Gain with hot air.....	0.61	0.64

This represents a gross gain of 7.5 per cent. of steam or 6.8 per cent. after deduction of the weight of cinders has been made. The average temperature of the air leaving the chamber was, in this experiment, 49.1°, that of the cold air being 24°; the average steam-pressures were, with hot air, 4.30 kilos.; with cold, 4.26 kilos.; the quantities of water evaporated were 175,810 litres with hot air, and 177,370 litres with cold, the consumption of coal being, with hot air, 20,300 kilos., and 21,900 kilos. with cold; the temperature of the feed-water was 35° in the case of the hot air experiment and 39.1° in the other case; using hot air there were contained in the steam  $(606.5 + 0.305 \times 155 - 35) \times 8.7 = 5,382$  heat units, the temperature of the steam being in each case about 155°, that of the feed-water in this case 35°, and the gross yield of steam 8.7 kilos. per kilo. of coal burnt; using cold air there were contained in the steam  $(606.5 + 0.305 \times 155 - 39.2) \times 8.09 = 4,979$  heat units, there being thus a gain of 411 heat units per kilo. of coal consumed with hot air. The specific heat of air being 0.2377, and the difference of temperature in the air-supplies being  $49 - 21 = 28^\circ$ , each kilo. of hot air has brought with it  $0.2377 \times 25 = 5.94$  heat units. Had the 411 heat units gained been brought in the hot air they would thus require  $\frac{411}{5.94} = 69.2$  kilos. of air or 53 cubic metres at 0°, whereas only about 15 cubic metres are calculated for each kilo. of coal.

The gain realised with hot air compares well with that realised with a Green's economiser. Taking the second experiment again, when the air was heated, 175,810 litres of

water entered the economiser at  $35^{\circ}8'$ , leaving the same at  $100^{\circ}5'$  on the average, representing an increase of heat of  $175,810 (100^{\circ}5' - 35^{\circ}8') = 11,374,907$  units; with cold air  $177,370$  litres entered at an average temperature of  $39^{\circ}2'$ , leaving at  $98^{\circ}1'$ , representing a gain of  $10,447,093$  heat units. By the use of hot air, as shown above, there was a gain of  $411$  heat units per kilo. of coal burnt, or for  $20,200$  kilos.  $8,302,200$  heat units.

It is pointed out that these results must not be taken as conclusive, as it remains to be seen whether the arrangements adopted, size of the pipes, chamber, &c., are the best, the author intimating his intention to resume his experiments in this direction.—E. B.

#### *The Spontaneous Ignition of Coal.* J. Gas Lighting, 55, 645.

At the Thirty-first Session of the Institution of Naval Architects, recently held at the Society of Arts, Adelphi, a paper on the above subject was read by Professor Vivian B. Lewes.

The author began by referring to the appointment of a Royal Commission in 1875, on the proposition of the Board of Trade and a Committee of Lloyds, to investigate the causes of the spontaneous ignition of coal cargoes, which had entailed serious loss of life and destruction of property. The Commission was assisted by Dr. Percy and Mr. (now Sir Frederick) Abel; and the result of their labours was the production of a report containing much sound advice for the guidance of persons employed in the shipment of coal. Nevertheless, accidents from the spontaneous ignition of cargoes continued; and between 1875 and 1883, no less than 57 coal-laden vessels were in consequence known to have been lost, while during the same period 328 were missing from unknown causes, though probably a large proportion of these losses were due to the one mentioned. In view of this fact, it was suggested to the author that an inquiry into the causes and possible prevention of this serious evil was greatly needed in the Royal Navy as well as in the merchant service; and therefore he undertook it, and laid the results before the members of the Institution.

The author proceeded to point out that, besides the carbon and hydrocarbons, coal contains certain mineral substances, as sulphate of lime or gypsum, silica and alumina, whilst in nearly all kinds there is to be found disulphide of iron and coal "brasses," or pyrites. Of these constituents, those that are likely to produce heat and cause spontaneous ignition in newly-won coal, are the carbon, hydrocarbons and brasses, which when they come into contact with air and moisture, give rise to certain chemical actions. He then described at length the influence of each of these constituents in the production of heat. Taking carbon first, he remarked that it is one of those substances which possess to an extraordinary degree, the power of attracting and condensing gases upon their surfaces—the power varying with the state of division and density of the particular form of carbon used. In the case of newly-won coals, the least absorptive would, he said, take up  $1\frac{1}{4}$  times, while others would absorb three times their own volume of oxygen. This absorption is very rapid at first, but gradually decreases; and it is influenced to a very great extent by temperature. Air-dried coal absorbs oxygen more quickly than wet coal.

Dealing in the next place with the action of the bituminous constituents of coal in causing spontaneous ignition, the author explained that all coal contains a certain percentage of hydrogen, which is in combination with some of the carbon, and also with the nitrogen and oxygen, and forms with them the volatile matter in the coal. The amount present in this condition varies very largely; being very small in anthracite and very great in cannel and shale. When the carbon of the coal absorbs oxygen, the compressed gas becomes very chemically active, and soon commences to combine with the carbon and hydrogen of the bituminous portions, converting them into carbon dioxide and water. This chemical activity increases rapidly with rise of temperature; so that the heat generated by the absorption of the oxygen causes the latter to rapidly enter into chemical combination. Chemical combination of this kind—*i.e.*,

oxidation—is always accompanied by evolution of heat; and this further rise of temperature again increases rapidity of oxidation, so that a steady rise of temperature is set up. This taking place in the centre of a heap of small coal, which, from the air and other gases enclosed in its interstices, is an admirable non-conductor of heat, will often cause such heating of the mass that if air can percolate slowly into the heap in sufficient quantity to supply the necessary percentage of oxygen for the continuance of the action, the igniting-point of the coal would be soon reached. It has been suggested that very bituminous coal (such as cannel, shale, and coals containing schist) is liable to spontaneous ignition from the fact that a rise in temperature would cause heavy oils to exude from them, which, by undergoing oxidation, might cause rapid heating. Experiment, however, not only shows that this is not the case, but that the heavy mineral oils have a remarkable influence in retarding heating.

The action of iron disulphide, pyrites, or "brasses," in promoting spontaneous ignition, was next discussed. The author said that ever since Berzelius first expressed the opinion that the heat given out by the oxidation of iron disulphide into sulphates of iron might have an important bearing on the heating and ignition of coal, it had been adopted as the popular explanation of that phenomenon; and although the work of Richters clearly proved this not to be the case, the old explanation was still given. Dr. Percy, in 1864, pointed out that probably oxidation of the coal had also something to do with spontaneous combustion—a prediction amply verified by Richters' researches some six years later. This disulphide of iron was found in coal in several different forms—sometimes as a dark powder distributed throughout the mass of the coal, and scarcely to be distinguished from coal itself. In larger quantities it often formed thin golden-looking layers in the cleavage of the coal, whilst it sometimes occurred as large masses and veins, often from 1 in. to 2 in. in thickness; but inasmuch as these masses of pyrites were very heavy, they rarely found their way into the screened coal for shipment, many hundreds of tons of these brasses being annually picked out from the coal at the pit's mouth, and utilised in various manufacturing processes. When the air was dry, the pyrites underwent but little change at ordinary temperatures. In moist air, however, they rapidly oxidised when in a finely-divided condition—the first action being the formation of ferrous sulphate and sulphur dioxide, together with the liberation of sulphur; the relative amounts of the two latter being regulated by the temperature and the supply of air, whilst longer contact with moist air converted the ferrous sulphate into a basic ferric sulphate generally termed "misy." It was during this process of oxidation that the heat supposed to cause the ignition was evolved. But when it was considered that some of the coals most prone to spontaneous combustion contained only 0.8 per cent. of iron pyrites, and rarely more than 1.25 per cent., the absurdity of imagining this to be the only cause of ignition became manifest. If 100 lb. of coal were taken, and the whole of the pyrites in it concentrated in one spot and rapidly oxidized to sulphate, the temperature would barely be raised to  $100^{\circ}$  C. if all loss of heat could be avoided. The author had carefully determined the igniting-points of various kinds of coal, and found them to be as follows:—

Cannel.....	$698^{\circ}$ F. = $370^{\circ}$ C.
Hartlepool.....	$766^{\circ}$ F. = $408^{\circ}$ C.
Lignite.....	$842^{\circ}$ F. = $450^{\circ}$ C.
Welsh steam.....	$870^{\circ}$ F. = $477^{\circ}$ C.

So that no stretch of imagination could endow the small trace of pyrites scattered through a large mass of coal, and undergoing slow oxidation, with the power of reaching the needful temperature. Richters fully realised this point, and discarded the idea of the pyrites doing anything more than adding their mite to the causes which bring about rise of temperature. In this, however, Lewes thought Richters was mistaken, his own experiments pointing to the fact that they might increase the liability to ignition when present in large quantities, and do so by liberating sulphur under certain conditions. A still more important part played by the pyrites, is that as they become oxidised to ferrous sulphate they swell in size, and so tend to split up the coal

into small pieces, and, by exposing a large extent of fresh surface to the air, cause increase of temperature and energetic chemical action.

The author, Lewes, then traced the actions which culminate in ignition. He said that on newly-won coal being brought to the mouth of the pit, it at once began, by virtue of its surface action, to absorb oxygen from the air. Unless piled in unusually large heaps, and a good deal broken, it did not however, as a rule, show signs of heating, as the exposed surface was comparatively small; and the air, finding its way freely between the lumps, kept down the temperature. The coal was now screened, and the obtrusively large lumps of brasses picked out. It was then put into trucks, and enjoyed the disintegrating processes of joltings and shuntings innumerable—every jar adding to the percentage of small coal present, and a corresponding increase in the size of the surface exposed to the air. Arrived at the docks, it had to be transferred from the truck to the ship, which was done by one of the numerous forms of tips, shoots, or spouts employed for the purpose; and it was during this operation that more harm was done than at any other period. The coal first shot into the vessel was, by reason of the distance which it had to fall, broken down into small lumps, and, having to bear the impact of the succeeding load falling upon it from a height, rapidly became powdered into slack; whilst the succeeding loads, falling in on the cone so formed, became more or less broken down, so that by the time the cargo was all taken in, a dense mass of small coal was to be found under the hatchway. It was invariably at this point that heating took place, as the large surface exposed fresh to the air by the breaking down of the coal caused rapid absorption of oxygen, and consequent rise of temperature. This set up chemical combination between the oxygen absorbed by the coal and the hydrocarbons and coal "brasses." The combination of the "brasses" with oxygen caused the swelling of the oxidised mass and the splitting up of the coal. Fresh surfaces were exposed, and more absorption of oxygen took place; and the igniting-point of the sulphur vapour and sulphur compounds distilled out of the pyrites was reached, and rapidly raised the temperature to the igniting-point of the coal. It was only, however, in cases where large quantities of dense coal brasses were present, that this action could occur. On examining the evidence to be obtained as to the conditions under which spontaneous ignition of coal in ships usually takes place, Lewes found that the liability to such accidents increases with (1) the tonnage of the cargoes, (2) the ports to which shipments are made, (3) the kind of coal of which the cargo consists, (4) the size of the coal, (5) the shipping of coal rich in pyrites while wet, (6) the ventilation of the cargo, and (7) the rise in temperature in steam colliers due to the introduction of triple-expansion engines and high-pressure boilers.

Having discussed the chemical and physical conditions which lead to the spontaneous ignition of coal, the author proceeded to formulate precautions tending to prevent such disasters. These had reference to the choice of coal for shipment to distant ports, and to the precautions to be taken during shipment and when the coal is on board. He advised that the coal should be as large, free from dust, and have as little "smalls" as practicable. It was better as free from pyrites as possible, in order to prevent disintegration after shipment; and it should contain, when air-dried, not more than 3 per cent. of moisture. No coal should, he said, be shipped to distant ports until at least a month had elapsed after it was brought to the surface at the pit's mouth. Every precaution should be taken to prevent the breaking up of the coal while being taken on board, and on no account should any accumulation of fine coal be allowed under the hatchways. When possible, the coal should be shipped dry, as external wet, by producing oxidation of the pyrites, caused disintegration. When the coal had all been taken in, it should be battened down, and the hatches not opened again until the vessel reached her destination; the only ventilation allowable being a 2-inch pipe just inserted into the crown of each coal compartment, and led 12 feet up the nearest mast—the top being left open. This would be quite sufficient to allow free egress to any gases evolved by the coals, but would not allow undue

access of air. Into the body of the coal cargo itself would be screwed, at regular intervals of about 6 feet, iron pipes, closed at the bottom, and containing electrical alarm thermometers, set at a certain temperature, the attainment of which would be indicated by the ringing of a bell, which would not cease until the temperature had again fallen, and then the spot in which the heating had taken place would be indicated by an index-board. The existence of over-heating having been reported by the bell, and recorded by the index, the author offered suggestions for suppressing it. He advocated the injection of compressed carbonic acid gas, and explained his plan as follows:—The nozzle attached to the screw valve on the bottle of condensed gas would have a short metal nose-piece screwed on to it, the tube in which would be cast in solid, with an alloy of tin, lead, bismuth, and cadmium, which could be so made as to melt at exactly 200° F. (93° C.). The valve would then be opened, and the steel bottle buried in the coal during the process of loading. The temperature at which the fusible metal plug would melt is well above the temperature which could be reached by any legitimate cause, and would mean that active heating was going on in the coal. Under these conditions, the pressure in the steel cylinder would have reached something like 1,700 lb.; and the moment the plug melted, the whole contents of the bottle would be blown out of it into the surrounding coal, producing a large zone of intense cold, and cooling the whole of the surrounding mass to a comparatively low temperature. The action, moreover, would not stop here, as the cold, heavy gas would remain for some time in contact with the coal—diffusion taking place but slowly through the small exit-pipe. When coal has absorbed as much oxygen as it can, it still retains the power of taking in a considerable volume of carbonic acid gas; and when coal has heated, and then been rapidly quenched, the amount of gas so absorbed is very large indeed, and the inert gas so taken up remains in the pores of the coal, and prevents any further tendency to heating. Indeed, a coal which has once heated, if only to a slight degree, and has then cooled down, is perfectly harmless, and will not heat a second time. It is not by any means necessary to replace the whole of the air in the interstices of the coal with the gas, as a long series of experiments show that 60 per cent. of carbonic acid gas prevents the ignition of the pyrophoric substances. A hundred cubic feet of gas can be condensed in the liquid state in a steel cylinder 1 foot long and 3 inches in diameter; and it has been shown that a ton of coal contains air spaces equal to about 12 cubic feet. One of these cylinders would therefore have to be put in for every 8 tons of coal, and these would be distributed evenly through the cargo, and near the alarm thermometers, which would be set to ring a degree or two below the point at which the fusible plug would melt. The bell ringing in the captain's room would warn him heating was taking place, and the bell would continue to ring until the cylinder had discharged its contents, and had cooled down to a safe degree, so that the whole arrangement would be purely automatic, and yet the officers would know if everything was safe. If the precautions advocated were taken, no danger could arise until the arrival of the ship at her destination, and the commonest precautions would then suffice.

In conclusion, the author remarked that the question of preventing the heating and ignition of stores of coal on land and ready for use in bunkers, could not be met so well by the use of the liquid gas; and in these cases it would be found beneficial to dress the coals with a little tar or tar oil, which would close the pores, and to a great extent prevent oxidation. He believed this was advocated by Lachman about 1870. Crude petroleum in small quantities for this purpose would also be found valuable, for it had no tendency to oxidise itself, and lowered the tendency in other bodies, besides coating them and so preventing access of oxygen.

In the discussion which followed, Professor Elgar, referring to the statements in the early part of the paper that, between the years 1875 and 1883, no less than 328 coal-laden vessels were missing from unknown causes, and that a large percentage of these losses were undoubtedly due to spontaneous

ignition, argued that there were a number of other causes which might account for their disappearance; and he then gave some particulars as to the losses during the three years from 1881 to 1883. In that period there were 23 known losses of vessels from spontaneous combustion of coal, but only one of these was a steamer—the other 22 being sailing vessels, the larger number of which were built of wood. He thought that one cause of spontaneous combustion in wooden ships was the impossibility of keeping the holds dry, and to the coal becoming damp and generating heat. Under the American law, ships registered in the United States were obliged to be fitted with steam-pipes in their holds, for the purpose of dealing with fire.

*Coal in the South-East of England.* W. Whitaker. Soc. of Arts J. 1890, 543–553.

For many years the presence of an underground range of old formations at no enormous depth in the south-east of England has been thought to exist by geologists, although in this tract only newer rocks crop out to the surface. The possibility of coal measures to be found amongst these older rocks gave zest to inquiry. The formations which come to the surface in and around London belong to the lower part of the Tertiary group, and to the upper part of the Secondary group, viz., the Cretaceous series, so called from its containing the chalk which comes to the surface near London and is found beneath the Tertiary beds in deep wells. The author proceeds to state that the various underlying members of the Cretaceous series were found to rise up and to crop out on the south in the following order from beneath the chalk in the tract known as the Weald:—

1. Upper Greensand. 2. Gault. 3. Lower Greensand.
4. Weald Clay. 5. Hastings Beds.

The general structure of the south-east of England in section is represented by a gently waved line with a central crest (the Weald), and a hollow on either side (the London and the Hampshire basins), the line being from north to south. Mr. Godwin-Austen worked out the idea (based on various observations) that the Lower Cretaceous beds along the slight trough of the London basin were likely to be interrupted underground by the uprise of older formations, against the flanks of which some of the Cretaceous beds would be found to thin out.

Now, the author points out that there is on the Continent a set of coal-fields with other rocks older than the coal measures along a curved line from Dortmund and Elberfeld (Germany) by Aachen, Liege, Namur, Charleroi, Valenciennes, Douai, and Bethune towards Calais whence the line has now been traced to Dover by the successful experiment of Sir E. Watkins and Professor Dawkins. In the west of England are the coal-fields of South Wales and Bristol. In the eastern part of this continental tract the coal measures are mostly at the surface; in the western they are covered by newer formations. The same may be the case in England eastward from the coal measures which are at the surface, and it is not improbable that the coal discovered at Dover has, in some way or another, underground connections with the western coal-fields.

The above-mentioned theory of an underground uprise of older rocks has been verified by various borings for water in the London basin, the borings being from 797 to 1,239 ft. deep. In the majority of cases the Gault was found to be underlain at comparatively little depths by old rocks, viz., Jurassic, Lower Carboniferous, Devonian, and Silurian beds, and some beds of doubtful age (perhaps New or Old Red sandstone). The author thinks that the variety of formations thus found suggests the possibility of the presence of other rocks being proved in other places. Of other borings beyond this district the proving of coal measures beneath Jurassic and Triassic rocks at Burford in Oxfordshire is of interest, and the finding of Carboniferous and older rocks by borings in and near Northampton. Two borings are remarkable in the south-east of England: the Subwealden boring near Battle (1872–76) through the Upper Jurassic rocks to the Oxford clay, and the Dover boring for coal in February last. This last boring, at the foot of Shakespeare Cliff, first passes through the Lower Chalk, Gault and Lower

Greensand, then through a series of Jurassic beds, until it reaches coal measures at a depth of 1,160 ft.

All these experiences and experiments lead the author to the following views and conclusions which may serve as a guide for further work in connection with this important subject.

1. We are to a great extent acquainted with the nature of the formations which have to be pierced to reach coal measures, and we can estimate the thickness of some of these formations.

2. The uprise of older rocks in Belgium and France seems to be in underground connection with the like uprise in the west of England; and coal-basins, separated by older rocks, are likely to occur underground in the unexplored part of this region in a similar manner as in the proved parts.

3. In the proved parts the coal measures contain profitable beds of coal, and at workable depths; therefore the same may be supposed for the unknown parts in the south-east of England.

4. The sites of the coal-basins can only be found by experiment, and we must be prepared to find that not all experiments will be as successful as the first one.

5. The places of the previous borings which reached some of the older rocks, must be avoided, as it is unlikely to meet with coal there.

6. Some of the borings which have already been carried to a considerable depth, ought to be continued, e.g., at Chatham a boring has reached the Oxford clay, and judging by the northerly thinning of the Jurassic rocks (from the Weald), a few hundred feet more down might bring a decisive result. For a similar reason St. Margaret's may be recommended, where a boring exists that has reached the Gault.

7. The Lower Cretaceous (including the Wealden) and the Jurassic beds thicken vastly southward from the valley of the Thames, a fact which has to be considered as well as the obvious advantage of commencing at a low level, for it is unnecessary to pierce a hill if a valley is in close proximity.

8. Magnetic properties and geological characters of various districts seem to bear certain relations one to the other, as Professor's Rücker and Thorpe have shown. This might afford hints about places to be avoided in searching for coal measures underground.

9. All the deep exploration heretofore noticed, being made by boring, the author recommends shaft-sinking, especially in places where there is little to fear from water. The beds to be pierced are not hard, and it is obvious that a shaft affords much more conclusive proofs and better means of investigation (e.g., horizontal working) than a bore-hole.

The author concludes his paper by stating that the geological structure of a large tract of country points to hidden coal-fields, lying in the folds or at the flanks of older rocks, in the south-east of England. To ascertain their position, trial work (perhaps of great extent) on a large area has to be done, and success must be expected to alternate with failure. The finding of these coal-fields would add greatly to the national wealth.—H. S.

*Annual Report of Gas Inspector to the Senate and House of Representatives of the Commonwealth of Massachusetts.* January 1890.

The inspector, C. W. Hiuman, states that ten towns are supplied with a gas made from petroleum or some of its products. Some of these towns use the pure gas, but generally more or less air is mixed with it to render the gas less liable to smoke. This addition of air reduces the illuminating value of the gas, but only slightly for the amount of air usually added. The gas made from petroleum contains so little sulphur that it was not thought necessary to determine it, especially as only about one-half as much of this gas is required to produce a certain quantity of light as when common coal is used.

The legal maximum for carbonic oxide is 10 per cent. of the total volume of the gas; coal gas usually contains from 5 to 8 per cent., and water-gas, as usually made, from 25 to 30 per cent. of carbonic oxide. It is evident that about 15 per cent. of water-gas can be added to coal gas and the resulting mixture yet contain less than the legal maximum of carbonic oxide.

Several of the companies using water-gas applied, under an Act passed in 1888, for leave to manufacture gas containing more than 10 per cent. of carbonic oxide. At Amesbury, Athol, and Spencer the gas has been found on three consecutive inspections to contain more than 10 per cent. of carbonic oxide, and the companies have been notified that a fine was due; at Athol, some time in the past summer, coal gas was substituted for water-gas and has since been used. More than 10 per cent. of carbonic oxide has been found in the gas of several other companies.

Two samples of water-gas were found on analysis to have a low illuminating value, although they contained a high percentage of illuminants; the percentage of carbonic oxide was also low for water-gas. The degree of heat used in the manufacture of these gases seems to have been too high for the amount of oil used. It seems certain that the hydrocarbons in these two gases were of less illuminating value than an equal volume of the hydrocarbons in coal gas; and it also seems certain that marsh gas has some value as an illuminant, as is shown by the following fact:—Two samples of gas which were found to contain practically the same percentage (14·4) of illuminants had an illuminating power of 28·3 and 22·1 candles respectively; this difference was due to the fact that the one contained 30·54 per cent. of marsh gas and 9·15 per cent. of carbonic oxide, the other (the poorer) 17·18 per cent. of marsh gas and 21·74 per cent. of carbonic oxide, the quantities of the other constituents being practically the same in the two samples. A knowledge of the percentage of illuminants in a gas, if nothing else about its composition be known, is therefore, very little guide to a knowledge of the illuminating power of the gas.—F. S. K.

#### PATENTS.

*A Process and Apparatus for Preparing Hydrogen and Carbonic Oxide Gases.* G. W. Hart, London. Eng. Pat. 7741, May 9, 1889. 8d.

THE process described is continuous and consists of the following operations:—

I.—Carbon dioxide is generated from chalk or other carbonates by heat or by treatment with acid, and such gas is passed over heated carbon, whereby it is converted into carbonic oxide.

II.—Steam is passed through retorts containing heated scrap iron, whereby hydrogen is evolved and the iron is converted into oxide, the hydrogen being led away for use.

III.—The carbonic oxide produced in operations I. or IV. is passed over the iron oxide formed by operation II., whereby the oxide of iron is reduced to metal and the carbonic oxide is reconverted into carbon dioxide.

IV.—The carbon dioxide so produced is passed over heated carbon and reconverted into carbonic oxide, which is led into a gas-holder, though a portion may be used for operation III.—F. S. K.

*Improvements in the Method of and Apparatus for the Manufacture of Water-Gas.* G. Rose, A. Baird, and M. B. Baird, Glasgow. Eng. Pat. 7812. May 10, 1889. 8d.

A CLOSED, cast-iron retort, charged with coke or other carbonaceous material, mixed or not with iron scrap or nails, is heated to a high temperature by means of one or more flames from liquid fuel, and steam, superheated or not, is admitted into the centre of the retort, together with hydrocarbon, or other oil if desired, the gas generated being then passed into a cooling and cleaning apparatus. The steam is generated by means of a coil placed in the funnel or flue of the generator furnace.—F. S. K.

*Improvements in Compounds to be used for manufacturing Illuminating Gas, the Prevention of Stoppages in Ascension Pipes, and the Deposition of Naphthalene.* S. B. Darwin, Portsea. Eng. Pat. 8263, May 17, 1889. 4d.

THE objects of this invention are to utilise coal tar and breeze, together with shale, petroleum or paraffin oil, or coal or cannel tar oil, or other suitable oil, with or without the addition of lime, for preventing stoppages in ascension pipes and the deposition of "naphthalene," while the gas produced by the use of this mixture as an addition to coal is of superior quality.

A scoopfull, or other suitable quantity, of a mixture of 160 gallons of tar, 28 gallons of oil, and 96 bushels of breeze, moistened with water if too dry and with or without the addition of lime, is placed on the top of the coal either when charging, or the retort is opened about an hour before the coke is drawn, and the mixture laid on the top of the red-hot coke.

The proportions given above may be varied according to the materials employed, but the mixture should be sufficiently hard to keep from running, and so made that it is not liable to be set on fire by hot drags, &c.—F. S. K.

*Improvements in Fastenings for Retort Lids.* G. King, Beckton. Eng. Pat. 9375, June 5, 1889. 8d.

THE object of this invention is to provide the lid of a gas retort with fastenings which will make a tight joint between the lid and mouthpiece, and in which the working parts of the fittings are protected from dirt. The lid is supported by a cross-bar by means of a bolt passing through a lug on the door. At one end of the cross-bar is a hole in which fits an eccentric boss attached to the lever closing the door. The cross-bar and lever when in position on the mouthpiece are fitted between two hinge-lugs cast on one side of the mouthpiece, and are retained there by a pin. The lid, cross-bar, lever and pin being fitted together on the mouthpiece, are free to swing round the hinge pin, the bearing surfaces being protected from dirt by the lugs. A catch and rest are fitted to a catch-box cast on the other side of the mouthpiece, and are held in position by a pin. The rest is a fixture, while the catch is free to swing round the securing pin so far as is necessary to allow the catch to be engaged or disengaged from the end of the cross-bar. A modification of the above is shown on the two sheets of drawings accompanying the specification. There are four claims.—E. S.

*New or Improved Artificial Fuel.* W. W. Popplewell, London. From H. Corried, New York, U.S.A. Eng. Pat. 2611, February 18, 1890. 4d.

THE patentee's improved fuel consists essentially of a mixture of pulverised coal dust, coal, culm, coke, peat, straw, and like material, mixed with a binding substance obtained by concentrating the waste liquor of the sulphite cellulose process until it has about the consistency of a syrup.

The proportions of the wood lixivium and pulverised combustible material vary according to the concentration of the former and the nature of the latter. In the case of very rich coal 5–6 per cent. of the highly concentrated lixivium is added: if the coal be not very rich, 10 per cent.

Combustible bricks can also be made by first mixing the pulverised combustible material with tar or molten pitch, and the wood lixivium with chopped straw, and then combining these two mixtures.—F. S. K.



### III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

*The Sundsha Naphtha Region of the Northern Caucasus.*  
Chem. Zeit. 14, 159.

ALTHOUGH the occurrence of naphtha in the territory situated between the Sundsha river and the main mountain range of the Caucasus east of Wladikawkas has been known for some time, borings have only been commenced recently by a number of local capitalists, the initiative in this direction having been taken by General Panin. In 1888 the average daily yield of crude oil amounted to 150 puds per well; in 1889, after deepening, it rose to 300 puds, and at the present time, the depth of the wells having been still further increased, the yield may be put down at 600 puds. The crude oil has a colour ranging from dark brown to black. The specific gravity is 0.886, and on distillation the oil yields from 3 to 4 per cent. of petroleum spirit of 0.700 sp. gr. and from 20 to 25 per cent. of kerosene, which has a flashing point of 25°.—D. B.

*On the Refining of the Heavier Paraffin Oils of Lignite-Tar.* E. von Boyen. Chem. Zeit. 14, 267—269, 289—291.

MANY patents are being constantly secured for refining mineral oil; there is a fair amount of literature, also, upon the subject, but it is on the whole useless for practical men on account of the different nature of oils from various sources.

Of the class of very heavy mineral oils such as those of lignite-tar very little has been published. They differ in their chemical composition from Russian and Galician oils, and consequently in their methods of refining. They most resemble the heavy mineral oils from Scotch shale oils, yet as there are marked differences the same method of refining cannot be entirely adopted.

The following facts are chiefly from the personal observations of the author:—The neutral hydrocarbons of lignite-tar boiling above 300° and previously freed by treatment from crystalline paraffin, when treated with concentrated sulphuric acid and caustic soda and distilled, distil with a steady uninterrupted rise of the thermometer. The fractions, taken every 5°, are very similar. The specific gravity and viscosity of the oil increases with the boiling point, while the colour becomes darker and the smell weaker. If each fraction be repeatedly agitated with 50 per cent. of concentrated sulphuric acid until no further action takes place, only an unimportant percentage of the original oil is left, the remainder being converted into a black mass which rapidly collects at the bottom of the vessel, technically known as "acid-tar" (Säureharz). It consists of sulphonic acids which are soluble in water.

If only 3 per cent. of acid be used then the black mass obtained more resembles resin and is insoluble in water, further treatment of the oil with acid giving products soluble in water. The residual oil gives a milky emulsion on shaking with water, from which there separates on standing in a warm place for a few days a black oil of sp. gr. .900.

Fuming sulphuric acid has the same effect, and crystalline sulphonates can be prepared from the residue. Fuming nitric acid dissolves the oil, forming a deep red-coloured solution, from which on addition of water a hard amorphous resinous mass separates, easily soluble in hot alcohol, and which can be crystallised. These reactions disprove the pre-existence of paraffins. The oil on heating with potassium hydrate volatilises all but a small portion, which is left as a black humid mass. With potassium bichromate and sulphuric acid the oil becomes viscous and red coloured by energetic oxidation, and with potassium permanganate in alkaline solutions acetic acid is formed.

All these reactions show that this oil differs in its properties from the mineral oil yielded ready made from the earth; and they give also a hint as to the method of refining.

In the ordinary process of rectifying Russian and Galician oils, the treatment with sulphuric acid, in order to

remove the small quantity of aromatic and unsaturated fatty hydrocarbon, is carried out till the acid exerts little or no further action, but, from the behaviour of the paraffin oil from brown coal tar described above, it will be seen that these latter oils cannot be rectified in this manner.

The author does not treat of oils of specific gravity of less than .900, as these present no particular difficulty. An oil obtained on the large scale after separation of the paraffin and of sp. gr. .900 if distilled boils at 300°, but mostly boils at over 360°. The first of the distillate has a very strong smell; the later portion is milder. At the end of the distillation a yellow body very rich in sulphur comes over and clings to the wall of the condenser tube.

The smell and decomposition are not due to the atmosphere, but to the temperature of distillation, as the author obtained a similar result on distilling in a vacuum. Superheated steam introduced gives a better coloured product. The first distillate is citron coloured and the others strong yellow. In distilling the loss in specific gravity is not regular, but never exceeds 0.010, and more commonly is .005.

The author gives the results of a distillation of the heaviest oil, dark green and thick, of sp. gr. .945 at 14° R. On distilling by means of superheated steam with addition of 0.5 per cent. of caustic soda solution, the following fractions were obtained:—

	Kilos.	Specific Gravity at 14° R.
First runnings.....	224	0.881
Mixture .....	1,624	0.933
Red product I. ....	224	0.956
" " II. ....	140	0.968
" " III. ....	100	0.988
Total .....	2,312	0.935

The mixture on redistillation gave:—

	Specific Gravity at 14° R.	Viscosity (Engler) at 14° R.
Fraction I. ....	0.900	2.692
" II. ....	0.926	4.385
" III. ....	0.932	5.308
" IV. ....	0.935	5.770
" V. ....	0.937	6.923
" VI. ....	0.943	8.404
" VII. ....	0.956	11.535
Average.....	0.935	5.770

The oil, after treatment with sulphuric acid and caustic soda, can be bleached by exposure for one or two months to the direct rays of the sun. The oil must be dry and quite free from sulphonic acids. Blue or green glass is best for the bleaching bottles, yellow or dark-coloured glass is useless. Broad and shallow vessels are best for bleaching, glass carboys are also used. The shape is of considerable importance. The place should also be carefully chosen, so as to be moderately warm (20°—25°) and in a dry place and free from dust, and so that the direct rays of the sun fall upon the bleachers. Bleaching is much more rapid in the rays of the summer sun than in winter.

*The Refining Process.*—Of distillation in a vacuum the author's experience does not extend beyond the laboratory. Distillation with the aid of exhaust or superheated steam he regards as a questionable advantage, as it is difficult to separate the water condensed with the oil. He has not had experience of conversion of crystalline into amorphous

paraffin by distillation without steam. The process of distillation is valuable, indirectly, in refining as a means of separating the varieties of oil and fitting them for fresh acid treatment.

The acid treatment is important. Acid of 66° B. or fuming acid may be used. Of the former, 4 per cent. is the most which should be used at one treatment. The oil must be free from water, and the temperature should not exceed 20° or be below 10°. Agitation is best performed according to the Scotch method, viz., using an air blast.

The treatment of the partly refined yet dark non-transparent oil is accomplished by treatment with the requisite quantity of caustic soda, and distilling. The distillate after being cooled below 20° is agitated for 15 minutes with 3 per cent. of acid of 66° B. Care must be taken that the air blast is dry.

After settling an hour the acid tar has mostly subsided, and the oil is then treated with clay. Rich, extremely finely-ground potter's earth, free from water and organic matter, possesses in the highest degree the property of collecting and precipitating the black acid tar suspended in the oil. Poor clay is useless. The process is best done as follows: The separated tar being run off, the air blast is turned on, and 1 per cent. of potter's earth is scattered on the oil. After 15 minutes it is allowed to settle for an hour, and more earth, if required, added till the oil is clear.

The oil should be tested by smelling if free from sulphurous acid, and by agitation of a small portion with caustic soda. The oil is then treated with 1 to 2 per cent. of strong caustic soda solution warmed to 40°–60° and allowed to stand till clear, which takes, as a rule, 24 hours.

The acid process can then be repeated. For the second treatment 4 per cent. of sulphuric acid is used. The oil, after being treated with 3 per cent. of the earth, is then run to a tank and washed. The finished oil should be clear, yellow, and transparent.

Though the greater part of the sulphonic acids separate, some are left dissolved in the oil and form a milky emulsion on agitation with water, and if not removed decompose, giving an oil which cannot be bleached. They are removed by washing the oil with successive amounts of water and direct steam until the water is no longer milky. The oil can be recovered from this milky emulsion, but it is of very inferior quality. The oil is treated after the water is run off with 1–2 per cent. of caustic soda lye, settled, and bleached.

Oil which gives a strong emulsion and thus is darkened in colour by the long boiling, may receive a last treatment with 2 per cent. of fuming sulphuric acid for 10 minutes, settling, and treating with 2 per cent. of potter's earth till the oil is clear, and then transferring to a clean air-agitator and agitating with 1–2 per cent. of finely-powdered sodium carbonate until the smell of sulphurous acid disappears, the colour becomes citron yellow and strong frothing occurs. Two hours is the longest required for this neutralising. The oil is then settled and bleached.

This latter plan of using powdered alkali is the cheapest.

Excess of treatment with acid must be avoided or the oil will not bleach.

For storing oil, glass or lead vessels are best. Iron tends to colour the oil. In wooden casks the oil also darkens.

—D. A. S.

#### On Paraffin. B. Pawlewski. Ber. 23, 327–329.

THE paraffin employed for the purpose of this research was white ozokerite as found in commerce. It had a density of 0.917, melted at 64–65°, and solidified at 61°–63°. Analysis gave 84.86 per cent. of carbon and 15.02 per cent. of hydrogen. The following table gives the solubility in 100 parts of solvent at 18°–22°:—

	Per Cent.
Formic acid .....	0.013
Glacial acetic acid .....	0.060
Benzene (commercial) .....	1.990
Xylene (commercial) .....	4.000
Chloroform .....	2.420

The author has determined the freezing points of paraffin in glacial acetic acid, benzene, and paraxylene according to Raoult's method, and from the results which are tabulated in the original paper the following conclusions are drawn:—(1.) The maximum molecular weight of paraffin is represented by formulae ranging from  $C_{25}H_{50}$  to  $C_{27}H_{56}$ . (2.) In dilute solutions (not saturated) of benzene and paraxylene, paraffin shows at least twice this quantity, whilst (3.) in solutions which approximate saturation the maximum is probably represented by formulae ranging from  $(C_{25}H_{50})_4$  to  $(C_{27}H_{56})_4$ . From the properties exhibited by paraffin in various solvents it is inferred that it must be regarded as a colloidal substance, although with acetic acid it behaves like a crystalloid.—D. B.

#### PATENTS.

*Improvements in Coke Ovens, and in Methods of and Apparatus for Collecting and Utilising the Products of Combustion from such Coke Ovens.* A. M. Chambers; Thorncliffe, and T. Smith, Chapeltown. Eng. Pat. 7424, May 3, 1889. 8d.

AN improvement on the plant previously described in Eng. Pat. 7358 of 1885 (this Journal, 1886, 375.), by which the quality of the coke is improved, the coke obtained in larger pieces, and the products arising from the coking of the coal are more thoroughly and effectively collected and utilised.

The improvement consists in making an opening in the top of the coke oven so that the products of carbonisation are taken partly at the bottom and partly at the top; a more uniform heat is thus produced throughout the whole mass of the coal than is obtained by any other method of coking, and the whole of the volatile products have not to be forced through the coking coal.—F. S. K.

*Improvements in and Connected with Retorts for Distilling Shale or other Oil-yielding Minerals.* J. Armour, Edinburgh, and H. Armour, East Brich. Eng. Pat. 7634, May 7, 1889. 8d.

THE retorts, which are vertical, are arranged as usual in two rows, the space between the outer walls being divided by transverse partition walls so as to divide the retorts into sets of four or other convenient number. The distilling parts of the retorts are constructed entirely of firebrick or similar refractory material, and are heated by means of horizontal flues formed round them and communicating each with the next above it in a manner to cause the heating gases to pass completely through each horizontal flue in succession. At the tops of the retorts there are iron casings which serve as hoppers, the vaporised oil and other products being withdrawn through a pipe formed on or fixed to the bottom of each casing. The top casings serve for drying, and to some extent heating the minerals freshly charged into them at suitable intervals; and they are heated by a mixture of the heating gases from all the retort flues in a bench or from those of two or more sets of four retorts, these gases acting in a kind of top oven or flue space through which they may be directed in various ways.—F. S. K.

*Process for the Treatment of Vegetable Fibres by Means of Acid, Neutral, and Alkaline Residues of Naphtha Manufacture, or Specially Prepared Substances Analogous to such Residues.* V. Schevelin, Balakhna, and P. Mindovsky, Moscow, Russia. Eng. Pat. 8106, May 15, 1889. 6d.

WHEN treating mineral oils, or the products obtained by distillation therefrom, with sulphuric acid, and after the removal of the mineral oil, dark-coloured, tar-like "acid residues" are obtained. The mineral oil, thus purified, is treated with alkali (generally caustic soda) whereby "soda residues" of various colours and consistency are obtained. The soda and acid residues (which were hitherto comparatively useless) can be employed for working fibrous plants, such as flax, hemp, and the like. The residues can either be employed as they are produced at the factories, or they are prepared by the following method:—The mineral oil

distillates or the mineral oil itself is mixed with 5 to 12 per cent. by weight of sulphuric acid at 15° to 35° C. during one-half to two hours. The mixture has to stand for not less than two hours. The mineral oil that floats on the top is then separated from the acid residues below, neutralised with alkali and heated to 70° or 80° C. It is then allowed to stand again, when the soda residue will separate. The mineral oil separated from the soda residue and heated to from 35° to 90° C. is again treated with 15 to 20 per cent. of sulphuric acid with an addition of from 1 to 3 per cent. of chromic acid (or the corresponding quantity of potassium bichromate with sulphuric acid). There is then again produced an acid residue, while the oil separated from the same is again neutralised by alkali and heated, again obtaining soda residue. If this alternate treatment with acid and then with alkali be repeated several times, the greater part of the mineral oil product can be converted into the acid and soda residues, which was not possible with the treatment generally used at the factories. All acid residues contain free acid which is removed by repeated washing with hot water, or by treating with steam and then neutralising by an alkali. One part of the neutralised acid residues and nine parts of water are stirred together at 25° to 35° C., the mixture is allowed to stand and the oil floating on the solution removed. The soda residues resulting from the treatment of kerosene and other mineral illuminating oils, are employed directly in an aqueous solution containing not more than 10 per cent. of the residues. If they contain unconverted mineral oil, they are first washed with warm or hot water.

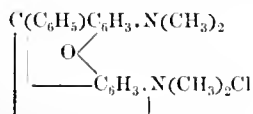
The fibres are separated from the woody matter as follows:—The fibrous plants are boiled from one-half to three hours at 70° to 95° C. in an aqueous solution containing not more than 10 per cent. of the residues, the reaction of which is alkaline, but can afterwards be rendered acid by adding sulphur dioxide, in order to obtain a lighter colour for the fibres, if required. The fibres are passed through pressing rollers, washed and brought into the drying chamber. After drying, they are broken and scutched, and the finished fibre passed through rollers. To obtain white flax or hemp, the fibres, after having been treated with the above-named alkaline solution, are introduced into a solution consisting of 2 parts of chloride of lime, 1 part of soda, and 60 parts of water where they are left for two or three hours, and further treated as above described.—H. S.

#### IV.—COLOURING MATTERS AND DYES.

*On Colouring Matters of the Benzene Group.* K. Henmann and H. Rey. Ber. 22, 3001—3005.

THE benzenees, obtained by the action of benzyl trichloride on phenols, resemble to a certain extent the phthaleins, but they are very weak colouring matters and of no value for technical purposes. As, however, the dimethyl-*m*-amidophenol-phthalein (rhodamine) has been proved to be a valuable colouring matter, the corresponding benzenee was prepared and is also a beautiful colouring matter, similar in strength, shade and fastness to rhodamine. The authors call this class of colouring matter "*Rosamines*."

*Tetramethylrosamine* is obtained by the action of benzyl-trichloride (1 mol.) on dimethylamidophenol (2 mol.) at 50°—60° C. As the reaction is very violent it is advisable to add sand or benzene as a diluting agent. The temperature is kept at 60° C., and the reaction completed on the water-bath. Impurities are removed by steam and the colouring matter is dissolved in water containing hydrochloric acid, and then precipitated by common salt. Its formula is—



Its salts are soluble in water and alcohol with a beautiful bluish-red colour and yellowish-red fluorescence. They dye wool, silk and jute from a pink to a dark bluish-red. The colours on silk or jute exhibit strong fluorescence, which is increased by treatment with sulphuric acid. Zinc dust reduces the rosamines, and the solutions remain colourless on standing, even with access of air. Addition of caustic soda in most cases does not produce an immediate precipitate. A sodium salt of the benzene seems to be formed. Extraction of the alkaline solution with benzene and evaporation of the latter yielded a product of the formula  $\text{C}_{24}\text{H}_{21}\text{N}_2(\text{OH})_3$ . The authors further prepared *tetra-ethylrosamine* and *diphenylrosamine*. The former is bluer than the corresponding methyl compound, the latter is a violet colouring matter, insoluble in water.—A. L.

*On Weselsky's Resorcinol Colouring Matters.* R. Nietzki, A. Dietze, and H. Mäckler. Ber. 22, 3020—3039.

WESELSKY obtained, by the action of nitric acid containing nitrous acid, two remarkable bodies, diazoresorcinol and diazoresorfinol. A bromine derivative of the latter compound is used as a dyestuff and sold under the name of "Fluorescent Blue" (this Journal, 1888, 315—316; also 1885, 50—51). Brummer and Krämer worked and published on the same subject, and their results are altogether different from those obtained by the discoverer of these compounds.

The authors have broken up this research and propose to call diazoresorfinol "*resorfinol*," and diazoresorcinol "*resazurinol*."

*Resazurinol* was prepared according to Weselsky and Benedikt's method by adding to a solution of 10 parts of resorcinol in 500 parts of ether, eight parts of red fuming nitric acid, and allowing the mixture to stand for two days. The only deviation from this method was that of cooling the resorcinol solution to from —5° to —8° C., and that of diluting the nitric acid with ether. The yield of crystals was 75 per cent. of the quantity of resorcinol taken. The purification was performed in the following manner: The mixture of Resazurinol and Resorfinol was washed with water and then triturated with a warm concentrated solution of sodium carbonate. After some time the product solidified to a mass of crystals of sodium resazurate, whilst the salt of resorfinol remained in solution. After filtering, the crystals are dissolved in hot water and sufficient concentrated soda solution added, to induce crystallisation to set in. The crystals are treated in the same way until perfectly pure. Sodium resazurate crystallises in long green needles, soluble in hot water, with difficulty in soda or solution of salt. The formula of Resazurinol is  $\text{C}_{12}\text{H}_7\text{NO}_4$ . It has the character of a weak base, forming salts with concentrated acids. With alkalis it acts as a mono-basic acid. It is rapidly decomposed by caustic alkalis, ammonia escaping. The barium salt,  $(\text{C}_{12}\text{H}_6\text{NO}_4)_2\text{Ba}$ , is with difficulty soluble in water.

*Resazurinol-ethylether* was prepared by treating silver resazurate with ethyl-iodide. Dark red needles are formed, melting at 215° C.

*Monacetylresazurinol* is obtained by heating sodium resazurate with dehydrated sodium acetate and acetic anhydride on the water-bath. Ruby-red needles, soluble in alcohol, and melting at 222° C. If the mixture of resazurin sodium acetate and acetic anhydride be boiled longer, acetylresorfinol is formed, and after very prolonged and energetic treatment products are obtained which cannot be crystallised.

*Tetrabromoresazurinol*, obtained as a blue colouring matter by Weselsky and Benedikt by the action of bromine on Resazurin. The authors prepared it by acidulating a mixture of sodium resazurate and an alkaline bromine solution. The sodium salt has the formula  $\text{C}_{12}\text{H}_2\text{Br}_4\text{NO}_4\text{Na} + 2\text{H}_2\text{O}$ . Reducing agents convert this non-fluorescent body into the fluorescent tetrabromoresorfinol. By heating resazurinol and acetyl chloride together an acetyl compound, containing chlorine, is formed, probably a mixture of different products.

*Resorufinol* is obtained from *Resazurinol* by various methods. For example, by heating the latter with concentrated sulphuric acid to  $210^{\circ}\text{C}$ .; by heating it with a bisulphite solution, or by reducing it with zinc dust or iron, and oxidising afterwards the product. It is distinguished by its beautiful pink colour and the magnificent fluorescence of its alkaline solution. *Resazurinol* and *resorufinol* yield the same reduction product, which, however, when re-oxidised in an alkaline solution, forms only *Resorufinol*. The analysis of the purified body agrees with the formula  $\text{C}_{12}\text{H}_6\text{N}_2\text{O}_3$ , a reduction product of *resazurinol*.

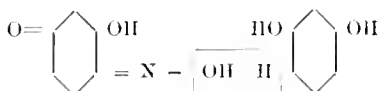
*Resorufinol-ethylether*, obtained by heating silver *resorufinate* with ethyl iodide and alcohol. It crystallises in orange needles, melting at  $225^{\circ}\text{C}$ . The formula is  $\text{C}_{12}\text{H}_{10}\text{N}_2\text{O}_3 \cdot \text{C}_2\text{H}_5$ . Krämer describes a product, which he calls "azoresorufinol-dimethylether," and which was obtained by heating *resorcinol* and *p*-nitrotoluene with concentrated sulphuric acid. This body is, however, identical with *resorufinol*, only of a not very pure character.

*Acetylresorufinol*,  $\text{C}_{12}\text{H}_6\text{N}_2\text{O}_3 \cdot \text{C}_2\text{H}_3\text{O}$ , prepared from *resorufinol* acetic anhydride and sodium acetate, crystallises from alcohol in long needles, melting at  $223^{\circ}\text{C}$ .

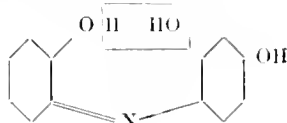
*Fluorescent Blue* is prepared according to Bindschedler and Busch's patent, by treating *Resorufinol* with bromine. The authors obtained it in the same way as *Tetrabromoresazurinol*. The sodium salt,  $\text{C}_{12}\text{H}_2\text{Br}_4\text{N}_2\text{O}_3\text{Na} + 2\text{H}_2\text{O}$ , crystallises in beautiful green needles.

*Hydroresorufinol* is formed when either *Resorufinol* or *Resazurinol* are heated with an acid stannous chloride solution. The compound crystallises in flat colourless needles, which, on dissolving in carbonates, reproduce *Resorufinol* on contact with air. It is doubtless the hydrochloride of a leuco base of *Resorufinol*. The body being heated for some time with sodium acetate and acetic anhydride forms the triacetyl derivative of a compound having two hydrogen atoms more than *Resorufinol*. It melts at  $216^{\circ}\text{C}$ ., and its formula is  $\text{C}_{12}\text{H}_6\text{N}_2\text{O}_3(\text{C}_2\text{H}_3\text{O})_3$ . Its molecular weight was estimated by Raoult's method and found to be 282, 306, 279, 303, whilst the formula demands 341. On distillation with zinc dust abundant quantities of diphenylamine are formed. On suspending *Resorufinol* in a strongly hydrochloric acid solution of hydroresorufinol, a hydrochloride crystallising in fine copper-coloured needles is formed, soluble in alcohol with a greenish-blue colour.

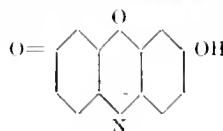
*Constitution of Resorufinol and Resazurinol*.—The *Resorufinol* contains three oxygen atoms, one of them as a hydroxyl group; the nitrogen is in form of trivalent nitrogen. Of all the methods of preparation the one which is based on the action of nitrosoresorcinol on *resorcinol* is the most interesting. According to Witt's, Meldola's, and Nietzi's researches, nitroso bodies act on amines or phenols in such a manner that the nitrogen of the nitroso group is joined to the benzene nucleus of the other molecule, oxygen being eliminated; in the para-position to the hydroxy- or amine-group. Thus indamines and indophenols are prepared. If molecular quantities of nitroso-resorcinol and *resorcinol* be dissolved in cold concentrated sulphuric acid, the mixture is coloured blue and dissolves in alkalis with a blue-violet colour. This cannot therefore be *Resorufinol*, which dissolves in concentrated sulphuric acid with a red colour. After prolonged heating to  $100^{\circ}\text{C}$ ., this body is converted into *Resorufinol*. It is, perhaps, the true indophenol of *resorcinol*—



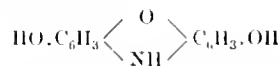
which, under the influence of sulphuric acid, changes into—



*Resorufinol* is therefore to be considered as—

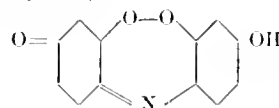


which, on reduction, yields—



hydroresorufinol or di-hydroxyphenoxazine. Further proofs for this theory are adduced through the medium of different new methods for preparing this body. It was obtained by the action of nitrosophenol on *resorcinol*, by nitrosoresorcinol on phenol, by oxidising *p*-amidophenol and *resorcinol* in sulphuric acid solution with manganese dioxide, &c. Amidoresorcinol and orthocresol yield an analogous body, but para-cresol does not. This confirms the author's hypothesis.

*Resazurinol* is possibly—



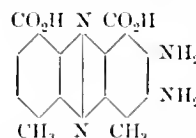
A. L.

#### On the Formation of Azines from Orthodiamines and Polyamines. R. Nietzki. Ber. 22, 3039—3040.

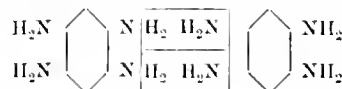
In a former treatise the author described the formation of tetramidophenazine from the symmetrical tetramidobenzene. Müller afterwards found that the unsymmetrical triamidobenzene yields in a similar way triamidophenazine. Fischer and Hepp proved the oxidation product of orthophenylenediamine to be a diamidophenazine, and Kehrman obtained from *o*-diamido-*p*-toluic acid—



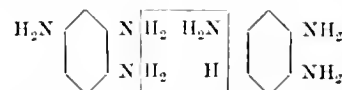
a diamido-azine, which must have the formula—



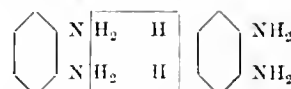
From these facts the author concludes that the same law which governs the action between paradiamines and monamines with a free para-position applies also to the orthodiamines, with the only difference, however, that in the case of ortho compounds the juncture is a double one. With Tetramidobenzene, the action is as follows:—



With Triamidobenzene—



and with *o*-Diamidobenzene—

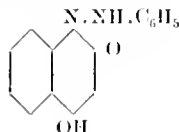


Meta-para-tolylenediamine,  $\text{C}_6\text{H}_3(\text{NH}_2)(\text{NH}_2)(\text{CH}_3)$  [1:2:4] cannot form an azine, as the para-position is not free.

—A. L.

On some Azo dyes from Naphthoresorcinol. St. v. Kostanecki. Ber. 22, 3163—3168.

ZINKE and Bindewald obtained by the action of phenylhydrazine on  $\alpha$ -naphthoquinone a product identical with phenylazo- $\alpha$ -naphthol. If phenylhydrazine acts in a similar way on hydroxy- $\alpha$ -naphthoquinone, phenylazonaphthoresorcinol ought to be formed. Zinke and Thelen, however, came to the conclusion that not this product, but in consequence of intramolecular changes,—



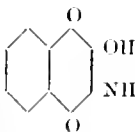
was formed.

The authors repeated Zinke's experiments and found the body to be phenylazonaphthoresorcinol, and not the hydrazide, which Zinke believed it to be.

The compound was prepared according to Zinke's method. It melted, however, at 218—220° C., and not at 228—230° C. as Zinke states.

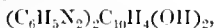
*Nitrosophenylazonaphthoresorcinol* is prepared by pouring an alkaline solution of phenylazonaphthoresorcinol and sodium nitrite into dilute hydrochloric acid. A brown floccular precipitate is formed, crystallising from dilute acetic acid in small bright brownish-red plates, soluble with difficulty in hot water and alcohol, easily in glacial acetic acid, and decomposing at 175° C. It is insoluble in alkalis, but soluble in concentrated sulphuric acid with a dark green colour. The compound dyes mordanted fibre, much more feebly, however, than nitrosophenylazoresorcinol.

*Reduction of Nitrosophenylazonaphthoresorcinol*.—On reduction with tin and hydrochloric acid amidonaphthalenic acid—



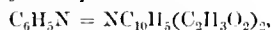
and aniline are formed.

#### *Phenyldisazonaphthoresorcinol*—



was obtained by acting with diazobenzene chloride on sodium phenylazonaphthoresorcinol. It forms, recrystallised from a mixture of chloroform and alcohol, long red needles, insoluble in caustic alkalis, decomposing whilst melting, at 225° C. On reduction, the same products are formed as from the nitroso-compound.

#### *Diacetylphenylazonaphthoresorcinol*—



If phenylazonaphthoresorcinol be boiled for some minutes with acetic anhydride and dehydrated sodium acetate, a diacetyl-compound is formed, crystallising from dilute alcohol in needles, melting at 122°—123° C.—A. L.

#### *On $\alpha$ -Naphthylamine and $\alpha$ -Naphthol- $\epsilon$ -disulphonic Acid.*

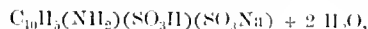
A. Bernthsen. Ber. 22, 3327—3335

$\alpha$ -NAPHTHYLAMINE- $\epsilon$ -DISULPHONIC acid is prepared, according to the patent of the Berlin "Actien Gesellschaft für Anilin fabrikation," by treating naphthalene with fuming sulphuric acid at the ordinary temperature. On nitrification of the sulphonic acids two isomeric nitro-, and from these by reduction two amidonaphthalene sulphonic acids are formed, which can be separated by crystallisation of their neutral sodium salts. The naphthylamine sulphonic acid which forms the more difficultly soluble sodium salt, is identical with the one patented by Schöllkopf. From the mother-liquor the acid sodium salt of the isomeric  $\epsilon$ -compound is precipitated by hydrochloric acid. Ewer and Pick

have applied for a patent to protect the preparation of a naphthylamine- $\alpha$ - $\beta$ -disulphonic acid, obtained from naphthalene- $\alpha$ - $\beta$ -disulphonic acid, which is identical with  $\alpha$ -naphthylamine- $\epsilon$ -disulphonic acid.

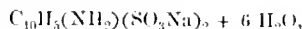
A third method forms the subject of an application of the Badische Anilin und Soda Fabrik. One part of naphthalene is converted into its disulphonic acid by heating it with five times its weight of concentrated sulphuric acid to from 80°—100° C., or by treating it first with ordinary concentrated acid at 90° C., and then with fuming acid at a temperature not higher than 120° C. The disulphonic acid is nitrated, and precipitated by common salt, and then reduced. The naphthylaminedisulphonic acid is then precipitated as acid sodium salt from its neutral salt solution by acids. It is identical with  $\alpha$ -naphthylamine- $\epsilon$ -disulphonic acid.

#### *The acid sodium salt—*



forms needles or thin prisms.

#### *The neutral sodium salt—*

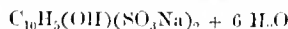


is very soluble in water and crystallises in long needles or prisms.

The *neutral barium salt* crystallises either with 3 mols. of water in light yellow needles, or with 4 mols. of water in colourless flat needles.

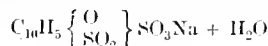
The *acid barium salt* forms microscopic needles containing 2½ mols. of water. The free acid crystallises with 3 mols. of water, and forms on treatment with nitrogen trioxide a very soluble diazo-compound acid, whilst on treatment with nitrites and sulphuric acid a difficultly-soluble diazo-sodium salt is obtained. If this diazo-compound be boiled with water containing sulphuric acid two very different bodies may, according to subsequent operations, be produced, viz., naphthol- $\epsilon$ -disulphonic acid and its "sultone" (Erdmann).

$\alpha$ -Naphthol- $\epsilon$ -disulphonic Acid is obtained by mixing the diazo-compound with water and boiling it with addition of some sulphuric acid. On treatment with lime and filtration from gypsum, a calcium salt is obtained, which is converted into the sodium salt by treatment with sodium carbonate. Long colourless prisms of the formula—

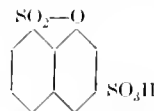


are formed, easily soluble in water. With diazo-compounds colouring matters are formed. The combination with diazobenzene is an orange yellow; with diazoxylene a scarlet; with  $\alpha$ -naphthylamine a red; and with benzidine a violet colour.

*Naphtholsultone Sulphonic Acid* ("Zeta-naphtholdisulphonic acid.")—If diazonaphthalenedisulphonic acid be boiled with water and sulphuric acid until the evolution of nitrogen is ended, and if the liquid be then rapidly cooled and not treated with calcium hydroxide, a crop of long fine needles is formed. The substance can be obtained pure by recrystallisation from hot water. Analysis gives figures for  $C_{10}H_5SO_3(SO_3Na) + 3 H_2O$ . Its constitution is—



and it must be considered as an intra-molecular anhydride; alkalis and carbonates convert it into a disulphonic acid. The author considers the compound to be—

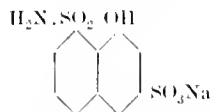


$\epsilon$ -Naphtholsultone sulphonic acid.

It forms azo colouring matters the same as those obtained from naphtholdisulphonic acid.

*Naphtholsulphamidesulphonic acid* is obtained by dissolving the sultone sulphonic acid in concentrated ammonia, and on addition of hydrochloric acid to the cold solution

the sodium salt of the new compound crystallises out in long needles. Its formula is  $C_{10}H_8NS_2O_6Na + H_2O$ , and its constitution—



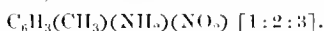
It forms colouring matters with diazo-compounds which are bluer than those obtained from the disulphonic acid.

—A. L.

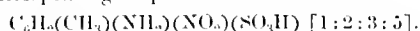
*On Nitrotoluidine Sulphonic Acids.* R. Nietzki and Pollini. Ber. **23**, 138—140.

ORTHOTOLUIDINE sulphonic acid, obtained by treating orthotoluidine with the calculated amount of sulphuric acid, was acetylated by heating its sodium salt with anhydrous acetic acid, and the acetyl compound was then nitrated in sulphuric acid solution. The temperature was kept below 5° C. The potash salt of the nitrotoluidine sulphonic acid was precipitated from its concentrated aqueous solution in orange needles by addition of an excess of caustic potash. The free sulphonic acid is precipitated from concentrated solutions of the potash salt by mineral acids.

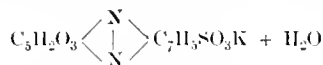
Another way to prepare the same compound is to treat *o*-acet-toluide with three times its weight of fuming sulphuric acid containing 15—20 per cent. of anhydrous sulphuric acid, on the water-bath, until completely soluble in alkalis, and to nitrate this sulphonic acid. The sulfo-group was eliminated by heating this body with dilute sulphuric acid in a sealed tube for three hours at 170°—180° C. On addition of ammonia a volatile base was obtained, which was carried over with steam. This is thus proved to be an ortho-compound, and has the formula—



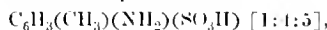
The corresponding sulphonic acid has the formula—



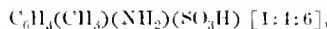
This latter body forms a difficultly soluble diazo-compound, which combines with phenols producing colouring matters. By careful reduction with stannous chloride a nitrohydrazine can be prepared. If the mixture of the nitrotoluidine sulphonic acid, stannous chloride and hydrochloric acid be heated, a diamidotoluene sulphonic acid is obtained, which crystallises from water in colourless needles. This acid is an ortho-diamido-compound forming azines with ortho-diketones as phenanthrenequinone or croconic acid. The potassium salt of the condensation product with the latter compound crystallises in dark greenish-black needles of the formula—



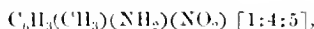
The para-toluidine sulphonic acids—



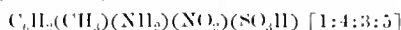
and



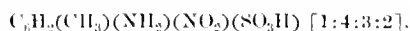
yield on analogous treatment as the above described ortho-compound, nitro-products which contain the nitro group in the ortho position with respect to the amido group. Both acids yield this same nitrotoluidine,—



melting at 114° C., and their constitution, therefore, is—



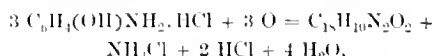
and



By reduction two diamido-compounds are obtained analogous in their reactions to the above described isomeric compound.—A. L.

*Triphenyldiazine as an Oxidation Product of Ortho-amidophenol.* P. Seidel. Ber. **23**, 182—189.

G. FISCHER obtained by oxidising an aqueous solution of ortho-amidophenol with red prussiate of potash a brown amorphous colouring matter and a body subliming in garnet-red crystals of the formula  $C_{21}H_{16}N_2O_2$ . The author improved the method of preparation by boiling a solution of ortho-amidophenol hydrochloride in 30—40 times its weight of water in a flask with reversed condenser, passing at the same time a current of air through the solution. The sole method of purification is to sublime the crude product. The analysis indicated the formula  $C_{18}H_{16}N_2O_2$ , and not, as Fischer found,  $C_{21}H_{16}N_2O_2$ . The formation of this body takes place according to the equation—



The colouring matter is nearly insoluble in water, alcohol, ether, acetone, carbon bisulphide, pyridine, benzene, &c., but exhibits in all these solvents, with the exception of water, a beautiful green fluorescence. Xylene, nitrobenzene, aniline, azobenzene, naphthalene, &c., are the best solvents for crystallising it from, and it is obtained in dark red crystals from these solvents.

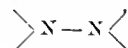
This colouring matter begins to sublime at 250° C., and forms at 300° C. a beautiful green vapour. Concentrated aqueous or alcoholic solutions of potash or soda are without action on this body. Anhydrous acetic acid does not produce an acetyl compound. Concentrated hydriodic acid reproduces at 170° C. ortho-amidophenol. Chromic acid in acetic acid solution even on boiling does not act upon it. Chlorine does not change it, but bromine forms substitution products. On treatment with sulphuric acid a sulphonic acid is obtained, forming alkaline salts soluble with difficulty in water. Nitric acid yields a mono-nitro-product, which yields an indigo blue colouration with concentrated sulphuric and hydrochloric acid. On reduction, an amido-compound is obtained, forming blue salts little soluble in water.

Hydrochloric acid forms a salt crystallising in dark green needles of the formula  $C_{18}H_{16}N_2O_2 \cdot 2 HCl$ .

The *Leuco-compound*,  $C_{18}H_{16}N_2O_2$ , is obtained by reducing the colouring matter with phenylhydrazine diluted with xylene. The reduction begins at 120° C., phenylhydrazine being decomposed into benzene, two nitrogen atoms and two hydrogen atoms, which are taken up by the colouring matter. The leuco-base is more easily soluble in the various solvents than the colouring matter. It can be crystallised from pyridine, to which some phenylhydrazine has to be added to prevent oxidation. It forms colourless plates, and the substance when heated to 250°—300° C. dissociates into hydrogen and the original colouring matter. On boiling the leuco-base with anhydrous glacial acetic acid a diacetyl compound,  $C_{18}H_{16}(C_2H_3O)_2N_2O_2$ , is formed, melting at 295° C. and crystallising in colourless plates. According to the author's views the formula of the colouring matter is—



It consequently contains the chromophoric group—



The colouring matter is therefore named *Triphenyldiazine*.

—A. L.

*On some Derivatives of Nitro-β-Naphthoquinone.* R. Zärtling. Ber. **23**, 175—180.

*Nitronaphthophenazine* was prepared by mixing a solution of 2 grms. of nitro-β-naphthoquinone in 40 cc. of glacial acetic acid with a solution of 1.8 grms. of *o*-phenylenediamine and 2.7 grms. of sodium acetate in 10 cc. of acetic acid of 50 per cent. strength. The colour of the mixture changes to orange, and a precipitate of a powdery nature

falls down. To complete the reaction the mixture was heated for some time on the water-bath. The dirty yellow substance formed was washed with glacial acetic acid and then recrystallised from a mixture of 4 parts of phenol and 20 parts of glacial acetic acid. It melts at  $221^{\circ}$ — $222^{\circ}$  C., forms prisms of greenish-yellow colour, is with difficulty soluble in alcohol, benzene, ether, and insoluble in petroleum spirit. Its formula is  $C_{10}H_5.NO_2.N_2.C_6H_4$ .

*Amidonaphthophenazine* was obtained by reducing 2 grms. of the nitro-compound with 25 cc. of alcoholic ammonium sulphide at  $100^{\circ}$  C. The yellow body is then changed into an orange one. It crystallises from aniline in small dark brownish-red crystals, which on being ground form a cherry-red powder, melting at  $191^{\circ}$  C. It is with difficulty soluble in alcohol and ether, but more easily in benzene. Its dark red ethereal solution is decolourised by hydrochloric acid, a hydrochloride being formed. *This is the first instance of a Eurlodine which forms colourless salts, whilst the base is itself coloured.* Its formula is—



Experiments with the object of producing an azonium base from orthophenylnaphthylenediamine and nitro- $\beta$ -naphthoquinone were unsuccessful.

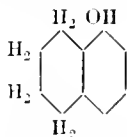
*Nitro- $\beta$ -hydroquinone* was obtained from the quinone by reduction with stannous acid. Groves' method of reducing it with stannous chloride and hydrochloric acid gives, however, far better yields. It melts at  $159.5^{\circ}$  C., and dyes fibres mordanted with metallic oxides. It forms with iron mordants a yellowish-brown, with chrome mordants a pale indigo blue, and with aluminium mordants a red. The colours are, however, not fast to soap.

Phenylhydrazine reduces nitro- $\beta$ -naphthoquinone to hydroquinone, phenylhydrazine forming benzene, azo-benzene, aniline and nitrogen.

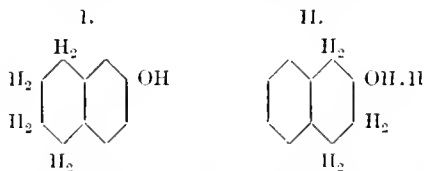
Hydroxylamine hydrochloride forms an addition product with the quinone  $C_{10}H_5.O_2.NO_2.NH_2.OH$ , melting at  $140^{\circ}$ — $141^{\circ}$  C., with evolution of gas, to a dark red liquid. It is very insoluble, and is decomposed on boiling with acetic acid or other solvents, the quinone being reduced to the hydroquinone.—A. L.

*On Alicyclic  $\beta$ -Tetrahydronaphthol and Secondary Ring Alcohols.* E. Bamberger and W. Lödter. Ber. **23**, 197—213.

The two naphthylamines form by the action of sodium and boiling amyl alcohol, bodies containing four atoms of hydrogen more than the original compounds. If they enter the substituted ring the resulting base belongs to the aliphatic series; if they enter the other ring they belong to the aromatic series. Alpha-naphthol is converted by the same treatment into a body of the formula—



The resulting compound is a true phenol, soluble in alkali, and forming colouring matters with diazo-compounds.  $\beta$ -naphthol forms two compounds richer by four hydrogen atoms—



No. I. differs from No. II. as a phenol from an alcohol. No. I. has all the properties of  $\beta$ -naphthol; No. II. presents no points of resemblance to it. It is a thick oil, insoluble in alkalis and not capable of forming colouring matters with diazo-compounds. It shows a great similarity in modes of reaction to menthol and borneol. It was prepared by

treating a boiling solution of 10 grms. of  $\beta$ -naphthol in 200 grms. of amyl alcohol with 20 grms. of sodium, which was gradually added in portions of 4—5 grms. The blue colour of the mixture soon changed to yellow. It is boiled until all sodium is dissolved, then it is poured hot into water. The reddish-brown layer of amyl alcohol is washed several times with caustic soda, then with water, and finally with dilute hydrochloric acid. It is then distilled under the ordinary pressure until the thermometer indicates  $150^{\circ}$ — $160^{\circ}$  C. The distillation is continued in vacuo. Two fractions are obtained, one at  $150$ — $205^{\circ}$  C., the other at  $210^{\circ}$ — $290^{\circ}$  C.

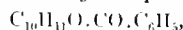
In the flask there remains a pitchy residue, not distilling at  $300^{\circ}$  C. The first fraction contains the new compound. It is purified by passing steam through a mixture of the first fraction with caustic soda of 1.5 per cent. strength. The last traces of aromatic hydronaphthol are removed by diazobenzene sulphonic acid, which does not act on the alicyclic compounds. It is then dissolved in ether and it remains after evaporating the solvent as a colourless oil boiling under a pressure of 716 mm. at  $264^{\circ}$  C. It is with difficulty soluble in water, easily in alcohol, ether, benzene, carbon bisulphide, and chloroform, but insoluble in the alkalis. The authors describe several derivatives of this compound.

*Alicyclic  $\beta$ -tetrahydronaphthylcarbonate of sodium*,  $C_{10}H_{11}.O.CO_2Na$ . This is a very unstable compound, and is obtained by passing carbonic acid through the sodium salt of the alcohol.

*Dihydronaphthalene*,  $C_{10}H_{10}$ , obtained by the action of solid potassium hydrate on the alcohol.

*Alicyclic acetyl- $\beta$ -tetrahydronaphthol*,  $C_{10}H_{11}.O.CO.CH_3$ . This is an oil boiling under a pressure of 34 mm. at  $169^{\circ}$  C.

*Alicyclic benzoyl- $\beta$ -tetrahydronaphthol*—



white crystals melting at  $62$ — $63^{\circ}$  C., and boiling under a pressure of 40 mm. at  $254$ — $255^{\circ}$  C.

*Alicyclic  $\beta$ -tetrahydronaphthylchloride*,  $C_{10}H_{11}Cl$ , obtained by the action of hydrochloric acid on the alcohol, could not be purified. Other compounds of less interest are described. —A. L.

*On Aromatic  $\alpha$ -Tetrahydronaphthol.* E. Bamberger and F. Bortt. Ber. **23**, 215—218.

The hydration of  $\alpha$ -naphthol proceeds analogously to that of  $\alpha$ -naphthylamine, the four hydrogen atoms entering into the non-substituted ring. The experiment was carried out in the ordinary way by treating 10 grms. of  $\alpha$ -naphthol with 20 grms. of sodium. The liquid turns, finally, light yellow. The  $\alpha$ -tetrahydronaphthol boils after purification at  $264.5^{\circ}$ — $265.5^{\circ}$  C., it solidifies to white plates, similar in appearance to naphthalene, and melting at  $68.5^{\circ}$ — $69^{\circ}$  C. It has the character of a phenol and forms azo-colouring matters with diazo-compounds.

*Phenylazo- $\alpha$ -tetrahydronaphthol*,  $C_6H_5.N_2.C_{10}H_{10}.OH$ , is obtained in the usual way from diazo-benzene chloride and an alkaline solution of the phenol. Green plates with a metallic lustre, insoluble in water, with difficulty soluble in benzene, soluble in glacial acetic acid, and easily soluble in chloroform.

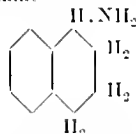
*Aromatic  $\alpha$ -tetrahydronaphthol-azo-benzene sulphonic acid*,  $(SO_3H)C_6H_4.N_2.C_{10}H_{10}.OH$ , is obtained from diazo-benzene sulphonic acid and  $\alpha$ -tetrahydronaphthol. The free acid forms scarlet-red leaves.

*Aromatic tetrahydro- $\alpha$ -naphthylethylether*,  $C_{10}H_{11}.OC_2H_5$ , boils at  $259^{\circ}$  C. under a pressure of 705 mm.—A. L.

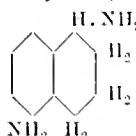


*Hydrogenised Naphthylamines.* E. Bamberger. Ber. **23**, 291—292.

AMONG the hydrated naphthalene bases two are distinguished by containing asymmetrical carbon atoms, viz., the alicyclic  $\alpha$ -tetrahydronaphthylamine—



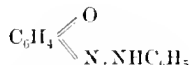
and the alicyclic 1, 5-tetrahydronaphthylendiamine—



These compounds are optically inactive. The author, however, succeeded in separating the first base into two active compounds, the one turning the ray of polarised light to the right, the other to the left. The separation was accomplished by preparing the acid tartrates, the salt turning the rays of polarised light to the left crystallising, whilst the remaining syrup contained the other base.—A. L.

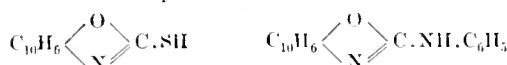
*On the Hydroxy-azo- (Orxazo-), and Amido-azo Compounds.* H. Goldschmidt and Y. Rosell. Ber. **23**, 487—508.

OXYAZO COMPOUNDS were considered, until a few years ago as hydroxy compounds of azo bodies. Since 1884 a series of reactions has been discovered, which render it possible to consider these products as quinone derivatives, and to explain, for example, the constitution of oxy-azo-benzene by the formula—



the phenylhydrazone of quinone. Zincke's synthesis of benzene-azo- $\alpha$ -naphthol by the action of phenylhydrazine on  $\alpha$ -naphthoquinone offers valuable support to this theory.

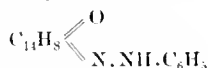
Ortho-oxy-azo compounds are converted by carbon bisulphide into anhydro compounds of ortho-amido-phenols. Benzene-azo- $\beta$ -naphthol forms, for instance—



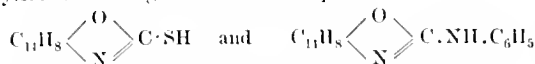
Thiocarbanilidonaphthol.

Carbanilamidonaphthol.

The hydrazones of orthoquinones behave in an analogous way. Phenanthrenequinonephenylhydrazone—



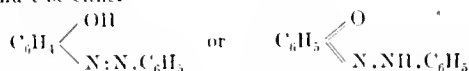
yields on heating with carbon bisulphide—



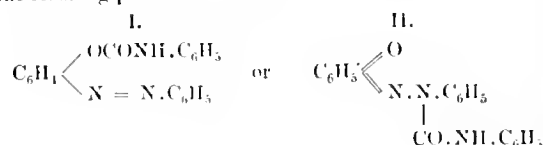
Thiocarbanilidophenanthrol.

Carbanilamidophenanthrol.

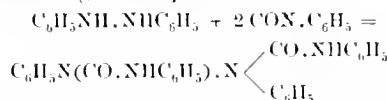
*Oxy-azo-benzene and Phenylecyanate.*—Oxy-azo-benzene must be either—



Phenylecyanate would act on this body in either case, and the resulting product would be either—



If oxy-azo-benzene and phenylecyanate be heated with addition of benzene to 170° C., a body is produced, which, when recrystallised from benzene, forms orange-yellow needles, soluble in alcohol and ether, insoluble in alkalis, and melting at 149° C. Its formula is  $\text{C}_{10}\text{H}_{13}\text{N}_3\text{O}_2$ . On heating this body with alcoholic potash, oxy-azo-benzene, aniline and carbonic acid are formed. If the first formula be correct, the product could be considered as a phenyl-carbanido-ether, whilst formula No. II. represents it as a kind of urea. The former bodies are easily acted upon by caustic alkalis, the latter proving far more stable under the action of these agents. The authors prepared a complicated member of the ureas by the action of phenylecyanate on hydrazobenzene in order to study the properties of a product which leaves no doubt as to constitution. By heating 2 mols. of phenylecyanate with 1 mol. of hydrazobenzene, in presence of a little benzene at 150° C., a greyish-blue amorphous mass was obtained, insoluble in all solvents, and melting at 218—220° C. Analysis proved that this body was the expected dicarbanilidohydrazobenzene, formed according to the equation—



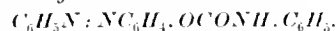
This body is extremely stable towards alcoholic potash, not suffering change even when heated to 150° C. Only at 200° C. it is converted into aniline, carbonic acid and benzdinehydrochloride.

This indicates that the oxy-azo-benzene must be represented as a phenol, i.e., as *hydroxyazobenzene*.

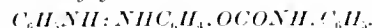
*Reduction of Carbanilido-oxy-azobenzene:* Carbanilido-oxyhydrazobenzene,  $\text{C}_{19}\text{H}_{17}\text{N}_3\text{O}_2$ .—If the reduction be performed with stannous chloride and hydrochloric acid, only aniline and paramidophenol are formed. If, however, zinc dust and glacial acetic acid are the reducing agents used, a colourless body, crystallising from benzene in needles is obtained, melting at 155° C. This is carbanilido-oxyhydrazobenzene. It is readily reoxidised to the original body. Alcoholic potash liberates aniline, whilst at the same time hydroxyazobenzene is formed, probably by immediate oxidation or the intermediate oxyhydrazobenzene. According to theoretical speculations of the authors the constitution of—

*Hydroxyazobenzene* is  $\text{C}_6\text{H}_5\text{N}:\text{NC}_6\text{H}_4\text{OH}$ .

*Carbanilido-oxyazobenzene* is—



*Carbanilido-oxyhydrazobenzene* is—



*Hydroxyazobenzene and  $\alpha$ -Naphthylecyanate* act on each other in the same way as above described, forming  $\text{C}_6\text{H}_5\text{N}=\text{NC}_6\text{H}_4\cdot\text{OCONH}\cdot\text{C}_{10}\text{H}_7$ , melting at 149° C. On reduction with zinc dust and acetic acid the corresponding hydrazo compound  $\text{C}_6\text{H}_5\text{N}:\text{NHNH}\cdot\text{C}_6\text{H}_4\cdot\text{OCONH}\cdot\text{C}_{10}\text{H}_7$  is obtained, melting at 155° C.

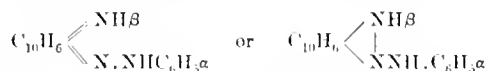
Two molecules of phenylecyanate acting on 1 molecule of carbanilido-oxyhydrazobenzene form a white crystalline powder melting at 215—218° C., of the formula  $\text{C}_{20}\text{H}_{17}\text{N}_5\text{O}_4$ . On boiling with alcoholic potash, diphenyl urea, aniline, and some other products were obtained, which were not further examined.

An isomeride of carbanilido-oxyhydrazobenzene was obtained by heating the latter with benzaldehyde to 150° C. It forms colourless needles, nearly insoluble in benzene, soluble in alcohol, and melting at 218°—222° C. The same body is formed by heating a mixture of the hydrazo compound and benzene to 150° C. Its constitution is not yet definitely settled.

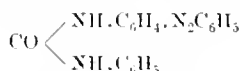
*Orthohydroxyazo Bodies and Phenylecyanate do not React on each other.*—Experiments with benzene-azo-para-cresol,  $\text{C}_6\text{H}_3(\text{CH}_3)(\text{OH})\text{N}:\text{C}_6\text{H}_5$  [1:4:3], benzene-azo- $\beta$ -naphthol,  $\text{C}_{10}\text{H}_6(\text{OH})\text{N}:\text{C}_6\text{H}_5$ , were without result, however the conditions were varied. These bodies have probably to be considered as hydrazones.

Phenoldisazobenzene,  $C_6H_3(OH)(N_2C_6H_5)_2$  [1:2:4] and phenyleyanate react on each other if heated to  $170^\circ C.$ , forming a body crystallising from benzene in small yellow needles, which melt at  $133^\circ-135^\circ C.$  It is carbanilido-phenoldisazobenzene,  $C_{10}H_5.NHC_6H_5.O.C_6H_3(N_2C_6H_5)_2$ .

**Amido-azo-Compounds.**—Whilst there are no reasons to alter the formula representing the constitution of the para-compound, a series of observations on ortho-amido-azo-compounds has been made, which seem to indicate that the present interpretation of these bodies is deficient. According to Zincke they have to be considered as imidohydrazones; the benzene-azo- $\beta$ -naphthylamine, for instance, being capable of representation as either—

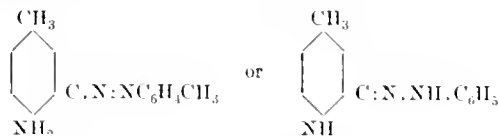


Carbodi-imido bodies form addition compounds with the phenyl amides, whereas such a reaction is not possible with imido derivatives. Acting with phenyleyanate on amido-azo-benzene the authors obtained a golden yellow compound, soluble in alcohol, nearly insoluble in benzene, and melting at  $216^\circ C.$  The substance was carbanilido-amido-azo-benzene,  $C_{10}H_{16}N_4O$ . Further experiments proved its constitution to be—

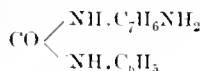


Benzene-azo-diphenyl urea. Amido-azo-benzene, therefore must be  $C_6H_5.N_2C_6H_4NH_2$ .

**Ortho-Amido-azo-toluene** must be either—



Phenyleyanate acts on its benzene solution at ordinary temperatures. The brownish-yellow precipitate, recrystallised from hot alcohol forms golden-yellow pointed needles, melting at  $219^\circ C.$ —it is carbanilidoamidoazotoluene. On reduction with stannous chloride and hydrochloric acid a white body, probably amidotolylphenyl urea—

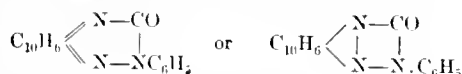


melting at  $195^\circ C.$ , is obtained.

**Benzene-azo- $\beta$ -naphthylamine** is acted upon by phenyleyanate at  $125^\circ C.$ , and an orange substance  $C_{20}H_{18}N_4O$ , melting at  $205^\circ C.$ , is formed. By reduction a white body melting at  $290^\circ C.$  Amidonaphthylphenyl urea—

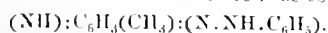
$C_{10}H_6(NH_2)NH.CO.NH.C_6H_5$ <sup>a</sup> is obtained.

To prove whether *o*-amido-azo-compounds are genuine azo-bodies or quinone derivatives benzene-azo- $\beta$ -naphthylamine was treated with 2 mols. of phenyleyanate at  $150^\circ C.$  A dark liquid and a light yellow crystalline mass were the result of this reaction. From the latter three different substances were isolated:—(1.) A compound crystallising in yellow needles melting at  $252^\circ C.$  (2.) Diphenyl urea. (3.) Monocarbanilido-benzene-azo- $\beta$ -naphthylamine. No. 1 product is, according to the author's view, either—



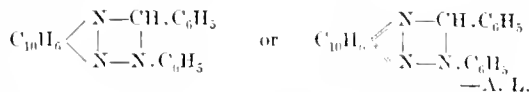
and proves the ortho-amido-azo-compounds to be quinone derivatives.

**Ortho-amido-azo-toluene** is therefore to be considered as—



**Action of Benzaldehyde on Ortho-amido-azo-toluene.**—On dissolving ortho-amido-azo-toluene in an excess of benzaldehyde, crystals are formed after a short time, and when recrystallised from alcohol form colourless needles melting at  $220^\circ C.$ , and having the formula  $C_{21}H_{19}N_2$ . The corresponding benzene compound is orange-yellow.

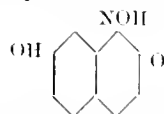
**Benzene-azo- $\beta$ -naphthylamine** is acted upon by benzaldehyde only after heating a mixture of equal molecules for some time at  $140^\circ C.$  As the result a white substance is obtained in microscopic crystals, melting at  $193^\circ C.$ , and having the formula  $C_{20}H_{17}N_2$ . Boiling this body with hydrochloric acid caused decomposition, and heating it under pressure only formed the hydrochloride. Ordinary benzylidene compounds have no basic character, and are decomposed by boiling with hydrochloric acid into benzaldehyde and the original base. The constitution of this body is either—



**On 2, 7.—Dihydroxy-naphthalene.** A. Clausius. Ber. **23**, 517—529.

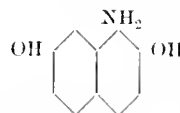
This compound crystallises from hot water in long needles melting at  $190^\circ C.$  Its acetyl compound forms white leaves, melting at  $136^\circ C.$ , whilst its benzoyl compound crystallises from alcohol in colourless leaves, melting at  $139^\circ C.$

**1, 2, 7.—Hydroxy-naphthoquinone oxime—**



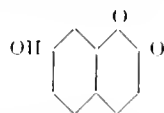
is obtained by the action of nitrous acid on the dihydroxy compound. It forms brownish-yellow needles melting at about  $235^\circ C.$  No acetyl compound could be prepared from it. The author did not succeed in introducing a second nitroso group.

**1, 2, 7.—Dihydroxyamidonaphthalene—**



The free base is very unstable; the hydrochloride is more stable and can be easily prepared by reducing the nitroso compound with stannous chloride.

**7, 1, 2.—Hydroxy-naphthoquinone—**



is an isomeric of Juglone and the ordinary hydroxy-naphthoquinone. It is obtained from the above described amido compound by oxidation with ferric chloride as a reddish-brown amorphous precipitate which did not admit of crystallisation. It is soluble in sulphites, but cannot be reprecipitated by acids. Chlorine in presence of acetic acid acts on the hydroxyquinone, but no pure products could be separated from the resinous mass.

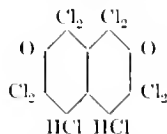
**1, 2, 7.—Benzene-azo-dihydroxy-naphthalene—**



obtained by combining diazobenzene chloride with an alkaline solution of the dihydroxy-naphthalene. Recrystallised from alcohol it forms bluish-green needles, melting at  $220^\circ C.$ , soluble in alkalis. Its acetyl compounds form white needles, melting at  $195^\circ C.$

1, 3, 6, 8, 2, 7.—*Tetra-chlorodihydroxynaphthalene* was obtained by reducing the deachlorodiketone with stannous chloride. Long fine needles, melting at 176° C., soluble in alcohol and hot glacial acetic acid. Its acetyl compound crystallises in needles, melting at 196° C.

*Decachloro-2, 7.—Diketohydronaphthalene—*



is the final product obtained by the action of chlorine on the dihydroxy compound in a solution of acetic acid. Colourless crystals, melting and decomposing at 200° C., with difficulty soluble in alcohol, glacial acetic acid, and benzene.

2, 7.—*Dianilidonaphthalene—*  
(2, 7.—*Phenylamidonaphthalene*)—



is formed by heating the dihydroxy compound with aniline and calcium chloride for 18 hours at 240°—290° C. By treating the brownish mass with alkali the mono- and dihydroxy-compounds are removed. Bright colourless plates, melting at 168° C. The diaetyl compound melts at 197.5° C. Its benzene-azo-compound, obtained by the action of the diazobenzene chloride on the dianilido-compound, forms brownish needles when recrystallised from alcohol.

2, 7.—*Hydroxynilidonaphthalene—*

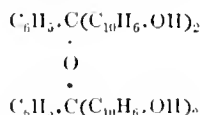


forms small needles, melting at 163° C., easily soluble in glacial acetic acid, alcohol, and benzene.—A. L.

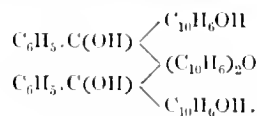
*On Combinations of Benzyltrichloride with Phenols.*  
O. Döbner. Ann. 257, 56—60.

BENZYLTRICHLORIDE acts only on such phenols as hold the para-position free with regard to the hydroxy-groups.

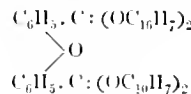
*Benzyltrichloride and α-Naphthol.*—On heating 1 mol. of benzyltrichloride with 2 mols. of α-naphthol on the water-bath, hydrochloric acid is evolved and a dark green melt formed. After the evolution of hydrochloric acid is at an end the melt is boiled with dilute caustic soda, from which after filtration the α-Naphthol-benzoin is precipitated by hydrochloric acid. For further purification it is dissolved in absolute alcohol and reprecipitated by concentrated hydrochloric acid. It could not be obtained in crystals, but forms a reddish-brown powder, insoluble in water, soluble in alcohol, ether, benzene, and glacial acetic acid, with a yellowish-red colour. It combines with alkalis and acids, if its alcoholic solution is treated either with one or the other. A benzoyl- or acetyl-compound could not be prepared. On fusion with caustic potash, benzoic acid and α-naphthol were obtained. By the action of zinc and glacial acetic acid it is converted into an amorphous colourless powder, which seems to be, according to analysis, dihydroxydinaphthylphenylmethane,  $C_{26}H_{20}O_2$ :  $(C_{10}H_6.OH)_2$ . The constitution of the α-Naphthol-benzoin is not yet finally decided; it is either—



or



One mol. of benzyltrichloride and 2 mols. of β-naphthol react, forming a body melting above 350° C., insoluble in alkalis and acids, crystallising from nitrobenzene in small white needles, insoluble in water, alcohol, ether, and benzene. It is isomeric with the α-compound, and in accordance with its properties, is *o*-benzoyl-β-naphthylether—



—A. L.

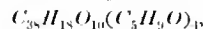
*On Pyrogallol-Benzoin.* O. Döbner and A. Förster.  
Ann. 257, 60—68.

THE blue colouring matter obtained by the action of pyrogallol on benzyltrichloride is prepared in the following way:—One mol. of the benzyltrichloride is gradually added to 2 mols. of the phenol and the mixture heated to 160° C., until evolution of hydrochloric acid ceases. The melt is boiled with water to remove the excess of pyrogallol and to convert the chloride first formed into the carbinol. The colouring matter is purified by crystallisation from glacial acetic acid and forms small dark green crystals. Yield from 100 grms. of pyrogallol = 60 grms. of pure colouring matter. The pyrogallol-benzoin is nearly insoluble in water, petroleum spirit, benzene, or carbon bisulphide, more easily soluble in alcohol, ether, hot chloroform, alkalis, alkaline carbonates, and a hot solution of sodium acetate. Its formula is  $C_{23}H_{15}O_{11}$ . From the neutral solutions of the ammonium salt of pyrogallol-benzoin most metallic salts precipitate blue-violet amorphous lakes. It is, however, of little value as a colouring matter. It dyes unmordanted cotton or cotton mordanted with tartar-emetic and tannin only very faintly. Better results are obtained with aluminium, iron, and other similar mordants; the colours are, however, not fast towards acetic acid.

*Tetra-acetyl Pyrogallol-benzoin*,  $C_{38}H_{20}O_{11}(C_2H_3O)_4$ , forms light red crystals, melting at 208° C., with difficulty soluble in alcohol and ether, insoluble in water.

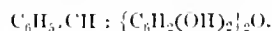
*Tetra-valeryl Pyrogallol-benzoin*,  $C_{38}H_{20}O_{11}(C_4H_7O)_4$ , red prisms from hot alcohol or acetone, melting at 251° C.

*Tetra-valerylhydro Pyrogallol-benzoin*,—

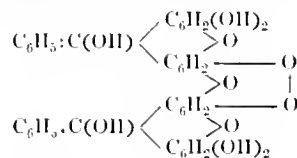


red needles soluble in alcohol or acetone, melting at 227° C., different from the above-described compounds, having lost one molecule of water.

*Hydropyrogallol-benzoin*,  $C_{19}H_{11}O_5 + 3 H_2O$ , was obtained by reduction of the colouring matter with glacial acetic acid and zinc dust. Either reddish crystalline powder or long silky needles of the constitution—



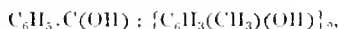
Concentrated sulphuric acid forms a sulphonic acid with pyrogallol-benzoin. The constitution of the pyrogallol-benzoin is expressed by the formula—



—A. L.

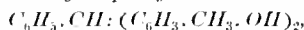
On *Ortho-cresol-benzoin*. G. Schröter. Ann. **257**, 68—75.

Forty-six grms. of benzyltrichloride are gradually added to 50 grms. of ortho-cresol and the mixture is heated on the water-bath until the evolution of hydrochloric acid ceases. The excess of ortho-cresol is removed by distillation with steam. The colouring matter is dissolved in dilute caustic soda, filtered, and precipitated by hydrochloric acid. On heating with sodium bisulphite it is dissolved, and after filtration reprecipitated. It melts at  $220^{\circ}$ — $225^{\circ}$  C., and its formula is  $C_{21}H_{20}O_3$ . An acetyl compound could not be prepared. Its constitution is—



on reduction of this compound with sulphurous acid,

*Dihydroxydimethyl-triphenylmethane*—



melting at  $170^{\circ}$ — $171^{\circ}$  C., is formed, easily soluble in the usual solvents, water excepted. With acetic anhydride a diacetyl-compound,  $C_{21}H_{18}O_5(C_2H_3O)_2$ , is produced; light yellow needles, melting at  $91^{\circ}$  C. Its benzoyl-compound,  $C_{21}H_{18}O_5(C_7H_5O)_2$ , crystallises in white leaves, melting at  $91.5^{\circ}$  C. By the action of bromine a dibromide,  $C_{21}H_{18}O_5Br_2$ , is obtained; yellowish-red needles, melting at  $130^{\circ}$  C., insoluble in water, soluble in alcohol, benzene, and glacial acetic acid. With nitric acid a dinitro-compound,  $C_{21}H_{18}O_5(NO_2)_2$ , is formed, soluble in benzene, alcohol, and glacial acetic acid, and melting at  $127^{\circ}$  C.

On fusing ortho-cresol-benzoin with caustic potash dimethyl-dihydroxybenzophenone is obtained, colourless needles, easily soluble in alcohol, ether, and glacial acetic acid, with difficulty in benzene, and melting at  $138^{\circ}$  C.

—A. L.

#### PATENTS.

*Improvements in Producing Yellow Colouring Matter suitable for Dyeing and Printing.* J. Erskine, Glasgow. From "The Farbenfabriken vormals Friedrich Bayer and Co.," Elberfeld, Germany. Eng. Pat. 9162, June 19, 1884. Amended March 5, 1890. 4d.

Is the original specification the patentee claimed the production of a yellow colouring matter by diazotising benzidine and combining the tetrazodiphenyl thus formed with *salicylic acid*, *phenol*, or *cresol* (this Journal, 1885, 400). The claim is now restricted to the use of *salicylic acid* alone for the combination with the tetrazo compound.

There are one or two minor emendations in the text, which do not affect the claim.—O. H.

*Improvements in the Manufacture of Colouring Matters.* J. E. Bedford, Leeds. Eng. Pat. 6777, April 23, 1889. 6d.

THE subject-matter of this patent has been incorporated in No. 6778 of 1889 (see this Journal, 1890, 385).—T. A. L.

*The Production of New Bluish-black Colouring Matters.* S. Pitt, Sutton. From L. Casella and Co., Frankfurt-on-the-Maine, Germany. Eng. Pat. 7977, May 13, 1889. 6d.

This is an extension of Eng. Pat. 9214 of 1885 (this Journal, 1886, 427), and provides for the combination of the diazo-azo compounds there described with substituted *m*-diamines and *m*-amido-phenols, producing black colouring matters, fast to milling, and some of them having an affinity for cotton. The two following examples illustrate the method. (1.) 34.7 kilos. of sodium  $\alpha$ -naphthylamine disulphonate are diazotised, combined with 18 kilos. of  $\alpha$ -naphthylamine hydrochloride, and this compound re-diazotised by the addition of 7 kilos. of sodium nitrite. It is then combined with an alcoholic solution of 16 kilos. of tetramethyltolylenediamine and an equivalent quantity of sodium acetate. The colouring matter remains almost entirely in solution, and crystallises out on distilling off the alcohol. It dyes wool a bluish-black from a neutral bath containing salt. (2.) The diazo-azo compound prepared as above is combined with an alkaline solution of 18 kilos. of

*m*-hydroxydiphenylamine (phenyl-*m*-amido-phenol), and the dyestuff which forms immediately is precipitated by the usual means. It dyes wool in a neutral or acid bath a black of a reddish shade, and can also be used as a cotton dye. In place of  $\alpha$ -naphthylaminedisulphonic acid, the sulphonic acids of aniline, toluidine, xylylene, benzidine, its homologues and amidostilbene may be employed. With respect to Eng. Pat. 4825 of 1889 (this Journal, 1890, 172) no claim is made in this patent to the invention described therein.

—T. A. L.

*Improvements in the Production of Blue-Black Colouring Matters.* H. H. Lake, London. From A. Leonhardt and Co., Muhlheim-on-the-Maine, Germany. Eng. Pat. 8264, May 17, 1889. 4d.

THE nitroso derivatives of tertiary aromatic amines, when heated with *m*-hydroxydiphenylamine in the proportions of three molecules of the former to one of the latter, yield blue-black colouring matters. 16.8 kilos. of nitrosodimethylaniline hydrochloride are heated with 5.6 kilos. of *m*-hydroxydiphenylamine and 16 litres of spirit until all the nitrosodimethylaniline has disappeared. The spirit is then distilled off, the residue dissolved in hot water, slightly acidulated with hydrochloric acid, filtered, and the colouring matter precipitated with salt.—T. A. L.

*Improvements in Preparing Colouring Matters of the Oxyketone Group, suitable for Dyeing and Printing with the Aid of Morchants.* J. Y. Johnson, London. From "The Badische Anilin und Soda Fabrik," Ludwigshafen, Germany. Eng. Pat. 8373, May 20, 1889. 6d.

New colouring matters which dye yellow on an alumina mordant are obtained by condensing (1) pyrogallol with certain carboxyl derivatives of the aromatic and fatty series, viz., benzoic acid, *p*-chlorobenzoic acid, salicylic acid, *m*-hydroxybenzoic acid, *p*-hydroxybenzoic acid, *m*-cresotic acid,  $\beta$ -resorcylic acid, propionic acid, *n*-butyric acid, valeric acid; (2), gallic acid with certain phenolic bodies, viz., resorcinol and pyrogallol. The condensation is performed in presence of sulphuric acid, chloride of zinc, or tetrachloride of tin. Trihydroxybenzophenone, which dyes cotton mordanted with alumina a golden-yellow colour, is obtained by heating 1 kilo. of pyrogallol and 1 kilo. of benzoic acid with 3 kilos. of zinc chloride to  $140^{\circ}$  C. The reaction is complete in about three hours, when a sample of the melt dissolved in water should deposit no laminae of benzoic acid on cooling, but light yellow needles of the new compound. The whole melt is then dissolved in water, boiled with animal charcoal, filtered and allowed to crystallise. The trihydroxybenzophenone thus obtained is very slightly soluble in cold water, readily soluble in alcohol, ether, acetone and glacial acetic acid. It dissolves in concentrated sulphuric acid and in alkalis with an intense yellow colour. The alkaline solutions in presence of an excess of caustic potash or soda rapidly absorb oxygen and form a green product. All these compounds contain hydroxyl groups in the ortho-position, and the colours obtained from them are said to be as fast as alizarine dyes. The melting points and properties of several of these new oxyketones are given in the patent.

—T. A. L.

*Improvements in Preparing Colouring Matters of the Oxyketone Group suitable for Dyeing and Printing with the Aid of Morchants.* J. Y. Johnson, London. From "The Badische Anilin und Soda Fabrik," Ludwigshafen, Germany. Eng. Pat. 9428, June 6, 1889. 4d.

AN extension of Eng. Pat. 8373 of 1889 (see preceding abstract), describing the preparation of the same compounds by substituting for the carboxyl derivatives the acid chlorides or anhydrides of the acids so far as known. For example, in place of heating pyrogallol with benzoic acid, the latter may be replaced by benzoyl chloride or benzoic anhydride.

—T. A. L.

*Process for the Manufacture of Yellow to Orange Colouring Matters.* T. R. Shillito, London. From A. Feer, Brombach, Germany. Eng. Pat. 12,549, August 8, 1889. *Ad.*

TETRAMETHYLDIAMIDO-DIPHENYLMETHANE, when heated with sulphur in presence of ammonia or a primary aromatic amine, forms yellow and orange dyestuffs. 12.7 kilos. of tetramethyldiamido-diphenylmethane, and 3.2 kilos. of sulphur are heated to 140° C. in an autoclave provided with an inlet and outlet pipe; 120 kilos. of salt and 7 kilos. of ammonium chloride are then added, the temperature raised to 175°, and a slow current of ammonia passed through the melt for 7–8 hours. When cold it is treated with cold water to remove the salt, dissolved in hot water and reprecipitated with salt. The colouring matter dyes cotton mordanted with tannin a pure yellow shade. An orange-red dyestuff is obtained by heating 25 kilos. of tetramethyldiamido-diphenylmethane, 20 kilos. of aniline, and 9.5 kilos. of sulphur to 200° C. When no more gas is evolved the melt is dissolved in 300 kilos. of 40 per cent. acetic acid, diluted with water, filtered, and the colouring matter precipitated with zinc chloride and salt. It forms a bright red powder and dyes mordanted cotton orange-red.—T. A. L.

*Production of New Red Colouring Matters.* S. Pitt, Sutton. From L. Casella and Co., Frankfort-on-the-Maine, Germany. Eng. Pat. 12,560, August 8, 1889. *Ad.*

HITHERTO in the benzidine series red colouring matters have only been obtained by combining benzidine and other para-diamines with naphthylamine sulphonic acids. The naphthols and their sulphonic acids only yield blue to violet colours. It has, however, been found that by alkylating or benzylating certain of the dyestuffs obtained from naphthol sulphonic acids and tetrazo-diphenyl, red colouring matters are formed. The process is worked as follows:—18.4 kilos. of benzidine are diazotised and added to a cooled solution of 40 kilos. of sodium  $\beta$ -naphthol- $\gamma$ -disulphonate. After a short time 10 kilos. of phenol are added, the whole heated to boiling, and the colouring matter precipitated by salt and filtered off. This is then dissolved in 200 litres of water, 200 kilos. of alcohol and 4.5 kilos. of caustic soda, and heated with 12 kilos. of ethyl bromide for 24 hours under pressure at 60° C. On cooling, the new colouring matter separates in shining crystals. It dyes unmordanted animal or vegetable fibres scarlet from an acid, neutral or alkaline bath.—T. A. L.

## V.—TEXTILES: COTTON, WOOL, SILK, Etc.

### PATENT.

*Process for Separating the Wool from Pieces of Rabbit Skins and other Skins of a similar Kind.* C. Puech, Paris, France. Eng. Pat. 10,010, June 18, 1889. *Ad.*

THE pieces with the fur on are "effectually" boiled in water, washed, and afterwards dried at 70° to 85° C. The albuminous and gelatinous constituents are hereby entirely removed from the leather, which loses its cohesive strength. The dried leather having thus become brittle, a simple crushing operation by means of stamps, crushing cylinders, &c., reduces it to powder, liberating the whole of the wool. The crushing is best carried out as soon as the material issues from the drying apparatus, and while still very hot. The material is then sifted or bolted, to separate the dust from the wool.—H. S.

## VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

*On Turkey-Red Oil.* P. Lochtin. Dingl. Polyt. J. 275, 594–603.

THIS article treats of the conditions necessary for successful application of the "Sulphated Oil Process" in dyeing cotton with Alizarine red. All recipes for preparing the oil agree in requiring the sulphuric acid to be added very slowly to the oil and with constant stirring, in order to avoid heating during the mixing and the evolution of sulphurous acid in any large quantity. The author describes experiments which demonstrate that the risk of spoiling the product is much less than is commonly supposed. He shows that at temperatures below 70° any evolution of sulphurous acid must be due to albuminoid substances present as impurities in the oil, the oil itself not giving rise to sulphurous acid below that temperature. (The paper is practically confined to the treatment of castor oil, as that is the only one in common use in Russia for Turkey-red dyeing.) The endeavour to avoid any evolution of sulphurous acid may even lead to very imperfect results being obtained, for a low temperature of reaction will leave much of the oil undecomposed—the author's view being that only the free fatty acid is of use in the dyeing process. In working on the large scale with 1,000 kilos. of castor oil the author adopts the following procedure. In winter 20 to 30 per cent., and in summer 15 to 20 per cent. of concentrated sulphuric acid are used. About one half of the acid is stirred into the oil during 9 hours of the working day and the mixture is allowed to stand overnight. On the next day the second half of the acid is stirred in and the mixture left to stand until a sample gives a clear solution when diluted with water, 5 to 10 drops being shaken with distilled water in a test-tube. The bulk is then mixed with an equal weight of water. It is necessary to test a sample frequently, as the mixture will give a cloudy solution in water if either too short or too long a time be given for the reaction. The proportion of free fatty acid may be increased if desired by using hot water for dilution. On allowing the mixture to stand, the oily part separates and floats on the aqueous acid layer, and it is drawn off to be treated with alkali. As some of the best preparations contain only 2 to 5 parts of  $\text{SO}_3$  to 100 parts of fatty acids, whereas the sulphonated fatty acid should contain 6 to 15 times more  $\text{SO}_3$ , it is evident that the sulphonated fatty acid is not of essential importance, and this conclusion is corroborated by an experiment on the large scale in dyeing with fatty acid prepared by saponification with alkali in which, of course, no sulphonated compound was present; but although the shade produced was very fine, the oil-bath showed a tendency to frothing, which caused the dyeing to be somewhat uneven. In order to fit the sulphated castor oil for use as a mordant, it has to be partially neutralised by an alkali, and the author finds that sufficient alkali has been added when it amounts to more than a quarter and less than half that required for theoretical neutralisation. In practice the alkali, either caustic soda or ammonia, or both, is added to the emulsified fatty acid until the mixture becomes suddenly clear, when the right degree of neutralisation has been attained. The use of ammonia for neutralisation has certain advantages, especially in the finishing and printing of steam colours, because the alkali volatilises and leaves only the free fatty acid on the cloth. The reason why castor oil has come to be selected as the most suitable for Turkey-red dyeing is its property of forming a soluble soap with a minimum proportion of alkali. Solutions of other fatty acids give emulsions rather than clear solutions when the proportion of alkali falls much below that required for complete neutralisation. Again, the decomposition of the oil by the action of sulphuric acid is more complete with castor oil than with any other oil which can be used, and maximum decomposition is essential to the achievement of the best results. A certain proportion of undecomposed oil has, however, the advantage that it lessens the tendency to frothing, and the decomposition by means of sulphuric acid in the case of castor oil provides for the ready attainment of this condition.—G. H. B.

*Thioflavine.* A. Scheurer. Bull. Ind. Soc. Mulhouse, 1889, 563.

Nothing further than what has already appeared (this Journal, 1889, 608).—E. B.

*Heilmann's Steaming Machine for Worsted Yarn.*

P. Heilmann. Bull. Ind. Soc. Mulhouse, 1889, 564—567.

In the usual process of steaming woollen yarns as effected by placing the cops, canettes, &c. in baskets inside closed steaming vessels, and there submitting them to the action of steam for some three-quarters of an hour, the results obtained are unsatisfactory. The cops at the top of the baskets are excessively steamed, becoming elongated and twisted at the ends, rendering difficult the winding off of the yarn, whilst the cops at the bottom are insufficiently steamed. These defects are overcome in the machine here described by creating a vacuum in the chamber in which the yarn is placed before admitting the steam, and the steaming is done in a minute or a minute and a half.

The steaming-vessel A (Figs. 1, 2, and 3) is provided with a false roof of sheet copper, which serves to protect the wool from being iron-stained by drops of condensed

Fig. 1.

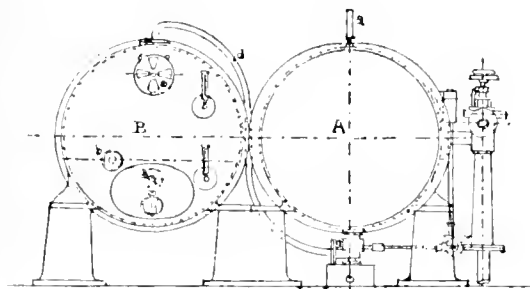


Fig. 2.

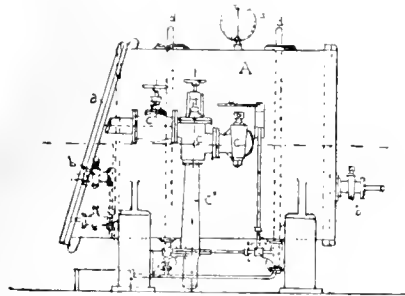
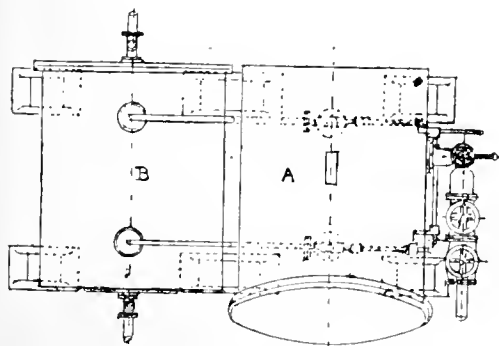


Fig. 3.



moisture, and is capable of holding two baskets containing each 20 to 25 kilos. of wool. The vacuum is produced by

the steam-injector C, the steam blowing away through C'' and aspirating the air through C'''. B is a second chamber into which the steam enters through a layer of boiling water at the bottom; b' (Fig. 1) is an over-flow pipe. There are also arrangements for the supply of water and regulation of the steam supply. c (Fig. 2) is the tap which places the two chambers in communication.—E. B.

*Chromium Oxides and Salts.* Prud'homme. Bull. Soc. Ind. Mulhouse, 1889, 599—609.

*Chromic Hydrate*, as precipitated in the cold from either a green or violet chromic salt, is soluble in sodium hydrate, the solution on standing depositing chromic hydrate in a gelatinous condition. According to Fieuny, when the alkaline solution is boiled, a precipitate of anhydrous chromic oxide is formed. The author finds both precipitates, namely, that obtained in the cold and that obtained on boiling, to be identical, having each, when dried at 100°, the composition  $\text{Cr}_2\text{O}_3 \cdot 5 \text{H}_2\text{O}$ .

On mixing boiling solutions of chromic alum and sodium hydrate in excess, a pale-green, powdery precipitate is produced, which settles rapidly, differing in this respect from the dark-green, gelatinous precipitate produced at a lower temperature. The attempt was made to fix this powdery precipitate on cotton tissues by preparing in a chromium salt, drying and fixing in a boiling solution of caustic soda containing 50 grms. of caustic soda of 36° B. per litre, better results being so obtained than with carbonate of soda, even at the rate of 200 grms. of the latter per litre. This powdery precipitate also contains five molecules of water when dried at 100°.

As regards the colour of the precipitate, it is found that the normal green colour always characterises the precipitates obtained by the addition to a violet chromic salt of an amount of alkali insufficient to completely precipitate it. Ammonium hydrate for instance, under this condition, yields a yellow-green precipitate, whereas when added in excess it produces a violet-blue precipitate. Sodium hydrate, again, when not added in excess to a solution of violet salt, yields a green precipitate dissolving in acids with a violet colour, but when even a slight excess of soda is used the precipitate dissolves in acids with a green colour. A green chromic salt, precipitated at the boil with ammonium hydrate not in excess, yields a green hydrate forming green salts; with excess of the reagent a pearl-grey precipitate is produced, which dissolves in cold acids with a violet colour. The uncrystallisable green salts in time become violet and crystallisable. Etard states that traces of potassium nitrite or thiocyanate rapidly induce this change. On the other hand, the author finds that the addition to a violet salt of small quantities of sodium bisulphite or borax changes its colour into green, and Etard has remarked a similar change under the influence of arsenic acid and arseniates. From these considerations it is inferred that the difference in colour is purely physical and of only momentary value, as the green salts at ordinary temperatures are slowly tending towards the stable violet state. Chemically, a green salt may be defined by the minimum temperature required for the transformation of a violet salt, namely, 65°, or still better, 100°, special reactions of the chromic salts taking place only at this latter temperature.

*Chromous Chloride* exists in two modifications, coloured respectively blue and green, a fact which seems to have escaped the notice of chemists. The blue salt is obtained by reducing a chromic salt with zinc and a little hydrochloric acid; it reduces stannous chloride and colouring matters. Heated in absence of air it becomes green, its blue colour returning on cooling. When a green chromic salt is reduced at 100° with zinc dust, green chromous chloride is obtained which only gradually passes into the blue form; the former modification will not reduce stannous chloride at the boil, but does so at lower temperatures. The existence of two modifications of chromous chloride explains the reason of the solubility of anhydrous chromic chloride in a 1 per cent. solution of chromous chloride.

*Chromic Chlorate* possesses either a green or violet colour according as it is prepared at or below 65°. As

prepared by double decomposition between chrome alum and barium chlorate it holds in solution barium sulphate, which may be precipitated by boiling or addition of hydrochloric acid. On heating a solution of chromium chlorate to 100°, chlorine is evolved and the liquid becomes reddish-yellow in colour, and then contains chromic acid and oxides of chlorine; this liquid constitutes a powerful oxidising mixture, and at one time was employed by the author for the purpose of rendering aniline blacks ungreenable.

The chromium salts have the singular property of carrying with them into alkaline solution the oxides of certain metals; ferric oxide thus dissolves very well, forming a clear brown solution which keeps for weeks without depositing either ferric or chromic oxide; cupric oxide forms a double salt of a fine blue colour, depositing cuprous oxide on boiling, and leaving sodium chromate in solution. When the copper salt is in excess, the liquid, after boiling, is of a greenish-yellow colour, and contains copper chromate along with sodium chromate, and this liquid, after some months' standing, deposits chromic oxide and insoluble copper chromate, becoming itself blue, and then containing no chromium salts. The blue liquid on boiling deposits a mixture of cuprous and cupric oxides; if kept in the cold the blue colour again becomes green, depositing cupric oxide and eventually containing nothing but cuprous oxide.

Berzelius has stated that chromic hydrate dissolves easily in sulphurous acid in the cold, and on boiling a green powdery precipitate of *chromic sulphite* is produced. The author finds that the solvent action of sulphurous acid and also that of alkaline bisulphites is limited to the chromic hydrate precipitated in the cold by ammonia or sodium hydrate not in excess. The yellow-green solution of chromic hydrate in sodium bisulphite after the lapse of some time deposits chromic sulphite, the solution remaining green. Addition of a large excess of ammonia to the freshly-prepared green solution transforms it into a magnificent red liquid, which probably contains a chromamine of the class discovered by Frémy. Such a solution can be used for mordanting cotton with chrome, but better results are obtained with the following mixture:—45 parts of potassium bichromate, 20 of sodium carbonate, 100 of ammonium bisulphite of 36° B., 100 of ammonia, and 1,000 of water, the pieces being prepared in this solution, dried, and aged or steamed.

The oxide of chromium obtained by strongly heating the dried hydrate is of a grey-black colour. Crystallised chromic oxide may be prepared by fusing a mixture of potassium bichromate and metallic tin, being thus obtained in small shining leaflets of a pale green colour, which are not attacked by the strongest acids.—E. B.

#### *Notes on the Dyeing of Aniline Black.* J. Persoz. Bull. Soc. Ind. Mulhouse, 1889, 609—615.

This is a sealed note deposited with the Industrial Society of Mulhouse in 1875, and opened November 1889, for the purpose, as the author states in a preface to the note, of showing the state of advancement of aniline-black dyeing in 1875. At this period several manufacturers at Rouen, notably Daniel Fauquet, were dyeing by a single bath process, which had previously been practised by Bretonnière of Laval, and others.

In a previous sealed note opened towards the end of 1871 the author described a mode of dyeing aniline-black, based on the method suggested by Paraf-Javal, in which potassium bichromate and aniline salts were employed. The modification proposed consisted in applying the two compounds separately, padding the cloth or saturating the yarn with the solution of one of them, and then spraying the solution of the other over the surface. Excellent results were obtained, but the process was not a commercial success, owing to the want of a suitable spraying machine.

In the present note two methods for dyeing aniline-black are described. In the first of these, several baths are used, the cloth passing successively through the various baths of one range and then similarly through two or more such ranges, being well squeezed on emerging from each bath. The range of baths employed is as follows:—

First bath: neutral solution of ferric nitro-sulphate at 20° B.

Second bath: 100 parts of water, 10 of potassium bichromate, 1 of ammonium chloride.

Third bath: 100 parts of water, 10 of nitric acid, 7·5 of aniline.

Fourth bath: water.

In the second method the dyeing is effected in a single bath. The bath is composed of a mixture of three solutions separately prepared, consisting of (1) ammonium chloride, manganese chloride, copper sulphate, common salt, and ferric nitro-sulphate; (2) potassium bichromate and sulphuric acid; and (3) aniline salts and chloride of potash. After dyeing, the cotton is worked at 50° in a fixing-bath of potassium bichromate and other oxidising salts, then boiled in a 1 per cent. solution of acetic acid, and finally soaped for an hour at the boil with a 0·2 per cent. solution of soap.

—E. B.

#### PATENTS.

*Improvements in the Process and Means for Dyeing Hides.* J. Kristen, Brünn, Austria. Eng. Pat. 8191, May 16, 1889. 8d.

The dressed hide which is to be dyed is stretched over a table supported on a vertical shaft and capable of being rapidly rotated. A sufficient quantity of dye-liquor is run from a projecting swivel pipe on to the middle of the table, which is then set in motion, the centrifugal action produced causing the liquor to saturate every portion of the hide. The table is provided with a rimmed edge to prevent the dye-liquor being "thrown off horizontally," and is enclosed in a tank, arrangements being made for returning to the table the liquor which collects in the tank. In cases where it is desirable to subsequently sadden the dyed colours with iron or copper sulphate, it is recommended, for facility of removal of the hide to a separate table, that the hide be attached to a copper plate.—E. B.

*Improvements in the Production of Coloured Compounds of Gallacetophenone within or upon Animal and Vegetable Fibres.* J. Y. Johnson, London. From "The Badische Aniline und Soda Fabrik," Ludwigshafen, Germany. Eng. Pat. 9429, June 6, 1889. 6d.

GALLACETOPHENONE (J. Prakt. Chem. 23, 147—538), when combined with mordants, is capable of dyeing animal or vegetable fibres. It is employed preferably as a paste containing 10 per cent. of the dry substance, and with various mordants will give the following colours:—

*Yellow.*—37½ lb. of gallacetophenone paste, 15 lb. of aluminium thiocyanate of 1·15 sp. gr., 7½ lb. of calcium acetate solution of 1·075 sp. gr., 7½ lb. of acetic acid of 1·045 sp. gr., and 32½ lb. of thickening.

*Yellowish-Brown.*—37½ lb. of gallacetophenone paste, 15 lb. of chromium thiocyanate liquor of 1·2 sp. gr., 7½ lb. of acetic acid of 1·045 sp. gr., and 40 lb. of thickening.

*Black.*—37½ lb. of gallacetophenone paste, 15 lb. of acetate of iron liquor of 1·116 sp. gr., 7½ lb. of acetic acid of 1·045 sp. gr., and 40 lb. of thickening.

The goods after printing are subsequently steamed, and finally soaped, washed, and dried.—T. A. L.

*Improvements in Doctors Employed in the Printing of Textile Fabrics.* J. Archer, Radcliffe. Eng. Pat. 18,145, November 13, 1889. 6d.

THE front edge of the improved doctor-blade is constructed of two or three layers of steel, brass, or other suitable material, soft-soldered or brazed together, by which means increased strength and durability are secured, whilst the necessary suppleness and elasticity are retained by the thin back portion of the blade. The same end may be gained by constructing the blade out of a single solid sheet of metal of the thickness required for the front edge, the back part being cut or ground down.—E. B.



## VII.—ACIDS, ALKALIS, AND SALTS.

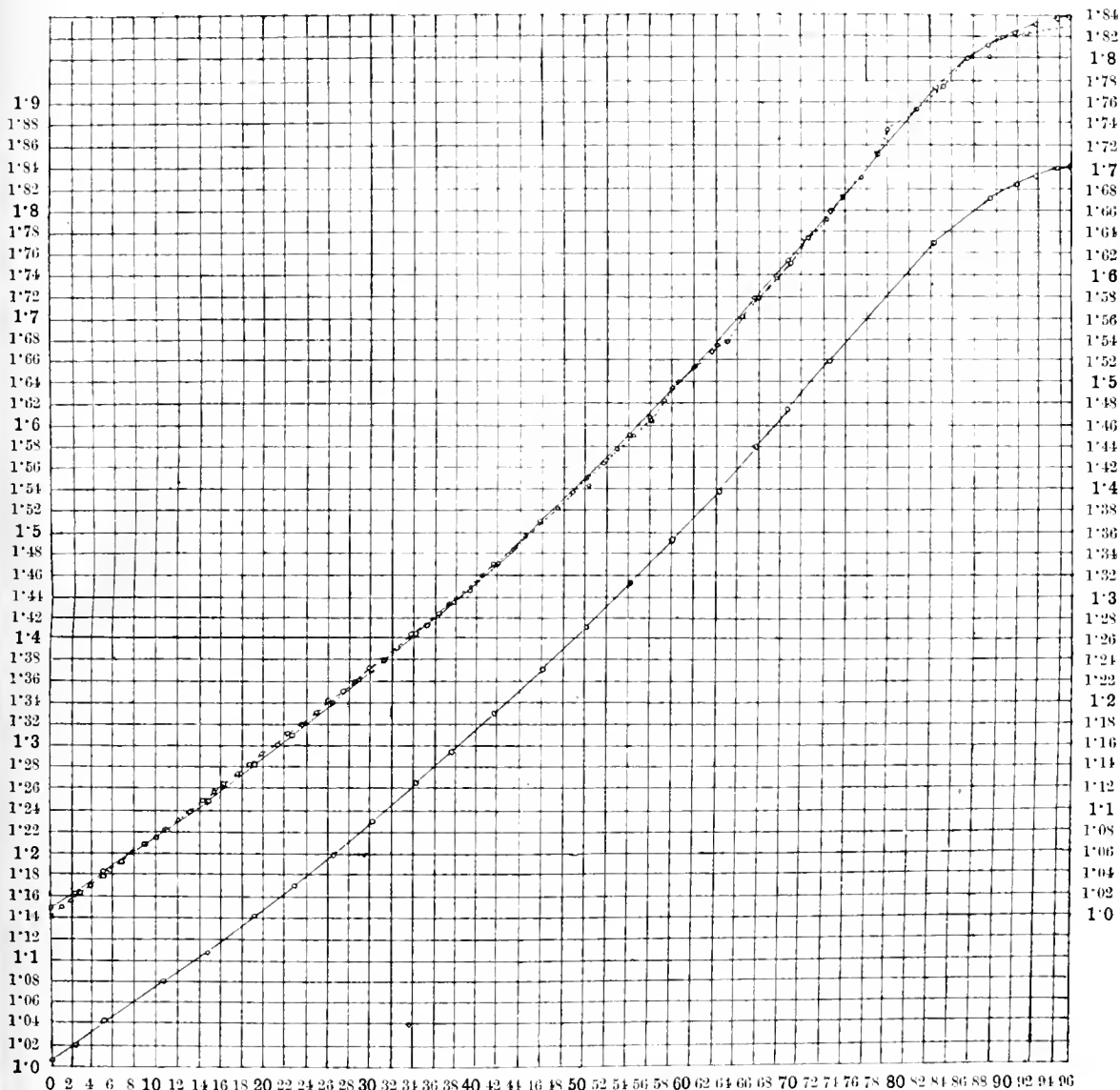
*Recent Determinations of the Specific Gravities of Sulphuric Acids of different Strengths.* G. Lange and M. Isler. *Zeits. f. angew. Chem.* 1890, 129—136.

For a number of years the table of specific gravities given by Kolb has been the one used, as it agrees very well with that of Bincau, previously in general use. Bincau made no determinations over 98.5 per cent. of  $\text{H}_2\text{SO}_4$ , but Kolb gave a result for the pure hydrate,  $\text{H}_2\text{SO}_4$ . By a series of distillations he obtained an acid which he stated contained 99.72 per cent. of the hydrate. This seems to be an error, as other observers have not been able by these means to obtain an acid containing more than 98.5 per cent. However, to this acid Kolb added sulphur trioxide, and heated the mixture to  $200^\circ$  in a sulphuric acid bath to drive off the excess of trioxide; the product so obtained he considered

to be the pure hydrate  $\text{H}_2\text{SO}_4$ , since his analysis of it showed 99.95 per cent. The analysis was gravimetrically performed with barium chloride. Fresenius has shown that in this method of analysis several grave sources of error exist, for in strongly acid solutions the results are apt to be too low, whilst excess of barium chloride has a great tendency to bring out results which are too high. Kolb gives no account of any precautions used. Furthermore, the pure hydrate,  $\text{H}_2\text{SO}_4$ , gives off anhydride at a temperature of  $30-40^\circ$ , and consequently at  $200^\circ$ , the temperature at which the acid was said to have been obtained, such a product is impossible.

It has since been repeatedly shown that the greatest concentration obtainable by heating is about 98.5 per cent., and furthermore that the maximum density is attained with a percentage of 97.5—97.7, from which point up to 100 per cent. the density decreases. On such grounds as these, redetermination of the specific gravity of acids varying in concentration from 90—100 per cent. has been made (Lange

SPECIFIC GRAVITY OF SULPHURIC ACID.



The specific gravities on the left-hand side refer to the lower curve; those on the right-hand side to the upper curve.

— Lange and Isler, at  $15^\circ\text{C}$ . (in vacuo).

- - - Kolb, at  $15^\circ\text{C}$ . (corr.).

and Naef, Chem. Ind. 1883, 37; this Journal, 1883, 279). Nevertheless, the old erroneous table of Kolb for these higher concentrations has been retained without remark by Bolley and Stahlschmidt ("Chemisch-technische Untersuchungen," 1889 edition). Work has also been published on this point in recent years by Kohlrausch, Schertel, Winkler, and Mendeleff. The present publication of the authors deals not only with the acid from 90—100 per cent., but also with acid varying in concentration the whole way below this. The result, as seen from the accompanying curve, is that more carefully conducted experiments have removed the deviations found by Kolb, and have ascertained the densities of acid below 3 per cent. to be decidedly in error. The percentages are given as abscissæ, the specific gravities as ordinates.

For the purpose of the tables given the curve was made on a large scale so that accurate readings were obtained by interpolation. The results are given for intervals of 1 Tw. (intervals in specific gravity of .005) since the Baumé scale and the "rational" Baumé scale in use are so conventional as to be to a certain extent misleading, for the "rational" scale is based on the false assumption that the pure hydrate,  $H_2SO_4$ , has the sp. gr. 1.842 at 15° C.

On the other hand the reading 66 with the sp. gr. 1.842 is by some taken for fuming sulphuric acid. The concentration of this acid, however, varies from 92 to 96 per cent. and its true specific gravity at 15° is 1.830—1.8405, though the commercial product possesses a specific gravity varying with the amount of impurities. Again, if it be assumed that 66° on the "rational" Baumé be equivalent to a specific gravity of 1.842, then 93 per cent. acid is only 65½, and 96 per cent. acid only 65½ B.

In the tables the percentages are given to hundredths, only, however, to enable more accurate determination to the first decimal place.

The values given are for the chemically pure substance. In practice, however, the deviations up to about 1.75 may be neglected; in the case of stronger acids the impurities present are so considerable that the specific gravities are very appreciably higher than the values given, so that the buyer cannot expect an acid of sp. gr. 1.840 to contain 95.6 per cent.

Observations should be made as near 15° C. as possible, or corrected by known means. In the preparation of the tables given, the experiments were made at 15° C. and the results reduced by calculation, to water at 4° C. and vacuum.

SPECIFIC GRAVITY OF SULPHURIC ACID SOLUTIONS AS DETERMINED BY LUNGE AND ISLER.

Sp. Gr. 15° C. at 1° C. (in Vacuum).	Degrees Baumé.	Degrees Twaddell.	100 Parts by Weight correspond to				1 Litre contains in Kilos.			
			Per Cent. $SO_3$ .	Per Cent. $H_2SO_4$ .	Per Cent. 60° Acid.	Per Cent. 50° Acid.	$SO_3$ .	$H_2SO_4$ .	60° Acid.	50° Acid.
1.000	0	0	0.07	0.09	0.12	0.14	0.001	0.001	0.001	0.001
1.005	0.7	1	0.08	0.83	1.03	1.33	0.007	0.008	0.011	0.013
1.010	1.4	2	1.28	1.57	2.01	2.51	0.013	0.016	0.020	0.025
1.015	2.1	3	1.88	2.30	2.95	3.68	0.019	0.023	0.030	0.037
1.020	2.7	4	2.47	3.03	3.88	4.85	0.025	0.031	0.040	0.050
1.025	3.4	5	3.07	3.76	4.82	6.02	0.032	0.039	0.049	0.062
1.030	4.1	6	3.67	4.49	5.78	7.18	0.038	0.046	0.059	0.074
1.035	4.7	7	4.27	5.23	6.73	8.37	0.044	0.054	0.070	0.087
1.040	5.4	8	4.87	5.96	7.64	9.54	0.051	0.062	0.079	0.099
1.045	6.0	9	5.45	6.67	8.55	10.67	0.057	0.071	0.089	0.112
1.050	6.7	10	6.02	7.37	9.44	11.79	0.063	0.077	0.099	0.124
1.055	7.4	11	6.59	8.07	10.34	12.91	0.070	0.085	0.109	0.136
1.060	8.0	12	7.16	8.77	11.24	14.03	0.076	0.093	0.119	0.149
1.065	8.7	13	7.73	9.47	12.14	15.15	0.082	0.102	0.129	0.161
1.070	9.4	14	8.32	10.19	13.05	16.30	0.089	0.109	0.140	0.174
1.075	10.0	15	8.90	10.90	13.96	17.44	0.096	0.117	0.150	0.188
1.080	10.6	16	9.47	11.60	14.87	18.56	0.103	0.125	0.161	0.201
1.085	11.2	17	10.04	12.30	15.76	19.68	0.109	0.133	0.171	0.213
1.090	11.9	18	10.60	12.99	16.65	20.78	0.116	0.142	0.181	0.227
1.095	12.4	19	11.16	13.67	17.52	21.87	0.122	0.150	0.192	0.240
1.100	13.0	20	11.71	14.35	18.39	22.96	0.129	0.158	0.202	0.253
1.105	13.6	21	12.27	15.03	19.26	24.05	0.136	0.166	0.212	0.265
1.110	14.2	22	12.82	15.71	20.13	25.14	0.143	0.175	0.223	0.279
1.115	14.9	23	13.36	16.36	20.96	26.18	0.149	0.183	0.234	0.292
1.120	15.4	24	13.89	17.01	21.80	27.22	0.156	0.191	0.245	0.305
1.125	16.0	25	14.42	17.66	22.63	28.26	0.162	0.199	0.255	0.318
1.130	16.5	26	14.95	18.31	23.47	29.30	0.169	0.207	0.265	0.331
1.135	17.1	27	15.48	18.96	24.29	30.34	0.176	0.215	0.276	0.344
1.140	17.7	28	16.01	19.61	25.13	31.38	0.183	0.223	0.287	0.358
1.145	18.3	29	16.54	20.26	25.96	32.42	0.189	0.231	0.297	0.371
1.150	18.8	30	17.07	20.91	26.79	33.46	0.196	0.239	0.308	0.385

## SPECIFIC GRAVITY OF SULPHURIC ACID SOLUTIONS AS DETERMINED BY LUNGE AND ISLER—continued.

Sp. Gr. 15° C. at 1° C. (in Vacuo).	Degrees Baumé.	Degrees Twaddell.	100 Parts by Weight correspond to				1 Litre contains in Kilos.			
			Per Cent. SO <sub>3</sub> .	Per Cent. H <sub>2</sub> SO <sub>4</sub> .	Per Cent. 60° Acid.	Per Cent. 50° Acid.	SO <sub>3</sub> .	H <sub>2</sub> SO <sub>4</sub> .	60° Acid.	50° Acid.
1.155	19.3	31	17.59	21.55	27.61	34.48	0.203	0.245	0.319	0.398
1.160	19.8	32	18.11	22.19	28.43	35.50	0.210	0.257	0.330	0.412
1.165	20.3	33	18.64	22.83	29.25	36.53	0.217	0.266	0.341	0.426
1.170	20.9	34	19.16	23.47	30.07	37.55	0.224	0.275	0.352	0.439
1.175	21.4	35	19.69	24.12	30.90	38.59	0.231	0.283	0.363	0.453
1.180	22.0	36	20.21	24.76	31.73	39.62	0.238	0.292	0.374	0.467
1.185	22.5	37	20.73	25.40	32.55	40.64	0.246	0.301	0.386	0.481
1.190	23.0	38	21.26	26.04	33.37	41.66	0.253	0.310	0.397	0.496
1.195	23.5	39	21.78	26.68	34.19	42.69	0.260	0.319	0.409	0.511
1.200	24.0	40	22.30	27.32	35.01	43.71	0.268	0.328	0.420	0.525
1.205	24.5	41	22.82	27.95	35.83	44.72	0.275	0.337	0.432	0.539
1.210	25.0	42	23.33	28.58	36.66	45.73	0.282	0.346	0.444	0.553
1.215	25.5	43	23.84	29.21	37.45	46.74	0.290	0.355	0.455	0.568
1.220	26.0	44	24.36	29.84	38.23	47.74	0.297	0.364	0.466	0.583
1.225	26.4	45	24.88	30.48	39.05	48.77	0.305	0.373	0.478	0.598
1.230	26.9	46	25.39	31.11	39.86	49.78	0.312	0.382	0.490	0.612
1.235	27.4	47	25.88	31.70	40.61	50.72	0.320	0.391	0.502	0.626
1.240	27.9	48	26.35	32.28	41.37	51.65	0.327	0.400	0.513	0.640
1.245	28.4	49	26.83	32.86	42.11	52.58	0.334	0.409	0.524	0.655
1.250	28.8	50	27.29	33.43	42.84	53.49	0.341	0.418	0.535	0.669
1.255	29.3	51	27.76	34.00	43.57	54.40	0.348	0.426	0.547	0.683
1.260	29.7	52	28.22	34.57	44.30	55.31	0.356	0.435	0.558	0.697
1.265	30.2	53	28.69	35.14	45.03	56.22	0.363	0.444	0.570	0.711
1.270	30.6	54	29.15	35.71	45.76	57.14	0.370	0.454	0.581	0.725
1.275	31.1	55	29.62	36.29	46.50	58.06	0.377	0.462	0.593	0.740
1.280	31.5	56	30.10	36.87	47.24	58.99	0.385	0.472	0.605	0.755
1.285	32.0	57	30.57	37.45	47.99	59.92	0.393	0.481	0.617	0.770
1.290	32.4	58	31.04	38.03	48.73	60.85	0.400	0.490	0.629	0.785
1.295	32.8	59	31.52	38.61	49.47	61.78	0.408	0.500	0.641	0.800
1.300	33.3	60	31.99	39.19	50.21	62.70	0.416	0.510	0.653	0.815
1.305	33.7	61	32.46	39.77	50.96	63.63	0.424	0.519	0.665	0.830
1.310	34.2	62	32.94	40.35	51.71	64.56	0.432	0.529	0.677	0.845
1.315	34.6	63	33.41	40.93	52.45	65.45	0.439	0.538	0.689	0.860
1.320	35.0	64	33.88	41.50	53.18	66.40	0.447	0.548	0.702	0.876
1.325	35.4	65	34.35	42.08	53.92	67.33	0.455	0.557	0.714	0.892
1.330	35.8	66	34.80	42.66	54.67	68.26	0.462	0.567	0.727	0.908
1.335	36.2	67	35.27	43.20	55.36	69.12	0.471	0.577	0.739	0.923
1.340	36.6	68	35.71	43.74	56.05	69.98	0.479	0.586	0.751	0.938
1.345	37.0	69	36.14	44.28	56.74	70.85	0.486	0.596	0.763	0.953
1.350	37.4	70	36.58	44.82	57.43	71.71	0.494	0.605	0.775	0.968
1.355	37.8	71	37.02	45.35	58.11	72.56	0.502	0.614	0.787	0.983
1.360	38.2	72	37.45	45.88	58.79	73.41	0.509	0.624	0.800	0.998
1.365	38.6	73	37.89	46.41	59.48	74.26	0.517	0.633	0.812	1.014
1.370	39.0	74	38.32	46.94	60.15	75.10	0.525	0.643	0.824	1.029
1.375	39.4	75	38.75	47.47	60.83	75.95	0.533	0.653	0.836	1.044
1.380	39.8	76	39.18	48.00	61.51	76.80	0.541	0.662	0.849	1.060
1.385	40.1	77	39.62	48.53	62.19	77.65	0.549	0.672	0.861	1.075

SPECIFIC GRAVITY OF SULPHURIC ACID SOLUTIONS AS DETERMINED BY LUNGE AND ISLER—*continued*.

Sp. Gr. 15° C. at 4° C. (in Vacuo).	Degrees, Baumé.	Degrees, Twaddell.	100 Parts by Weight correspond to				1 Litre contains in Kilos.			
			Per Cent. SO <sub>3</sub> .	Per Cent. H <sub>2</sub> SO <sub>4</sub> .	Per Cent. 60° Acid.	Per Cent. 50° Acid.	SO <sub>3</sub> .	H <sub>2</sub> SO <sub>4</sub> .	60° Acid.	50° Acid.
1.390	40.5	78	40.05	49.06	62.87	78.50	0.557	0.682	0.873	1.091
1.395	40.8	79	40.48	49.59	63.55	79.34	0.564	0.692	0.886	1.107
1.400	41.2	80	40.91	50.11	64.21	80.18	0.573	0.702	0.899	1.123
1.405	41.6	81	41.33	50.63	64.88	81.01	0.581	0.711	0.912	1.138
1.410	42.0	82	41.76	51.15	65.55	81.86	0.589	0.721	0.924	1.154
1.415	42.3	83	42.17	51.66	66.21	82.66	0.597	0.730	0.937	1.170
1.420	42.7	84	42.57	52.15	66.82	83.44	0.604	0.740	0.949	1.185
1.425	43.1	85	42.96	52.63	67.44	84.21	0.612	0.750	0.961	1.200
1.430	43.4	86	43.36	53.11	68.06	84.98	0.620	0.759	0.973	1.215
1.435	43.8	87	43.75	53.59	68.68	85.74	0.628	0.769	0.986	1.230
1.440	44.1	88	44.14	54.07	69.29	86.51	0.636	0.779	0.998	1.246
1.445	44.4	89	44.53	54.55	69.90	87.28	0.643	0.789	1.010	1.261
1.450	44.8	90	44.92	55.03	70.52	88.05	0.651	0.798	1.023	1.277
1.455	45.1	91	45.31	55.50	71.12	88.80	0.659	0.808	1.035	1.292
1.460	45.4	92	45.69	55.97	71.72	89.55	0.667	0.817	1.047	1.307
1.465	45.8	93	46.07	56.43	72.31	90.29	0.675	0.827	1.059	1.323
1.470	46.1	94	46.45	56.90	72.91	91.04	0.683	0.837	1.072	1.338
1.475	46.4	95	46.83	57.37	73.51	91.79	0.691	0.846	1.084	1.354
1.480	46.8	96	47.21	57.83	74.10	92.53	0.699	0.856	1.097	1.370
1.485	47.1	97	47.57	58.28	74.68	93.25	0.707	0.865	1.109	1.385
1.490	47.4	98	47.95	58.74	75.27	93.98	0.715	0.876	1.122	1.400
1.495	47.8	99	48.34	59.22	75.88	94.75	0.723	0.885	1.134	1.417
1.500	48.1	100	48.73	59.70	76.50	95.52	0.731	0.896	1.147	1.433
1.505	48.4	101	49.12	60.18	77.12	96.29	0.739	0.906	1.160	1.449
1.510	48.7	102	49.51	60.65	77.72	97.04	0.748	0.916	1.174	1.465
1.515	49.0	103	49.89	61.12	78.32	97.79	0.756	0.926	1.187	1.481
1.520	49.4	104	50.28	61.59	78.93	98.54	0.764	0.936	1.199	1.498
1.525	49.7	105	50.66	62.06	79.52	99.30	0.773	0.946	1.213	1.514
1.530	50.0	106	51.04	62.53	80.13	100.05	0.781	0.957	1.226	1.531
1.535	50.3	107	51.43	63.00	80.73	100.80	0.789	0.967	1.239	1.547
1.540	50.6	108	51.78	63.43	81.28	101.49	0.797	0.977	1.252	1.563
1.545	50.9	109	52.12	63.85	81.81	102.16	0.805	0.987	1.264	1.579
1.550	51.2	110	52.46	64.26	82.34	102.82	0.813	0.996	1.276	1.593
1.555	51.5	111	52.79	64.67	82.87	103.47	0.821	1.006	1.289	1.609
1.560	51.8	112	53.12	65.08	83.39	104.13	0.829	1.015	1.301	1.624
1.565	52.1	113	53.46	65.49	83.92	104.78	0.837	1.025	1.313	1.640
1.570	52.4	114	53.80	65.90	84.44	105.44	0.845	1.035	1.325	1.655
1.575	52.7	115	54.13	66.30	84.95	106.08	0.853	1.044	1.338	1.671
1.580	53.0	116	54.46	66.71	85.48	106.73	0.861	1.054	1.351	1.686
1.585	53.3	117	54.80	67.13	86.03	107.41	0.869	1.064	1.364	1.702
1.590	53.6	118	55.18	67.59	86.62	108.14	0.877	1.075	1.377	1.719
1.595	53.9	119	55.55	68.05	87.20	108.88	0.886	1.085	1.391	1.737
1.600	54.1	120	55.93	68.51	87.79	109.62	0.895	1.096	1.405	1.754
1.605	54.4	121	56.30	68.97	88.38	110.35	0.904	1.107	1.419	1.772
1.610	54.7	122	56.68	69.43	88.97	111.09	0.913	1.118	1.432	1.789
1.615	55.0	123	57.05	69.89	89.56	111.82	0.921	1.128	1.446	1.806
1.620	55.2	124	57.40	70.32	90.11	112.51	0.930	1.139	1.460	1.823

## SPECIFIC GRAVITY OF SULPHURIC ACID SOLUTIONS AS DETERMINED BY LUNGE AND ISLER—continued.

Sp. Gr. at 45° C. (in Vacuo).	Degrees Baumé.	Degrees Twaddell.	100 Parts by Weight correspond to				1 Litre contains in Kilos.			
			Per Cent. SO <sub>3</sub> .	Per Cent. H <sub>2</sub> SO <sub>4</sub> .	Per Cent. 60° Acid.	Per Cent. 50° Acid.	SO <sub>3</sub> .	H <sub>2</sub> SO <sub>4</sub> .	60° Acid.	50° Acid.
1·625	55·5	125	57·75	70·74	90·65	113·18	0·938	1·150	1·473	1·840
1·630	55·8	126	58·00	71·16	91·19	113·86	0·947	1·160	1·486	1·857
1·635	56·0	127	58·43	71·57	91·71	114·51	0·955	1·170	1·499	1·873
1·640	56·3	128	58·77	71·99	92·25	115·18	0·964	1·181	1·513	1·889
1·645	56·6	129	59·10	72·40	92·77	115·84	0·972	1·192	1·526	1·905
1·650	56·9	130	59·45	72·82	93·29	116·51	0·981	1·202	1·540	1·922
1·655	57·1	131	59·78	73·23	93·81	117·17	0·989	1·212	1·553	1·939
1·660	57·4	132	60·11	73·64	94·36	117·82	0·998	1·222	1·566	1·956
1·665	57·7	133	60·46	74·07	94·92	118·51	1·007	1·233	1·580	1·973
1·670	57·9	134	60·82	74·51	95·48	119·22	1·016	1·244	1·595	1·991
1·675	58·2	135	61·20	74·97	96·07	119·95	1·025	1·256	1·609	2·009
1·680	58·4	136	61·57	75·42	96·65	120·67	1·034	1·267	1·623	2·027
1·685	58·7	137	61·93	75·86	97·21	121·38	1·043	1·278	1·638	2·046
1·690	58·9	138	62·29	76·30	97·77	122·08	1·053	1·289	1·652	2·064
1·695	59·2	139	62·64	76·73	98·32	122·77	1·062	1·301	1·667	2·082
1·700	59·5	140	63·00	77·17	98·89	123·47	1·071	1·312	1·681	2·100
1·705	59·7	141	63·35	77·60	99·44	124·16	1·080	1·323	1·696	2·117
1·710	60·0	142	63·70	78·04	100·00	124·86	1·089	1·334	1·710	2·136
1·715	60·2	143	64·07	78·48	100·56	125·57	1·099	1·346	1·725	2·154
1·720	60·4	144	64·43	78·92	101·13	126·27	1·108	1·357	1·739	2·172
1·725	60·6	145	64·78	79·36	101·69	126·98	1·118	1·369	1·754	2·191
1·730	60·9	146	65·14	79·80	102·25	127·68	1·127	1·381	1·769	2·209
1·735	61·1	147	65·50	80·24	102·82	128·38	1·136	1·392	1·784	2·228
1·740	61·4	148	65·86	80·68	103·38	129·09	1·146	1·404	1·799	2·247
1·745	61·6	149	66·22	81·12	103·95	129·79	1·156	1·416	1·814	2·265
1·750	61·8	150	66·58	81·56	104·52	130·49	1·165	1·427	1·829	2·284
1·755	62·1	151	66·94	82·00	105·08	131·20	1·175	1·439	1·845	2·303
1·760	62·3	152	67·30	82·44	105·64	131·90	1·185	1·451	1·859	2·321
1·765	62·5	153	67·65	82·88	106·21	132·61	1·194	1·463	1·874	2·340
1·770	62·8	154	68·02	83·32	106·77	133·31	1·204	1·475	1·890	2·359
1·775	63·0	155	68·49	83·90	107·51	134·24	1·216	1·489	1·908	2·381
1·780	63·2	156	68·98	84·50	108·27	135·20	1·228	1·504	1·928	2·407
1·785	63·5	157	69·47	85·10	109·05	136·16	1·240	1·519	1·947	2·432
1·790	63·7	158	69·96	85·70	109·82	137·14	1·252	1·534	1·965	2·455
1·795	64·0	159	70·45	86·30	110·58	138·08	1·265	1·549	1·983	2·479
1·800	64·2	160	70·94	86·90	111·35	139·06	1·277	1·564	2·004	2·503
1·805	64·4	161	71·50	87·60	112·25	140·16	1·291	1·581	2·026	2·530
1·810	64·6	162	72·08	88·30	113·15	141·28	1·305	1·598	2·048	2·558
1·815	64·8	163	72·69	89·05	114·11	142·48	1·319	1·621	2·071	2·587
1·820	65·0	164	73·51	90·05	115·33	144·08	1·338	1·639	2·099	2·622
1·821	..	..	73·63	90·29	115·59	144·32	1·341	1·643	2·104	2·628
1·822	65·1	..	73·80	90·40	115·84	144·64	1·345	1·647	2·110	2·635
1·823	..	..	73·96	90·60	116·10	144·96	1·348	1·651	2·116	2·643
1·824	65·2	..	74·12	90·80	116·35	145·28	1·352	1·656	2·122	2·650
1·825	..	165	74·29	91·00	116·61	145·60	1·356	1·661	2·128	2·657
1·826	65·3	..	74·49	91·25	116·93	146·00	1·360	1·666	2·135	2·666
1·827	..	..	74·69	91·50	117·25	146·40	1·364	1·671	2·142	2·675

## SPECIFIC GRAVITY OF SULPHURIC ACID SOLUTIONS AS DETERMINED BY LUNGE AND ISLER—continued.

Sp. Gr. 15° C. at 1° C. (in Vacuo).	Degrees Baumé.	Degrees Twaddell.	100 Parts by Weight correspond to				1 Litre contains in Kilos.			
			Per Cent. SO <sub>3</sub> .	Per Cent. H <sub>2</sub> SO <sub>4</sub> .	Per Cent. 60° Acid.	Per Cent. 50° Acid.	SO <sub>3</sub> .	H <sub>2</sub> SO <sub>4</sub> .	60° Acid.	50° Acid.
1·828	65·4	..	74·86	91·70	117·51	146·72	1·368	1·676	2·148	2·682
1·829	..	..	75·03	91·90	117·76	147·04	1·372	1·681	2·154	2·689
1·830	..	166	75·19	92·10	118·02	147·36	1·376	1·685	2·159	2·696
1·831	65·5	..	75·35	92·30	118·27	147·68	1·380	1·690	2·165	2·704
1·832	..	..	75·53	92·52	118·56	148·03	1·384	1·695	2·172	2·711
1·833	65·6	..	75·72	92·75	118·85	148·40	1·388	1·700	2·178	2·720
1·834	..	..	75·96	93·05	119·23	148·88	1·393	1·706	2·186	2·730
1·835	65·7	167	76·27	93·43	119·72	149·49	1·400	1·713	2·196	2·743
1·836	..	..	76·57	93·80	120·19	150·08	1·406	1·722	2·207	2·755
1·837	..	..	76·90	94·20	120·71	150·72	1·412	1·730	2·217	2·769
1·838	65·8	..	77·23	94·60	121·22	151·36	1·419	1·739	2·228	2·782
1·839	..	..	77·55	95·00	121·74	152·00	1·426	1·748	2·239	2·795
1·840	65·9	168	78·04	95·60	122·51	152·96	1·436	1·759	2·254	2·814
1·8405	..	..	78·33	95·95	122·96	153·52	1·441	1·765	2·262	2·825
1·8410	..	..	79·19	97·00	124·30	155·20	1·458	1·786	2·288	2·857
1·8415	..	..	79·76	97·70	125·20	156·32	1·469	1·799	2·305	2·879
1·8410	..	..	80·16	98·20	125·84	157·12	1·476	1·808	2·317	2·893
1·8405	..	..	80·57	98·70	126·48	157·92	1·483	1·816	2·328	2·906
1·8400	..	..	80·98	99·20	127·12	158·72	1·490	1·825	2·339	2·920
1·8395	..	..	81·18	99·45	127·44	159·12	1·494	1·830	2·344	2·927
1·8390	..	..	81·39	99·70	127·76	159·52	1·497	1·834	2·349	2·933
1·8385	..	..	81·59	99·95	128·08	159·92	1·500	1·838	2·355	2·940

—T. L. B.

*On the Production of Sulphuretted Hydrogen from Solutions of Calcium Sulph-hydrate by means of Steam.*  
B. Deutecom and F. Roth. Chem. Ind. **13**, 25—28.

A DETAILED account of experiments on a semi-industrial scale illustrated with tables showing the course of the reactions and the quantitative results obtained. On heating a solution of calcium sulph-hydrate to boiling, sulphuretted hydrogen is evolved and crystalline calcium hydrate deposited. When the solution has a density above 20° B. scarcely any steam is evolved until that density is reached, whilst solutions of less density give off steam only until 20° B. is reached, the result being that steam, sulphuretted hydrogen, and calcium hydrate are formed simultaneously only when the boiling solution has a density of 20° B. and the density of the filtered liquid is preserved until the whole is evaporated to dryness and all the sulphur is given off. Miller and Opl found that this decomposition is partially accomplished on passing steam into the solution, and Hasenclever (this Journal, 1890, 182) has worked out this reaction on a practical scale. The results, however, were obtained with too large an expenditure of steam, and the author, in proceeding on the same lines, has arrived at a like conclusion. The solutions of calcium sulphhydrate are obtained by passing steam through a series of receptacles containing vat waste and water, and the author has treated such solutions with steam superheated to 140° to 160°, and under a pressure of 5 to 6 atmospheres. The sulphuretted hydrogen evolved was absorbed by milk of lime for the purpose of measurement. In one series of experiments 100 parts of sulphur were evolved as sulphuretted hydrogen at an expenditure of 678 parts of coal used for generating the steam. Calculations based on the thermo-chemical conditions of the reaction show that the heat required for the production of 1 part of sulphur should be furnished by 1·51

parts of steam, but as the heat supplied in this way has a low potential, the practical results necessarily fall much below the theoretical. More favourable results were obtained with an expenditure of 20 parts of steam or 3·1 parts of coal for 1 part of sulphur evolved, but the yield is still far below that required for profitable working. A further drawback is the length of time taken by the reaction, and its incompleteness, as the solutions are only partially denuded of sulphur. The deposited lime was found to consist of 72 per cent. of Ca(OH)<sub>2</sub>, 20 per cent. of CaCO<sub>3</sub>, 0·5 per cent. of CaSO<sub>4</sub>, and 7 per cent. of H<sub>2</sub>O, and was far inferior to ordinary slaked lime for the production of bleaching powder or for causticising soda liquors.—G. H. B.

*The Degree of Exhaustion of Gases.* K. W. Jurisch.  
Chem. Ind. **13**, 46—48.

WHEN a stream of gas is used for the purpose of carrying out a chemical reaction, and no foreign gas is mixed with it during the process, the degree of exhaustion (*Ausnutzungsgrad*) may be found from the amount of the active constituent contained in the in-coming and out-going streams respectively. Suppose, for example, that the carbonic acid from a limekiln is employed for the purpose of precipitating out sodium bicarbonate in the ammonia-soda process, or magnesium bicarbonate in the manufacture of magnesia, &c., or for the purpose of converting solid substances into carbonates or bicarbonates: the reaction takes place by an abstraction of carbonic acid from the stream of gas, so that it is poorer in carbonic acid after the process.

Let V denote the percentage (by volume) of carbonic acid originally present in the limekiln gas, and the percentage by v (again by volume) of carbonic acid in the gas after the reaction. The degree of exhaustion of the

carbonic acid (a factor of considerable importance in following the course of the process) is in many factories wrongly calculated on the assumption that the difference  $V - v$  represents the volume of the carbonic acid absorbed. On this assumption the degree of exhaustion would be simply given by the proportion—

$$V : V - v = 100 : n$$

$$\text{and } \therefore n = \frac{100(V - v)}{V}$$

This, however, is incorrect;  $v$  does not relate to the same volume as  $V$ ; for a reduction in the volume of the gas has been caused by the absorption of some of the carbonic acid.

Let us follow out more closely the process, beginning with 100 volumes of the limekiln gas. This contains  $V$  volumes of carbonic acid and  $(100 - V)$  volumes of inert gases which pass through the process unchanged. That these latter are of service in certain ways is true, but this does not concern us at present. A portion of the volume  $V$  of carbonic acid is absorbed: suppose  $x$  to denote the volume of carbonic acid left. The bulk of the original gas has now been reduced in the proportion of 100 to  $R$ , where—

$$R = 100 - V + x$$

The gas now contains only  $x$  volumes of carbonic acid to  $(100 - V)$  volumes of inert gases. If (as before) its volume-percentage of carbonic acid be denoted by  $v$ , then it may be stated to contain  $(100 - v)$  volumes of inert gases to  $v$  volumes of carbonic acid. These facts may be tabulated as below:—

Components.	In-going Gas.	Out-going Gas.	
	Volume.	Volume.	Percentage Composition by Volume.
Carbonic acid...	$V$	$x$	$v$
Inert gases.....	$100 - V$	$100 - V$	$100 - v$
Gas.....	100	$100 - V + x$	100

It is now evident that  $x$  can be calculated from the proportion—

$$100 : v = (100 - V + x) : x$$

$$\text{and } \therefore x = \frac{100 - V}{100 - v}$$

The reduction in volume is therefore in the ratio of 100 to  $R$ , where—

$$R = 100 - V + \frac{100 - V}{100 - v} \cdot v = 100 \cdot \frac{100 - V}{100 - v}$$

$R$  also represents the volume of the out-going gas as a percentage of that of the in-going gas.

If from the volume  $V$  of the carbonic acid originally present be subtracted the volume  $x$  of that which remains, there is obtained for the volume of the absorbed carbonic acid the expression—

$$V - x = V - \frac{100 - V}{100 - v} \cdot v = 100 \cdot \frac{V - v}{100 - v}$$

The degree of exhaustion ( $y$ ) of the carbonic acid in the form of a percentage of the amount employed, may then be found the proportion—

$$V : \frac{100(V - v)}{100 - v} = 100 : y$$

$$\text{and } \therefore y = 100 \cdot \frac{100(V - v)}{(100 - v)V}$$

This formula is clearly applicable to any other case in which a gas suffers partial absorption (e.g. for chlorine in the manufacture of chlorine products), but the volume of the inert gases must remain constant during the process.

The writer discusses the difference between the true value ( $y$ ) of the degree of exhaustion and the incorrect, but commonly-used, value ( $n$ ), and proceeds to find when this difference (i.e., the error) reaches its maximum. Calculations and tabulated results are given for dilute chlorine gas (Hunter-Deacon process, where  $V = 10$  per cent.), for strong Weldon gas ( $V = 50$  per cent.), for limekiln gases ( $V = 30$  per cent. of carbonic acid), and for strong carbonic acid such as is obtained by decomposing limestone with hydrochloric acid or by heating sodium bicarbonate ( $V = 90$  per cent.). For these the original paper must be referred to. It is shown that the error is greatest when the mixture is richest in the active constituent, and when the diminution in volume on account of the absorption is greatest.—D. E. J.

#### *On the Manufacture of Anhydrous Sulphuric Acid in France.* Chem. Ind. **13**, 48—49.

ANHYDROUS sulphuric acid is now made on the manufacturing scale in the Malétra works at Quevilly by Lunge's patent freezing process. The firm referred to holds the monopoly of the use of this process in France, and an account of the method of working it has been given by their chemists, Osterburger and Capelle (this Journal, 1890, 390—391).

The concentrated acid of commerce usually contains only 90—93 per cent. of pure acid, and as stronger acid is now frequently required for manufacturing purposes, it has latterly been produced (up to 97—98 per cent.) by concentration in platinum vessels, of which specimens were shown in Class 51 at the Paris Exhibition. It has now been found at the Malétra works that Lunge's freezing process can be satisfactorily carried out on the large scale. The commercial acid is first concentrated in platinum vessels, and it is advisable to bring it up to a strength of 96—97 per cent., for the mother-liquors from the process are then found to contain as much as 94—95 per cent. of  $\text{H}_2\text{SO}_4$ . The acid is cooled and transferred to a reservoir standing at a higher level than the "filling apparatus." This is made of iron, and is divided by vertical partitions into a number of compartments, each connected to a cell of the cooling apparatus beneath. (Each cell contains about  $12\frac{1}{2}$  kilos. of acid.) The cooling is effected by means of a solution of calcium chloride kept at a temperature of  $-20^\circ$  by an ice machine. In this solution are placed the iron cells.

In order to empty the cells containing the frozen acid, they are placed for a very short time in hot water, so as to loosen the crystals from the sides of the cells. The crystals are then separated from the strong mother-liquor, and placed in an enamelled reservoir, which is heated by steam or hot water. From this the melted acid is transferred directly to vessels suitable for transport.

No information is given respecting cost of manufacture. If this be not too high the process should be of great value in preparing concentrated acid suitable for the manufacture of nitroglycerin and celluloid, for dissolving indigo, purifying mineral oils, &c.—D. E. J.

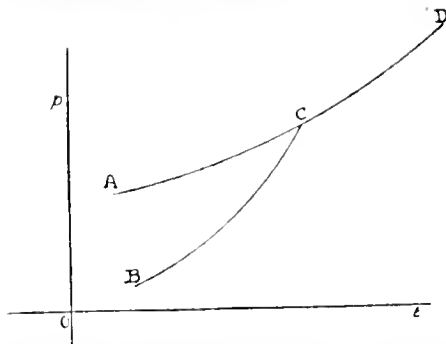
#### *The Employment of Atmospheric Oxygen.* G. Kassner. Chem. Ind. **13**, 104—106 and 120—123.

See this Journal, 1890, 187—188.

#### *On the Compounds of the Alkali Metals with Ammonia.* H. W. Bakhuis-Roozchoom. Compt. Rend. **110**, 134—137.

A CRITICISM of the researches on sodammonium (Compt. Rend. **109**, 900) by Joannis, who found that the saturated solution of sodammonium in liquid ammonia had a vapour tension equal to the tension of dissociation of the solid sodammonium. The author declares this to be impossible except at one definite temperature  $C$ , the point of intersection of two distinct curves representing, (1), A D the variation of vapour pressure with temperature of a heterogeneous system composed of solid sodammonium, a solution of sodammonium in liquid ammonia, and ammonia gas. (2), B C the variation of vapour pressure with temperature of a system composed of sodium, solid sodammonium and ammonia gas intersect in a point.





He compares Joannis's observations with those of Lescœur (Recherches sur la dissociation des hydrates salins, Lille, 1888) on solutions of the hydrates  $\text{SrCl}_2 \cdot 6 \text{H}_2\text{O}$ , and  $\text{Na}_2\text{HPO}_4 \cdot 7 \text{H}_2\text{O}$ , and with his own researches on the hydrates of calcium chloride. References given for the author's general theory to Rec. Trav. Chim. Pays Bas., t. III.—VII and VIII., 1—146. He predicts that at a temperature above C. solid sodammonium will decompose into the system:—solid sodium + ammoniacal solution of sodammonium + ammonia.—P. J. H.

*On the Compounds of the Alkali Metals with Ammonia.*  
Joannis. Compt. Rend. 110, 238—240.

AN answer to the note by Roozeboom (see preceding abstract). The author confirms the results previously obtained at  $0^\circ$ : (1) by observing the vapour pressures with greater accuracy; (2) by showing that at that temperature the saturated solution of sodammonium in liquid ammonia cannot have a higher vapour tension than the solid sodammonium, since this solution does not act on metallic sodium to form the solid sodammonium. He, moreover, disproves the accuracy of Roozeboom's prediction that there exist two curves which intersect only in one point, by showing that sodammonium and its saturated solution possess identical vapour tensions between  $-10^\circ$  and  $+22.4^\circ$ .

The saturated solution was never found to decompose directly into ammonia and metallic sodium in the experiments made. But it is quite possible that this may occur at a temperature beyond the highest one as yet employed ( $+26^\circ$  with a pressure of 426 cm.).

Potassammonium and its saturated solution in liquid ammonia have the same vapour pressure at  $0^\circ$  and  $+8.41^\circ$ . (See also, for the theoretical discussion, Lescœur, Compt. Rend. 110, 275—277.)—P. J. H.

*On the Oxides of Manganese. 1st Part. The Psilomelanes and Wads.* A. Gorgeu. Compt. Rend. 110, 247—249.

FROM the examination of a certain number of wads and psilomelanes, the author concludes that these minerals are to be considered as true hydrated acid manganites, the composition of the most characteristic specimens being represented by the formula  $(3 \text{MnO}_2 \cdot \text{RO} + 1 \text{ to } 3 \text{H}_2\text{O})$ , where R stands for Mn, Ba, Ca, K, &c. They are extremely stable salts; repeated treatment with boiling dilute nitric acid only extracts a small quantity of the base from the finely powdered mineral.—P. J. H.

*Chromium Oxides and Salts.* Prud'homme. Bull. Soc. Ind. Mulhouse, 1889, 599—609.

See under VI., page 499.

PATENTS.

*Improvements in and connected with the Obtainment of Chlorine and Sulphate of Lime.* A. Campbell, London, and W. Boyd, Glasgow. Eng. Pat. 3407, February 26, 1889. 6d.

AFTER the evolution of chlorine in the ordinary way by heating a mixture of hydrochloric acid, peroxide of manganese,

and water, a solution of chloride of manganese remains. The invention consists in obtaining a large additional quantity of chlorine from this chloride of manganese, and from a further quantity of chloride of manganese obtained by treating calcium chloride with sulphate of manganese, and in combining "the various processes in such a manner as to economically obtain a large aggregate production of chlorine whilst repeatedly reconverting and re-using the manganese and making sulphate of lime as a useful by-product."

Chloride of calcium from either the Weldon or Dunlop manganese recovery process, or obtained in any other suitable known way, is treated with a nearly equivalent quantity of sulphate of manganese, preferably keeping the mixture neutral or slightly acid so that the sulphate of lime which is precipitated may be as pure as possible. This precipitate is allowed to settle, and then is filtered, washed, and dried.

Of the solution of chloride of manganese thus obtained, about 60—70 per cent. are mixed with the neutralised residual liquor from ordinary chlorine stills and is worked up for the recovery of the manganese it contains either by the Weldon or Dunlop process. "Instead of using simple milk of lime in the Weldon re-oxidising process, we prefer to mix the lime after slaking in the solution of chloride of calcium, in which it dissolves to some extent, thereby obtaining a thinner liquor or one with less water for a given quantity of lime, and diminishing the quantity of fuel required for concentrating solutions. Or we may use milk of lime formed with washings from other parts of the processes."

The remaining 30—40 per cent. of chloride of manganese solution is concentrated to a suitable density such as  $60^\circ$ — $70^\circ$  Tw., and after mixing with an equivalent proportion of mud which has been washed free from calcium compounds, is run into a still where it is heated with a quantity of sulphuric acid equivalent to the chlorine present; all or nearly all the chlorine is liberated, and sulphate of manganese is left in solution. This sulphate of manganese is neutralised with washed peroxide of manganese mud, and after settling, the clear liquor is used for treating chloride of calcium as above described.

In the operation by which chlorine is liberated by heating the mixture of sulphuric acid and chloride of manganese with manganese mud, it is necessary that the mud and chloride should be freed, as far as possible, from calcium compounds. To effect this, mud from the Weldon or Dunlop processes is treated in a chlorine still with an excess of hydrochloric acid sufficient to dissolve all the calcium present. After driving off the chlorine, as in the ordinary process, the liquor is drawn off, allowed to settle, and may then be used in the manganese-recovery parts of the processes, or may be mixed with sulphate of manganese to form sulphate of calcium. By a modification of this method of working instead of running the mud containing calcium into the chlorine still, it may be treated with chloride of manganese liquor sufficient to convert most of the lime present into chloride of calcium; the mud after settling is then treated with the residuary liquor from the stills, so that the free acid remaining in such liquor may convert any remaining lime into chloride of calcium. After drawing off the clear liquor the remaining mud is filtered, pressed, and washed to render it practically free from calcium chloride; the weak washings from the precipitated sulphate of calcium which contain chloride of manganese may be advantageously used for washing the mud. The manganese cakes obtained are then mixed with a solution of chloride of manganese and run into a still along with sulphuric acid. The peroxide is preferably used in excess, so that the residual liquid is neutral, and this excess can, after expelling the chlorine, be separated by settling or filtering and added to fresh material.

Impurities which tend to accumulate are from time to time separated by precipitation from a suitable proportion of the residual liquor of the stills in which the first portion of the chlorine is evolved, and the purified liquor is then returned to the remainder.—H. S. P.

*An Improved Process and Apparatus for the Manufacture and Transport of Liquid Chlorine.* J. B. Hamay, Loch Long. Eng. Pat. 4037, March 7, 1889. 8d.

The inventor produces "gaseous chlorine in any known manner under a little pressure, sufficient to cause it to bubble through water contained and agitated in a series of vessels which are kept cooled to about freezing point. In each of these vessels, through which the chlorine passes successively, a portion of the chlorine forms a combination with water, producing a crystalline deposit." This deposit forms most rapidly in the first vessel, and more slowly in succeeding ones. When a sufficient amount of deposit has formed in the first vessel, it is disconnected, and the second vessel then becomes the first in the series, and so on. The crystalline deposit is removed and freed from water by squeezing, and then charged into a strong vessel with a funnel-shaped bottom, having a small outlet. On closing this vessel and applying heat externally, the chlorine separates from the water with which it was combined, and subsides in the liquid condition to the bottom, while the water floats above it. "To the bottom of the separating vessel (preferably with an intermediate vessel which may be charged with sulphuric acid to trap any water accompanying the chlorine) is connected one of the holders in which the liquid chlorine is charged for transport." The holder is a thin, strong, steel tube, closed at one end, and fitted at the other end with a projecting boss, into which a plug can be screwed. This plug, before the holder is charged, is contained in a tube attached to the boss, this tube being connected to the separating vessel by a lateral branch provided with a valve. The valve being opened, the liquid chlorine passes into the holder; the plug is then screwed home, and the holder detached. When it is desired to use part of the chlorine contained in the holder, a branch tube, similar to that employed for charging, may be connected to the boss, and the plug can be unscrewed. Drawings of the apparatus accompany the specification.

—H. S. P.

*Improvements in the Manufacture of Anhydrous Chloride of Magnesium and in the Production of Chlorine therefrom.* W. L. Wise, London. From Solvay and Co., Brussels, Belgium. Eng. Pat. 4417, March 13, 1889. 6d.

A common feature in all the processes for obtaining chlorine from magnesium chloride is the heating in a current of air of a mixture of chloride of magnesium and magnesia in the solid state. The chief difficulty in these processes is caused by the pulverulent condition assumed by the material during its decomposition; further, a mass of magnesia is treated which merely serves as a support and does not itself yield chlorine. By this invention these disadvantages are overcome by decomposing anhydrous chloride of magnesium in the fused state by a current of hot air. The process comprises two stages; 1, the preparation of anhydrous magnesium chloride; 2, the production therefrom of chlorine.

The anhydrous magnesium chloride is prepared from the magnesium chloride of commerce, which is a concentrated solution, or from the crystallised salt  $MgCl_2 \cdot 6H_2O$ . By heating this salt at a temperature below  $120^\circ C$ . it can be brought to contain about 80 per cent. of anhydrous salt without fusing and without giving off hydrochloric acid. When once brought to this condition it will stand a still higher temperature without fusing or giving off  $HCl$ , and almost the whole of the water of crystallization can be expelled; but during the first part of this operation the point of fusion is very nearly reached so that the desiccation requires considerable care, and in order to render the operation more practicable, the following process is adopted: A hot concentrated solution of hydrated magnesium chloride is mixed with 50 per cent. of anhydrous  $MgCl_2$ . On cooling this gives a compact mass which is broken into pieces and heated in a vertical and continuous action apparatus by a current of hot air. In this way it can be heated to  $300^\circ$  or  $400^\circ C$ . without melting, whilst the original salt would melt at  $115^\circ C$ . The anhydrous chloride may be also mixed with the hydrated crystallized salt instead of with a solution

of it. The current of air employed is first dried by suitable means such as sulphuric acid or chloride of calcium, and it may be made to circulate through the apparatus so as to be used over and over again; as it merely acts as a vehicle for carrying off water, any other suitable gas may be used instead of air if required.

To obtain chlorine from the anhydrous magnesium chloride this salt is fused and subjected at a red heat to the action of a current of air. It is considered advantageous to melt the magnesium chloride in a crucible or in a vessel resembling a Bessemer converter, either by heating from the outside or by the heat supplied by the current of air which enters by a lower tuyere or a "bubbling tube." In any case the current should not enter through the bottom of the vessel as a zone of quiet fusion should be left underneath the air inlet. The magnesia formed by the action of the air on the molten chloride is dense and collects on the bottom of the vessel whence it can be removed by scooping it out or drawing it off through a tap hole. The magnesia thus obtained is contaminated with a small quantity of magnesium chloride from which, if desired, it may readily be freed by washing.

The current of chlorine obtained contains 15 to 20 per cent. of chlorine, and this percentage is maintained invariable from the beginning to the end of the operation.—H. S. P.

*Improvements in the Treatment of Black Ash and Alkali Waste to obtain Sulphuretted Hydrogen, Sulphur, and Cement, and in Apparatus employed therein.* James Hargreaves, T. Robinson, and John Hargreaves, Widnes. Eng. Pat. 6968, April 26, 1889. 8d.

The apparatus, which is made of iron, is a combination of black-ash lixiviating tanks and of carbonators, the two

Fig. 1.

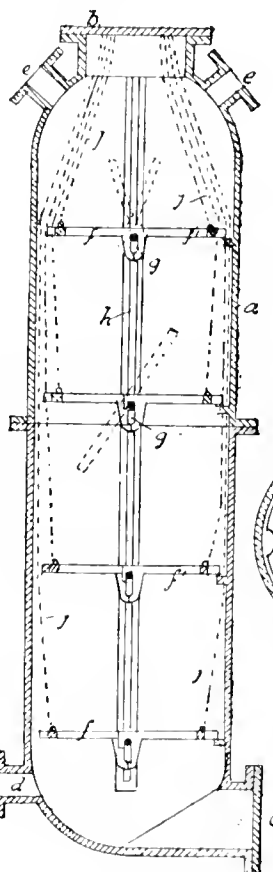
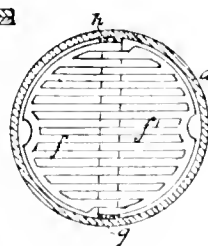


Fig. 2.



operations being carried out in the one vessel. The black ash is supported on movable perforated grids, by which means both the lixiviation and the carbonation are facilitated. In Figs. 1 and 2 *a* is the vessel, *b* the charging door, *c* the discharging door, *d* inlet for gas, and *e* exit for gas, *f, f'* grids supported on bars *g*, which work in grooves *h*; the grids can be raised or lowered by the chains *j*. Instead of grids perforated plates may be employed.

The vessels can also be arranged in varying series, or entirely isolated. Sufficient  $\text{Na}_2\text{O}$ , from about  $1\frac{1}{2}$  to  $2\frac{1}{2}$  per cent., is left in the black ash to dissolve out at the proper time the various sulphur compounds. Carbonic acid at a pressure of about 10 lb. per square inch is forced in through the alkali waste, which is kept moist. When strong sulphuretted hydrogen is required the inert gases are allowed to escape into the atmosphere, when, on the other hand, dilute sulphuretted hydrogen is to be utilised for the production of sulphur or sulphurous acid, then this and the air to be supplied to it are first heated before combination. The heat evolved in the act of combustion is employed for heating the gases as above to a temperature of about  $400^\circ\text{F}$ . Heated air is now blown in to oxidise the residual sulphur compounds; then water added and steam injected to heat the paste thus formed. The sodium carbonate is causticised and the sulphur dissolved out. The residual carbonate of lime is mixed with clay and converted into cement.—S. G. R.

#### *Improvements in Apparatus for Treating Gases by Liquids.*

E. Edwards, London.—From G. Lunge, Zurich, Switzerland, and L. Rohrmann, Kranschwitz, Prussia. Eng. Pat. 6989, April 26, 1889. 8d.

THE improvements relate to the "plate columns" already described in this Journal, 1886, 468; 1887, 538, 584, and 597, and consist in making the "jacket surrounding the plate column" of metal, stone, or other suitable material. The perforated plates are made of earthenware and the annular supports or brackets are either made of one solid piece or in segments and have supporting webs or ribs, the segments being butt- or lap-jointed or provided with grooves to be filled up by lead or other suitable substance.

—O. H.

*Improvements relating to the Recovery of Soda from Slags containing the same.* "The Alkaline Reduction Syndicate, Limited," and A. B. Cunningham, Hebburn-on-Tyne. Eng. Pat. 7733, May 8, 1889. 4d.

See under X., page 519.

*Improvements in the Manufacture of Soda Alum.* E. Augé, Montpellier, France. Eng. Pat. 16,711, October 22, 1889. 6d.

To a solution of sulphate of alumina in slight excess sulphate of soda in the equivalent proportion and in the anhydrous condition is added and the solution concentrated by steam until it attains a density of from 1.38 to 1.45. It is then poured out upon tables and allowed to remain until it becomes pasty, when it is carefully spread out upon inclined tables and allowed to crystallise, the temperature being kept at about  $60^\circ\text{F}$ . In the course of two or three days a further change takes place in the crystals with the separation of more mother-liquor, which removes with it most of the iron and other remaining impurities. The alum is washed in a centrifugal machine and kept in a cool place. The inventor also points out that soda alum thus kept is not efflorescent, that its solubility is about one-third of that usually given, and that a solution of soda alum which has been boiled will still crystallise.—S. G. R.

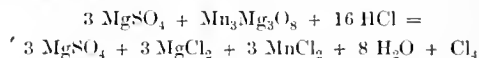
*Processes for the Production of Chlorine and of obtaining Concentrated Gaseous Hydrochloric Acid for that Purpose.* P. de Wilde and A. Reyckler, Brussels, Belgium. Eng. Pat. 17,272, October 31, 1889. 6d.

In this patent a number of methods are described for obtaining chlorine by passing first gaseous hydrochloric acid

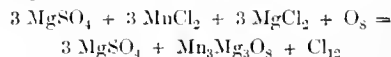
and then air over certain substances heated to redness in a system of cylinders of cast-iron or earthenware and communicating with each other like the cylinders in the sulphate process of Hargreaves. The gas given off when the air is being passed through the cylinders contains from 20 to 25 per cent. of chlorine, and may be directly used for making hypochlorites and chlorates. Instead of using hydrochloric acid and then air a mixture of these gases such as is obtained from sulphate furnaces may be used. In this case the operation becomes continuous, but the gases which come from the cylinders are less rich in chlorine and contain hydrochloric acid and steam which have to be removed by known processes before the chlorine can be used for making hypochlorites and chlorates. The following are the substances mentioned in the patent to be treated as above described:—

1. A calcined mixture of sulphate of magnesium and of magnesia, which is obtained as a residue of "solid porous consistency" from a process patented in France and Germany by one of the patentees. At a dark red heat gaseous hydrochloric acid acting on this mixture converts it into a mixture of magnesium sulphate, magnesium chloride, and magnesium oxychloride. On passing heated air over the last mixture the chloride and oxychloride are decomposed, chlorine is liberated, and a mixture of magnesium sulphate and of magnesia is regenerated.

2. A mixture of magnesium sulphate and manganite of magnesium ( $\text{Mn}_2\text{Mg}_3\text{O}_7$ ), which is made by heating in a muffle furnace equivalent quantities of magnesium sulphate, magnesium chloride, and manganese chloride, all three hydrated. The action of the hydrochloric acid gas on this mixture is as follows:—



On passing a current of dry air over this mixture of substances at a dull red heat chlorine is disengaged, and the mixture of magnesium sulphate and magnesium manganite is regenerated as follows:—



3. A mixture of magnesium sulphate and intermediate oxide of manganese ( $\text{Mn}_2\text{O}_3$ ) obtained by calcining a mixture of magnesium sulphate and manganese chloride. Magnesium chloride alone and manganese chloride alone, or a mixture of the two, fuse before reaching a dull red heat, but the presence of magnesium sulphate prevents this and facilitates the decomposing action of the air.

4. In the processes above referred to, instead of using magnesium sulphate, the sulphate of an alkaline earth or a not readily fusible phosphate, arseniate, borate, or silicate of magnesium or of an alkaline earth, or alumina or silicate of aluminium, or sand or any other not readily fusible substance, which has no action on magnesia, manganite of magnesium, or intermediate oxide of manganese, may be used.

5. Further, a claim is made for "the addition of sulphate of magnesium or of sulphate of an alkaline earth to chlorides of metals to promote their decomposition by dry air at high temperature with liberation of chlorine."

The gaseous hydrochloric acid used in these operations alternately with air should be as concentrated as possible. It is taken from the pan of a sulphate furnace, or obtained by treating commercial hydrochloric acid with sulphuric acid of 60 B. The two acids are simultaneously introduced into the top of a lead tower four or five metres high filled with coke. Gaseous hydrochloric acid is given off and dilute sulphuric acid is drawn off at the bottom of the tower and can be again concentrated.—H. S. P.

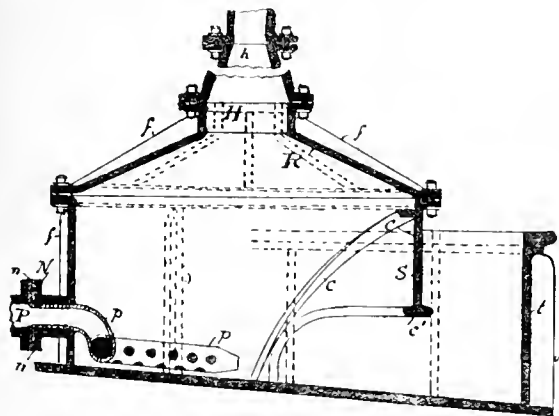
*Improvements in the Manufacture of Anhydrous Barium Oxides.* H. H. Leigh, London. From T. de Brochoeki, Paris, France. Eng. Pat. 1300, January 24, 1890. 4d.

Barium sulphate is converted into the chloride by roasting with calcium chloride "either alone or in combination with coal and calcium carbonate," and the mass is extracted with

water. The barium is then precipitated as carbonate with ammonium carbonate. The precipitate is made up into balls or briquettes with coal dust and calcined, barium oxide being the result which can now be readily converted into the dioxide, hydrate, or other compounds. During the final calcination the muffle may be kept filled with some neutral gas, to prevent the passage inwards of carbonic acid from the furnace gases and from the air.—S. G. R.

*Improvements in and connected with Saturators for Sulphate of Ammonia Plant.* G. Kennedy, East Ham. Eng. Pat. 1653, January 30, 1890. 6d.

THE saturators, instead of being made of wood lined with lead, are entirely of lead or of an alloy of lead and antimony, and are cast preferably in only two pieces. In order to



support the curtain C, which is the part most apt to fail in the saturators of ordinary construction, an arched rib *c* is cast solid with C and the side walls *t t*, and this may be also further strengthened by a bead *c'*. The cover *R* is either dome- or cone-shaped. *H* is a flanged aperture, and bolted to it is *h*, which can either serve as a sulphuretted hydrogen exit pipe or may be entirely removed and the aperture used as a manhole. The joint at *N* is also patented. *N*, which is cast with the saturator, is furnished with a packing ring, *n*, of asbestos. The inlet pipe for the ammonia, *p*, is made of lead, and is expanded over the asbestos packing ring. To this is applied the flanged supply pipe *P*, which can be readily secured and disconnected by means of bolts. *S* is the body and *ff f* are stiffening ribs.—S. G. R.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

*Cements which withstand Frost.* Bernhofer. *Thonindustrie Zeit.* 1890, 47.

THE author observed that mixtures of (a) 1 litre of Portland cement, 3 litres of river sand, and (b) 1 litre of lime cement and 3 litres of river sand, mixed with a solution of 1 kilo. of crystallised soda in 2 litres of water, when exposed to a temperature of  $-12.5^{\circ}$ , are not in the least altered in their adhesive properties.—J. B. C.

*Action of Boiling Water on Cement.* L. Erdmenger. *Thonindustrie Zeit.* 1890, 62.

GOOD Portland cement should after hardening withstand the action of boiling water; but water under a steam pressure equivalent to 10–40 atmospheres reduces its hardness. Mixtures of one part of cement and three parts of sand

when hardened can be subjected to the action of boiling water without undergoing change, but when considerable quantities of magnesia are present in the cement it does not withstand the continued action of boiling water, but gradually becomes soft.—J. B. C.

*Henry's Slag Cement.* H. Le Chatelier. *Bull. Soc. d'Encouragement l'Ind. Nat.* 1890, 26–28.

THIS is a report made to the *Société d'Encouragement* on behalf of the *Comité des Arts Chimiques*, and refers to an improved method of manufacturing hydraulic slag cements introduced into France by Henry. The manufacture of a cement of good quality from slags presents this difficulty that whilst only vitreous slags have any puzzolanic properties whatever, the most active varieties are those which contain the largest percentage of base. All attempts, however, hitherto made to increase the percentage of lime and alumina resulted in the formation of a crystalline slag which in virtue of its structure is totally inert.

Henry has overcome this difficulty by running the highly basic slag into water; the rapid cooling preserves the active amorphous structure; the slag is then finely crushed and mixed with a certain proportion of slaked lime. The product is not much inferior to Portland cement, and may be further improved by the addition of a few per cent. of precipitated silica.—S. B. A. A.

## PATENTS.

*A Process for Producing Mono-Carbonate of Calcium.* F. Mühlenbrueh, Magdeburg, Germany. Eng. Pat. 4195, March 9, 1889. 6d.

POWDERED calcium carbonate is mixed with finely ground quicklime in the proportion of 4.5 parts of the former to six parts of the latter and the mixture is stirred up with water; it is then exposed to the influence of carbonic acid gas or to the atmosphere (which always contains carbonic acid) whereby a solid and entirely homogeneous mono-carbonate of calcium is produced. Other materials such as marble chips, sand, colouring matters, &c., may be incorporated in the mixture. The substance produced may be used as artificial limestone, marble, for wall-compo, wall-covering or mortar, or it may be formed into artificial stone blocks.—H. S. P.

*An Improved Mode of Treating Hemp, Flax, Jute, and other Fibres, and Employing such Materials for the Manufacture of Tiles, Slabs, Cisterns, Boats, and other Articles or Structures.* B. A. Weatherdon, London. From T. A. Compere, Naples, Italy. Eng. Pat. 7058, April 27, 1889. 6d.

THE fibres are cut (by any suitable cutting machine) and boiled four to six hours in a strong solution of lime by steam at a pressure of from 20 to 40 lb. to the square inch. After being thoroughly washed, the material is passed to a pulping machine, where it is treated with alum about 7 to 14 lb. to the cwt., and in some cases with animal size, and in others with vegetable or other oils. From the pulping machine it is run into receiving or storing tanks, fitted with machinery for keeping the pulp in agitation, and whilst therein it can be dyed, if required. Now the pulp fibre is pumped into vats, and from thence into wire moulds, to be formed into slabs, blocks, &c., the liquor being extracted by suitable pressure. The slabs, blocks, &c. are brought into the drying-room, and when dry passed through solid steel rollers 4 to 12 times, steeped in a warm solution of alum, and again subjected to hydraulic pressure. Then they are brought into a bath of vegetable or other oil for four to six hours, and again pressed and steam-rolled as many times as necessary. To finish the process they are dried in hot air, and in some cases passed through the rollers once more.—H. S.

*An Improved Method of making Cement from Chalk, Marls, and Argillaceous Limestones, and of Utilising Poor Clays for the Purpose of making Cement, and Apparatus therefor.* G. P. Gildea, Ipswich. Eng. Pat. 8145, May 16, 1889. 6d.

THE patentee, having observed that the clay commonly used in the manufacture of Portland cement contains more organic matter than is generally present in cement stones and marls, adds to such materials, and thoroughly incorporates with them by the agency of hot water, 2—2½ per cent. of some oil or fat, allowing the mixture to rest for about a week, in order that the fatty matter may exert a "solvent influence" upon the clay. At the end of this time the slurry is dried and burnt as usual, producing, it is claimed, a better cement than would otherwise be obtained. The use of the fat is not claimed as a combustible, the quantity being inconsiderable, and the principle being already known.

—B. B.

*Improvements in Machines for the Manufacture of Bricks and Briquettes.* N. Procter, Leeds; A. Middleton, Ben Rhydding; C. E. Fraser and H. M. Carter, Leeds. Eng. Pat. 11,403, July 16, 1889. 6d.

HEAT being necessary and employed for moulding briquettes for fuel, the materials are apt to become unduly hot during their passage down the hopper by which they are fed into the moulding machine. The patentees insulate the hopper from the machine by means of an intermediate non-conducting piece of wood or similar material, and also provide a plate cutting off the connexion between the two, which plate is withdrawn periodically to allow the entrance of the materials into the machine by a rack and pinion apparatus actuated by a cam.—B. B.

## X.—METALLURGY.

*Zinc and Lead in Burnt Gases from Blast Furnaces.* A. Vita. Zeits. f. angew. Chem. 1890, 69—71.

WHERE ores are used containing zinc and lead, the oxides of these metals are contained in the burnt gases. The present paper concerns itself with the quantities of the oxides contained in the burnt gases, when the gases have before combustion undergone a certain amount of "purification." Such "purification" was effected by passing through a Maccos's ascension pipe and five tanks, each divided into two parts. Each tank was 14 metres high and had a base of 2 × 3 metres. A distributing channel of 1.5 × 2 metres section conducted the gases thence to the Cowper apparatus. The results obtained show that even after the passage of the gases through this system a notable quantity of zinc and lead still escapes.

Samples were aspirated from the gases after passing the Cowper apparatus, and were passed through five wash-bottles containing respectively water, hydrochloric acid, nitric acid, bromine in hydrochloric acid, and alcohol, the last-named being intended to absorb any bromine vapour which might pass over.

On six different days 1,202 litres of burnt gases were aspirated and examined. Samples of the unburnt gases were also examined. From the quantity and composition of the materials fed into the furnace in each day of 24 hours, and from the composition of the gases before combustion and after, it was calculated that for every ton of pig iron produced the burnt gases carried off 3.89 kilos. of zinc—that is, so much zinc is totally lost. The zinc recovered for every ton of iron produced was 15 kilos., so that the loss is 27 per cent. of the recovered zinc.

The loss of lead is naturally very much smaller than that of zinc. For every ton of iron produced the flue-dust contained 3.9 kilos. of lead, and the amount lost was 360 grms. Besides the losses here considered, there are other sources of loss. For instance, the furnace-slag contains zinc; loss is sustained at the furnace mouth; the masonry of the furnace takes up zinc.—T. L. B.

*The Nature of Amalgams.* W. L. Dudley. Address to the American Association for the Advancement of Science. August 1889.

THE author at the outset remarks upon the small amount of work which has been done on this subject. After a short account of early knowledge of amalgams he proceeds to more recent investigations. It was formerly supposed that the production of amalgams was attended with an evolution of heat, but Demachy in 1774 proved that a reduction of temperature occurred in the amalgamation of tin, lead and bismuth. At a later date J. Regnault found that amalgamation caused metals to change their place in the electro-chemical series. When heat is absorbed the amalgam is more electro-positive than the metal, when heat is evolved it becomes less electro-positive. Berthelot in 1880 examined the constitution of potassium and sodium amalgams by measuring the heat evolved in their formation. He obtained evidence of the existence of the alloys  $\text{Hg}_{12}\text{K}$  and  $\text{Hg}_5\text{Na}$ . He also found that the relative affinities of the alkali metals in these alloys were reversed, thus accounting for the displacement of potassium by sodium when sodium amalgam is added to a solution of potassium hydrate. Indications of the existence of the amalgams  $\text{Hg}_4\text{K}$ ,  $\text{Hg}_4\text{Na}$ , and  $\text{Hg}_7\text{Na}$  were also obtained. The electric conductivity of amalgams has been investigated by Mattiessen and Vogt, Sabine, Weber and others. Weber's researches were confined to the amalgams of tin, lead, bismuth and cadmium. It was found that a small quantity of impurity in mercury considerably reduces the resistance of the mercury. The conductivity of the amalgam is not the mean of the conductivities of its constituents. Bismuth and lead amalgams conduct better than either of their constituents. In 1850 Joule prepared amalgams of iron and copper by electrolysis, removing excess of mercury by submitting the alloys to a pressure of 60 tons. The iron alloy consisted of 100 parts of mercury to 103.2 parts of iron. The iron was found to retain its magnetic properties. The copper amalgam consisted of mercury, 100 parts; copper, 27.76 parts, and had a sp. gr. 13.17. A silver alloy,  $\text{AgHg}$ , and amalgams of platinum, zinc, lead and tin were also prepared. Considerable contraction took place in the formation of the copper, silver and zinc amalgams, but little with those of platinum and lead. Joule tried to amalgamate hydrogen by developing it at a low temperature (0° F.) in mercury. The specific gravity of solid mercury calculated from the copper amalgam was found by Joule to be 14.985. The latest direct determination gives 14.931.

De Souza, in 1875, examined the effect of heat upon amalgams. Gold amalgam had a composition  $\text{Au}_3\text{Hg}$ , after heating to the temperature of boiling diphenylamine, but became  $\text{Au}_9\text{Hg}$  when heated to the temperature of boiling mercury. Silver amalgam became  $\text{Ag}_3\text{Hg}$  in the vapour of boiling mercury,  $\text{Ag}_2\text{Hg}$  in that of diphenylamine. Copper formed the compound  $\text{Cu}_2\text{Hg}$  in vapour of boiling sulphur, but  $\text{Cu}_3\text{Hg}$  in boiling mercury and diphenylamine. At a later date Merz and Weith repeated de Souza's experiments and found that amalgams were gradually decomposed at the above temperatures, the small amount of mercury left being retained by mechanical attraction. The author has repeated these experiments on gold amalgam, and finds that it has a composition  $\text{Au}_3\text{Hg}$  when heated to the temperature of boiling mercury. He considers that the gases, hydrogen and nitrogen, used by the last experimenters to remove mercury may have caused the decomposition of their amalgams, especially as they observed that in the case of potassium and sodium amalgams the mercury was more rapidly removed in hydrogen than in nitrogen.

With regard to the melting points of amalgams, Merz and Weith found that they bore no relation to the composition of the alloy; for example, a sodium amalgam containing 3 per cent. of sodium, melted at 152°—160°; with 4.7 per cent. of sodium the melting point was 305°—315°, the maximum, a further increase of sodium lowering the melting point. The chemical activity of some metals is increased by amalgamation, whilst that of others is decreased. Thus Wanklyn and Chapman found that magnesium amalgam oxidised rapidly when exposed to air and decomposed water violently. Aluminium amalgam behaves in the same manner, whilst potassium and sodium are less energetic when amal-

gated than in the free state. The author gives a very full account of ammonium amalgam. The most conclusive experiments in proof of its existence are those of Davy, Landolt, and Routledge, showing that the amalgam contains ammonia and hydrogen in the proportion of 2 vols. of the former to 1 of the latter. The last observer examined the change in volume of the amalgam under different pressures, and found that the compressibility was due to gases resulting from the decomposition of the amalgam. In addition to the above mentioned experiment of Joule on hydrogen amalgam the author quotes that of Doebereiner and Loew, in which a zinc amalgam containing 5 per cent. of zinc was agitated with a 10 per cent. solution of platine chloride, the mixture being kept cool. The zinc amalgam swelled up considerably, and continued to evolve hydrogen until it was entirely decomposed. The hydrogenium amalgam thus prepared was dried by filter paper. On exposure in thin layers to the air, the temperature rose considerably and aqueous vapour was evolved.

The specific heat of alloys was shown by Regnault to be the mean of the specific heats of their constituent, but the amalgams of cadmium, tin, lead, and bismuth are exceptions. Wiedemann considers that these alloys are mixtures of a solid and a fluid, the proportions of which vary with the temperature. Two amalgams,  $\text{SnHg}$  and  $\text{Sn}_2\text{Hg}$ , were examined at  $128^\circ$  and  $164^\circ$  respectively, and behaved like lead alloys in the neighbourhood of their so-called second melting points, but the temperature did not remain constant for any appreciable time.

The above researches prove that amalgams are unstable chemical compounds, the absorption of heat on the amalgamation of some metals being probably due to the solution of the amalgam in the excess of mercury. The greater portion of an amalgam in excess of mercury is, however, only in suspension, since the author has found that the upper portion of at 0.1 per cent. gold amalgam, which had remained in a glass cylinder for some months, contained only 0.0683 per cent. of gold. After filtration through boxwood it contained only 0.0601 per cent. The effect of excess of mercury may be to reduce the amalgam to crystal units instead of breaking up the molecule as in ordinary solution. These crystal units would pass through the pores of chamois leather, but not through those of boxwood. The following index to literature on amalgams is given:—

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—H. K. T.

*The Use of Fluorspar in Metallurgy.* Foehr. Chem. Zeit. **14**, 253—254.

THE comparatively high price of fluorspar has excluded its use in iron smelting. It has, however, proved itself to be an energetic flux in the blast furnace, being introduced as powder through the tuyere holes. It plays an important part in the production of ferro-silicon. This compound can be produced in an ordinary blast furnace from any siliceous iron ore. It only requires a very basic slag and the addition of fluorspar. The latter greatly promotes the reduction of the silicon, probably forming silicon fluoride, which is reduced by the furnace gases or directly by the coke. The author considers it not unlikely that, owing to the enormous increase in the price of coke, fluorspar may be introduced as a fuel-saving flux, as the smallest addition of it effects the formation of dark grey iron.

The peculiar property of fluorspar in readily bringing about the reduction of the most different substances makes it a valuable flux in the production of spiegeleisen. Manganese fluoride is probably first formed and undergoes reduction at the expense of the carbon. It appears indispensable to form a highly basic fluoride slag in the production of a rich ferro-manganese. The property of fluorspar to combine with phosphorus in the slag plays an important part in dephosphorising pig iron in Krupp's basic cupola process.

A small quantity of fluorspar is now added in the Thomas and Bessemer processes, in order to reduce and concentrate the amount of slag and produce a better yield of metal. With an acid lining, fluorspar must be used with caution, as it is apt to corrode the lining. In puddling, the Martin's and other steel-making processes, a small quantity of fluorspar is said to be used as flux.

In the pig-iron manufacture the case is different and the much cheaper limestone is to be preferred. In melting down the pigs for casting, the addition of fluorspar effects the introduction of silicon into the iron and assists in eliminating phosphorus and sulphur. Too large a quantity of fluorspar is more deleterious than beneficial, probably in consequence of the manganese being retained in the iron. For 100 kilos. of pig iron only  $\frac{1}{3}$ — $\frac{1}{2}$  kilo. of fluorspar should be used. The addition of fluorspar is particularly noticeable in the newer cupolas, especially in that of Herbert's. For pure qualities of pig iron, such as Swedish charcoal pig, fluorspar is essential. Fluorspar assists greatly in fluxing heavy spar.—J. B. C.

*Experiments with Ferro-silicon.* Jüngst. Zeits. f. Berg.-Hütten-u-Salinenw, 1890, 1.

THESE experiments have shown that the presence of silicon increases the tenacity of cast iron. Whereas the best classes of grey cast iron resist a bending strain of 25 kilos., and have a tenacity of 14 kilos. per sq. cm., the addition of ferro-silicon imparts a tenacity of 37 and 23 kilos. respectively. The use of ferro-silicon may be introduced with success if the approximate chemical composition of the cast iron be known. It is important that ferro-silicon should not be added indiscriminately to grey cast iron containing silicon; but that the amount of alloy should be predetermined mainly by the quantity of combined carbon and graphite present. In addition to the composition of the cast iron, the structure has also an influence on the tenacity. The greatest tenacity may be attained both by re-melting grey cast iron as well as melting it down with ferro-silicon. The success of re-melting grey cast iron in this way depends on a variety of conditions, whereas the use of white iron with ferro-silicon is a much more certain operation, although the present price of ferro-silicon has precluded its extended introduction in Germany. With regard to the best quality of ferro-silicon, the experiments at the Gleiwitz works, with a medium quality containing 10.38 per cent. of silicium, gave the best results. A 5.32 per cent. ferro-silicon increased the tenacity, but was less satisfactory in other respects. A rich ferro-silicon, on account of the small quantity added, is liable to produce an irregular casting, although the tenacity is largely increased. The presence of manganese or phosphorus up to 1 per cent. and sulphur to the extent of .16 per cent. has no injurious effect. It must, however, be observed that in selecting the qualities of iron for castings the above quantities are not exceeded.

—J. B. C.

*On the Role of Certain Foreign Bodies in Iron and Steel.* F. Osmond. Compt. Rend. 110, 242—245.

It is known that iron exists under two states, which the author calls  $\alpha$  and  $\beta$ . When electrolytic iron containing only 0.08 per cent. of carbon is cooled down, an evolution of heat takes place at two points. The first at 855°, which he calls  $a_3^*$  is very marked; the second  $a_2$ , less marked, attains its maximum about 730°.

$a_3$  is due to the transformation of the allotropic form of iron  $\beta$  into the form  $\alpha$ ;  $a_2$  might be looked on as due to another transformation, but seems to be merely a retardation of the first taking place in those parts of the metal which contain most carbon. The carbon itself, passing from the state of *carbone de trempe* (hardening carbon) to *carbone de recuit* (annealing carbon) in iron containing this element, determines a third evolution of heat at a point  $a_1$ , which was discovered by Barrett under the name of *recalcescence*.

The position of these critical points is not fixed, but varies with the composition of the metal; as the percentage of carbon increases  $a_3$  is lowered, and finally coincides with  $a_2$ ; and then  $a_3$  and  $a_2$  united become lower still, and finally coincide with  $a_1$ , which has become slightly raised.

The action of certain bodies on these phenomena has been already studied by the author (Compt. Rend. 103, 743 and 1135; 104, 985 and 1800), and details are now given with regard to the influence of Bo, Ni, Cu, Si, As, and W.

1. *Boron* (specimen prepared by Professor Roberts Austen) acts like carbon; the evolution of heat normally taking place at  $a_3$ , occurred partly at 815°—805°, partly at 735°—725°, i.e.,  $a_2$ .

2. *Nickel*. The specimen contained C = .34 per cent. Ni 5.97 per cent., Mn "traces." With a steel containing the same percentage of C, but no Ni,  $a_3$  and  $a_2$  would coincide. In this case  $a_3$ ,  $a_2$ , and  $a_1$  all coincide at a single point, 660°—640°, a temperature below the normal point of recalcescence.

\* The author uses an extension of Tchernoff's notation of the lowest temperature at which the steel can be hardened, by the letter  $a$ .

Hopkinson has shown by another method that for a hard steel with 25 per cent. Ni, the critical point is below 0°. Manganese acts like nickel.

3. *Copper*. Three specimens prepared by Messrs. Ball and Wingham, given by Professor Roberts Austen:—

	I.	II.	III.
Cu .....	Per Cent. 0.847	Per Cent. 4.19	Per Cent. 4.44
C .....	0.102	0.183	0.10

As the percentage of Cu is increased,  $a_3$  and  $a_1$  are both lowered.

With II. and III.  $a_3$  coincides with  $a_2$  at a point 730°—720°, and  $a_1$  is lowered to 625°—620°. Hence copper acts like carbon, but much less energetically, the action of 4 per cent. Cu being equal to that given by 0.25 to 0.30 of C. Copper acts on the point of recalcescence in the same way as nickel and manganese.

4. *Silicon* (five specimens prepared and given by Mr. R. A. Hadfield, containing 0.20 to 4.40 per cent. Si). As the percentage of silicon increases the quantity of heat evolved at the point  $a_3$  diminishes, and is sensibly nil when 2 per cent. is reached. Contrary to what occurs with the previous elements, this heat is not evolved at  $a_2$  or any other point. Hence it follows that silicon prevents the allotropic transformation of the iron, which remains in the state  $\alpha$ . The evolution at  $a_3$  is unchanged in quantity, but  $a_2$  is lowered from 730°—720° to 710°—700°, and  $a_1$  is raised from 660°—650° to 710°—700°.

5. *Arsenic* (four specimens prepared by Messrs. Harbord and Tucker, given by Professor Roberts Austen). As the proportion of arsenic increases from "traces" to 0.55 per cent., the point  $a_3$  is raised, but the heat given out is less than the normal amount. Arsenic acts like silicon, the iron being kept in the state  $\alpha$ . It had no appreciable effect on the points  $a_2$  and  $a_1$  in the specimens examined.

6. *Tungsten* (four specimens, due to the same origin as the preceding). Tungsten (varying from "traces" to 1.50 per cent.) seems to cause no change in the position of  $a_3$  or the amount of heat evolved at this point. But  $a_1$  is considerably lowered if the metal has been sufficiently heated; it was lowered to 540°—530° in the specimen containing most tungsten, a phenomenon observed before in a steel of much harder character. The behaviour of tungsten is peculiar.

—P. J. H.

*On the Role of Foreign Bodies in Iron and Steel, and the Relation between their Atomic Volumes and the Allotropic Transformations of Iron.* F. Osmond. Compt. Rend. 110, 346—348. (See preceding abstract.)

THE author points out that his experiments confirm the suggestion of Roberts Austen (Phil. Trans. 179, 339—349, 1888) that the effect of the admixture of small quantities of metallic impurity with metals is related to the atomic volumes of the elements concerned.

In the following table, column I. contains the elements examined, whose atomic volume is less than that of iron (7.2), column II. those with greater atomic volume.

I.	Atomic Volume.	II.	Atomic Volume.
Carbon .....	3.6	Chromium .....	7.7
Boron .....	4.1	Tungsten .....	9.6
Nickel .....	6.7	Silicon .....	11.2
Manganese .....	6.9	Arsenic .....	13.2
Copper .....	7.1	Phosphorus .....	13.5
		Sulphur .....	15.7

The bodies in column I. (other things being equal) retard the transformation of the  $\beta$ -modification of iron into the  $\alpha$ -modification, and of hardening carbon into annealing carbon. Hence, under the same conditions of cooling, they tend to increase the proportion of the  $\beta$ -iron in the metal *and to harden it*. Hydrogen, in the form of Graham's "hydrogenium," may also be included under this head, electrolytic iron in which it is contained being very hard and brittle. Gaseous hydrogen seems to have but little effect on the critical temperatures. The bodies under column II., with larger atomic volume than that of iron, would tend either to increase or at any rate to keep the temperature of transformation of  $\beta$ -iron into  $\alpha$ -iron at the normal point, and also (with the exception of tungsten) raise the temperature at which hardening carbon is transformed into annealing carbon. Hence they would tend, like annealing, to render the metal softer and more malleable; but this effect is sometimes masked by the properties of the admixed metal itself. We may say that admixed metals, if their atomic volume be low, tend to keep iron in the molecular modification of low atomic volume; if their volume be high, tend to keep it in the modification of high atomic volume.

Carbon, though obeying the general law, exerts a peculiar influence on iron, owing to the transformation which it undergoes at a certain temperature, and the existence of which is undeniable; hence its importance in the metallurgy of iron. (See this Journal, 1889, 75.)—P. J. H.

*Hadfield's Manganese Steel.* H. Le Chatelier. Bull. Soc. d'Encouragement l'Ind. Nat. 1890, 22—26.

This is a report made to the *Société d'Encouragement* by Le Chatelier on behalf of the *Comité des Arts Chimiques*, completely bearing out all that Hadfield claims for his alloy. The results of a series of experiments made for the purpose of direct verification of the properties of the alloy are exhibited in the following table:—

#### COMPOSITION OF THE ALLOY.

Mn = 13.9 per cent. C = 1.36 per cent.

##### (1.) TENSILE STRESS.

Dimensions of test-piece.—Length, 80 mm. Diameter, 8 mm.

	Breaking Load in Kilos. per sq. mm.	Elongation Per Cent.
Tested as forged .....	81.3	1.23
Heated to a red heat and oil-toughened .....	80.9	18.9
Heated to a yellow-heat and water-toughened .....	91.2	35
Heated to a yellow heat and toughened in a refrigerating mixture .....	106.9	51.8

##### (2.) BENDING STRESS.

Dimensions of test-piece.—70 × 24 × 9 mm.

	Interior Angle at Fracture.
Tested as forged .....	172°
Heated to a red heat and toughened in oil .....	154°
Heated to a red heat and toughened in water .....	Folded completely.
Heated to a yellow-heat and toughened in water .....	Showed deep cracks on bending double.

—S. B. A. A.

#### *The Treatment of Waste "Pickle."*

THE treatment of galvanisers' waste pickle—a most serious matter for West Bromwich, Wolverhampton, and Great Bridge in particular—has been recently made the subject of experimental tests with a view of neutralising its deleterious effects. Thomas Turner, of Mason's College, has patented a process which promises to achieve the desired ends, and which has been for some little time past in operation at the works of Walker Brothers, Walsall and Netherton (this Journal, 1889, 780). The principle of the process is simple in the extreme. The waste liquor is merely boiled down to dryness, and the solid residue heated to low redness. Oxide of iron remains in the furnace, while free hydrochloric acid distils off, is condensed, and can be used over again *ad infinitum*. But though the idea is thus simple, the practical application of it was a matter which involved costly experiments, extending over many months. The difficulties to be overcome were very great, since the liquor contained so much free acid that to use any metal vessel was quite impossible; nor could it be contained, when at boiling heat, in any kind of brick or stone evaporating-pan. At the same time it yielded, on evaporation, such a large quantity of solid residue that the bottom of any vessel used became covered with a thick deposit after a few hours' working, and this deposit was so hard that it could be removed only by means of a sledge-hammer and a crow-bar. Even this was not all. The ferrous chloride, when heated in the presence of air, formed a considerable amount of ferric chloride, which, volatilising freely, contaminated the recovered acid. All these difficulties have at length been overcome, and the process has been in successful operation for six months, and has been proved on the large scale to entirely do away with the waste "pickle," and what is even more remarkable, to yield a clear profit after paying all working expenses.

The ferrous chloride and the waste liquor are heated in one and the same furnace by the reverberatory principle. Much depends upon the regular introduction and distribution of the liquor. The corrosive action of the free acid is avoided by leaving on the brick floor of the retort a coat of the thick deposit above mentioned; the formation of ferric chloride is checked by roasting the residue inaccessibly to air; and the method of working by heat from above prevents the deposit from caking beyond such a depth as is desired. The distilled acid is condensed in a tall stack of special construction; and the oxide of iron remaining is raked out of the furnace in the state in which it is commonly known as "blue billy," and is of use in puddling operations for "fettling." As a result of experience it is found that the furnaces will work three months without stoppage, that they use only 4 cwt. of fuel to completely treat a ton of waste liquor, that the acid recovered is perfectly suitable for using over and over again, and that the oxide of iron recovered has a value which goes a long way towards paying for the fuel used.

#### PATENTS.

*Improvements in the Treatment of Complex Gold and Silver bearing Ores and other Substances, and the Recovery of Metals therefrom.* H. Hutchinson. Eng. Pat. 14,400, October 6, 1888. 6d.

COMPLEX gold and silver "sulphurets, arsenurets," &c., or artificial products such as matte and regulus, are treated with an alkali or alkaline earth, or with the carbonates of these latter, either before or after calcination, whereby "sickening or flouring" of the mercury used in the amalgamation process is prevented, or loss of chlorine, bromine, &c. in the solvent processes. In one modification of the process the ore is calcined, either with or without addition of common salt, care being taken not to decompose the sulphates which are formed by oxidation of the sulphur. The calcined ore is then treated with water to remove soluble sulphates and chlorides, leaving a residue of silica, oxide of iron, sulphate or chloride of lead, and free gold. On treating this residue with an alkali or alkaline carbonate the lead salts are decomposed, and their "flouring" effect prevented. The

oxidation of the finely-divided iron derived from the stamps is one of the causes of the "flouring" of mercury.

—H. K. T.

*An Improved Method of Lining Barrels or other Vessels to resist Acids for use in extracting Metals by Chlorination or similar Process from their Ores.* T. Bell, Lincoln. Eng. Pat. 2350, February 9, 1889. 4d.

VESSELS of wood or iron are lined with lead, the lead is coated with gutta-percha, which is again coated with lead. The outer lead coating may be omitted.—H. K. T.

*Improvements in the Treatment of the Calcined Products or "Small Mine" of certain Iron Ores, Ironstone Mines and Shale, and the Manufacture of the same into Blocks for the better Extraction of Iron therefrom.* A. Hodgkinson, Peacock's Hey. Eng. Pat. 4537, March 15, 1889. 4d.

"SMALL mine," the waste product left on the mine-hearths in the calcining of various ironstones in Staffordshire, is compressed into blocks and fired, in order to render it fit for smelting. Sometimes the powdered material is riddled before compression.—H. K. T.

*Improvements in Machinery or Apparatus for the Extraction of Gold and Silver from their Ores by Amalgamation.* T. R. Jordan, London. Eng. Pat. 4719, March 18, 1889. 11d.

A NUMBER of circular dishes of amalgamated copper are mounted on a vertical spindle and made to rotate. Beneath each dish is a tray sloping towards the centre where it is perforated. The auriferous sand, when charged into the uppermost dish, is forced by centrifugal force up the edges of the dish with considerable friction, thus ensuring perfect contact with the amalgamated surface. It then falls on to the sloping tray and passes to the next dish, and so on to the bottom. There it falls into a well, and is kept in agitation by a stream of water. Mercury is placed in the dishes or in wells formed on the peripheries of the fixed partitions.—H. K. T.

*Improvements in Means or Apparatus for the Purification of Molten Tin or its Alloys.* D. Owen, Morriston. Eng. Pat. 5037, March 23, 1889. 8d.

THE molten tin or alloy is made to pass through a "reticulate or filtering masses formed of assemblages of plates, wire, shot, or woven wire work, fibre, or other forms of iron or other metal or suitable refractory material," for the purpose of removing "scruff" before passing to the tinning pot.—H. K. T.

*Improvements relating to the Drying and Calcination of Ores or other Substances, and to Apparatus therefor.* W. Butlin, Wellingborough. Eng. Pat. 5869, April 5, 1889. 8d.

AIR heated to a temperature of from 900°—1,000° F. is used for drying and calcining ores, limestone, and other substances, to prepare them for the smelting furnace. A furnace or kiln is employed, having a perforated chamber, of cylindrical or other form, built up of boiler plates or of cast metal cylinders firmly united, &c., into which air passes, heated by the waste heat from the smelting furnace. The heated air then passes from the perforated chamber into the furnace or kiln.—E. G. C.

*An Improved Method for and Apparatus for Separating Liquid containing Precious Metal from Ores treated by Wet Processes.* T. K. Rose and D. Dennes, Denver, Colorado, U.S.A. Eng. Pat. 6026, April 8, 1889. 8d.

THIS method, which is applicable to any of the wet chlorination processes, consists in enclosing the material to

be leached in closed filtering vats or tanks, and forcing air, steam, water, or other fluid into these vessels, so as to drive the solution under pressure (usually about 60 lb.) through a bed of filtering material.—E. G. C.

*Improvements relating to the Recovery of Soda from Slags containing the same.* "The Alkaline Reduction Syndicate, Limited," and A. B. Cunningham, Hebburn-on-Tyne. Eng. Pat. 7733, May 8, 1889. 4d.

THE slag remaining after the preparation of metallic sodium or after the reduction of metallic ores by the use of an alkali, is run, whilst in a molten condition, directly into water, solution at once taking place. If the slag be run into milk of lime then any carbonate of soda will be at once causticised and a solution of pure caustic soda produced.—S. G. R.

*A New or Improved Alloy.* A. M. Clark, London. From "La Société en participation Bourbouze et Compagnie," Paris, France. Eng. Pat. 13,718, August 30, 1889. 4d.

AN alloy of 100 parts of aluminium with 10 parts of tin, possessing a density 2.85 and an index of specific heat 0.180, together with the characteristic properties of pure aluminium, viz. "strength, lightness, inoxidability, ductility," &c.—E. G. C.

*An Improved Process or Means for extracting Precious Metals from their Ores.* D. Deunes. Eng. Pat. 19,621, December 6, 1889. 6d.

THE ore passes through a crushing mill, then through a muffle furnace, where it is roasted by gaseous fuel. It next passes into a vessel rotating on hollow trunnions, where it is treated with bleaching powder and sulphuric acid. Any excess of chlorine is removed by blowing air or steam through one of the trunnions, the gases passing out through the other one and thence through slaked lime or other absorbing materials. The liquid contents of the vessel then pass through filtering beds and finally through layers of sulphides.—H. K. T.

ERRATUM.—Owing to a misprint in the original journal the conclusion of the last sentence of first abstract on second column, page 296, has been made to read " $\text{Fe}_2\text{Fe}_3\text{C}$ ,  $\text{Fe}_3\text{C}$ ," instead of " $\text{Fe}$ ,  $\text{Fe}_2\text{C}$ ,  $\text{Fe}_3\text{C}$ ," as intended by the author.

## XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

*Experimental Researches in the Reduction of the difficultly-reducible Metals.* A. J. Rogers. Reprinted from Proceedings of the Wisconsin Natural History Society, 1889. J. Franklin Inst. 1889, 98, 486—487.

THIS paper gives the results of experiments on the electrolysis of sodium and aluminium salts at high temperatures. An average of 61 per cent. of the theoretical yield of sodium can be obtained by electrolysis from the fused chloride, the yield being 5—6 lb. of sodium per electrical horse-power in 24 hours. Alloys of sodium with tin and lead were prepared by making these metals the negative electrodes in the electrolysis, either of sodium chloride alone, or of the latter mixed with cryolite. They do not attack silicious crucibles so rapidly as pure sodium. Aluminium can be prepared by heating the alloys with cryolite, or with the double chloride of aluminium and sodium. The yield was 1 lb. of aluminium per electrical horse-power in 24 hours.—H. K. T.

*On the Ratio between the Electric and Thermal Conductivities of Metals.* A. Berget. *Compt. Rend.* **110**, 76—77.

THE author has determined to measure the electric conductivity  $k$ , and the thermal conductivity  $c$ , of copper, zinc, iron, tin, lead, antimony, mercury and brass, at temperatures between  $0^\circ$  and  $30^\circ$ . The ratio  $\frac{k}{c}$  varies from  $1.6 \times 10^3$  to  $1.8 \times 10^3$ . The temperature-coefficient of the two conductivities is also different. Hence they can only be regarded as approximately proportional to one another.

—P. J. H.

*On the Electrolysis of Alumina and Aluminium Fluoride in the Fused State.* A. Minet. *Compt. Rend.* **110**, 342—343.

THE author has made a large number of experiments for the purpose of obtaining aluminium by electrolysis on a large scale. He gives the following details of 22 experiments: (1) Nature of cathode (iron or carbon); (2) Duration of experiment (varying from 7 to 24 hours); (3) Current intensity (from 90 to 1,200 ampères); (4) Difference of potential at electrodes (from 4 to 6.35 volts); (5) Weight of metal obtained (0.363 to 5,440 kilos.); (6) Percentage of theoretical yield (55 to 82 per cent.) No further account of the process is given. The experiments were carried out at the works of MM. Bernard, at Creil.—P. J. H.

#### PATENTS.

*Improvements in the Manufacture of Incandescence Electric Lamps.* J. W. Edmundson, Gateshead-on-Tyne. Eng. Pat. 3353, February 25, 1889. 4d.

TO prevent lamps from blackening and to obtain uniformity, the filaments are coated with chromium, in some such way as the following:—They are painted with chromium chloride dissolved in spirits of wine, dried, and then raised to very bright incandescence in a vessel exhausted of air as completely as possible. The chloride is reduced and the metal deposited, the filament getting a smooth grey surface and a lowered resistance. The operation may be repeated if necessary.—E. T.

*An Improved Process for the Manufacture of Aluminium and Apparatus therefor.* J. L. E. Daniel, Westminster. Eng. Pat. 4169, March 9, 1889. 11d.

IN this process aluminium is produced by electrolysis the double chloride of aluminium and sodium, and means are provided for maintaining a constant and continuous replenishment or regeneration of the bath with chloride of aluminium, as fast as it loses the same while undergoing electrolysis. For the production of the chloride of aluminium, an upright retort is filled with alumina and carbon, and is heated while dry chlorine is caused to pass through it.—E. G. C.

*Improvements in the Electrolytic Process of Obtaining Copper, Zinc, and Tin.* H. A. Seegall, Berlin, Germany. Eng. Pat. 4871, March 20, 1889. 6d.

THE material to be treated, containing copper with zinc or tin, is first lixiviated with a moderately concentrated solution of ferric chloride, slightly acidulated. Proto-chloride of iron, and the chlorides of zinc or tin, are formed and pass into solution, while cuprous chloride remains suspended. The whole is filtered, and the filtrate treated electrolytically for zinc or tin. The deposit of cuprous chloride is mixed with copper borings, &c., and again lixiviated with a concentrated solution of the ferric chloride, also acidulated with at the same time the addition of calcium or sodium chloride, to hold the cuprous chloride in solution. This solution is then treated electrolytically for its copper. The various liquids to be electrolysed pass from cathode cell to

cathode cell of a series of baths, till the metal is removed, and then through the anode cells of the same baths, to be oxidised again, being then used for lixiviating fresh masses of the substance.—E. T.

*Improvements in the Method of and Means for Electrolysis of Substances in a State of Fusion.* M. Kiliani, Xenhausen, Switzerland. Eng. Pat. 6745, April 20, 1889. 8d.

IN order to keep the temperature of the fused mass as low as possible, and also to keep the crust which is formed on the surface of the mass constantly stirred back into the active path, the positive electrode is so arranged that it can be rotated either concentrically or eccentrically about its axis. The positive electrode is also so arranged that it can be raised from or lowered into the fused mass as it burns away.—B. T.

*Improvements in Electric or Galvanic Elements or Cells.* P. Schoop, Zurich, Switzerland. Eng. Pat. 7719, May 8, 1889. 6d.

IN order to convert the electrolytes of batteries into a solid or semi-solid state "water-glass" (i.e., silicate of soda or potash) is added in suitable proportions, or, instead of water-glass, a solution of sulphate of iron, either alone or mixed with hydrate of soda or potash, or with water-glass, may be added.—B. T.

*An Improved Process and Apparatus for the Continuous Extraction of Gold and Silver from Substances containing the same by the Agency of Chlorine.* J. A. H. T. Ranft, Sydney, N.S.W. Eng. Pat. 19,944, December 11, 1889. 8d.

CHLORINE is liberated by electrolysis from a solution of sodium chloride in contact with the gold or silver or the substances containing the same. The chlorides produced are immediately split up electrolytically, the chlorine being set free at the anode and regenerated for further use, and the metallic elements deposited upon the cathode. There are ten claims in connexion with this process, and the apparatus therefor.—E. G. C.

*Improved Battery Plates for Storage Batteries.* A. E. Woolf, New York, U.S.A. Eng. Pat. 20,911, December 31, 1889. 8d.

A LONG plate of lead is pasted with lead oxide, and then rolled tightly upon itself in a close scroll. This is then heavily pressed, so as to convert the almost circular cross section into an elongated one. Slices are now cut off the end, each slice forming a complete plate. This is encircled by a strap of hard rubber, which being wider than the plate is thick, not only serves to hold it together, but acts as a separator as well.—E. T.

*Improvements in Secondary or Storage Batteries, and in the Manufacture of Plates therefor.* H. H. Lake, London. From P. H. Alexander, New York, U.S.A. Eng. Pat. 302, January 7, 1890. 8d.

THE inside of the battery case is lined with rubber, or some such material, in which grooves have been moulded or rolled. Into these grooves the individual plates slide, and the compartments thus formed are rendered quite water-tight by compressing the rubber from one end. One side of each plate is thus positive and the other negative, there being as many cells—all of course in series—as there are compartments. Moreover, the inventor packs his plates with dry oxide, then puts them in a pile, separated from one another by felt soaked with dilute acid, and passes a current through the whole till formed.—E. T.

*Improvements relating to Secondary Batteries.* H. H. Lake, London. From P. H. Alexander, New York, U.S.A. Eng. Pat. 303, January 7, 1890. 8d.

THE battery-box is made of some suitable material, such as hard rubber, and fastened to the outside are strips of metal from the terminals of the cell, so arranged that should the temperature of the cell rise above a certain point, the consequent expansion of the material of the box brings these metal strips into contact and short-circuits the cell through a safety cut-out, which cut-out is fused by the passage of the extra current and cuts the cell out of circuit. A second device for cutting out a cell which is giving off gas freely is a U-tube filled with the electrolyte and inverted in the liquid. As the gas is evolved in the cell some of it enters the U-tube and displaces the liquid in it so that it rises bringing two strips of metal into contact and cutting out the cell by means of the fuse arrangement described above.

—B. T.

*Improvements relating to Secondary or Storage Batteries.* H. H. Lake, London. From T. Ewing, New York, U.S.A. Eng. Pat. 1094, January 21, 1890. 6d.

THE inventor packs the plates with *Massicot* (litharge) since the active material obtained from this has, he states, the same rate of expansion and contraction as the lead support itself. There is therefore no buckling or deformation of the plates. The litharge may be applied as a paste, or packed dry into the plates by pressure. In the latter case immersion of the plates must not go on at a more rapid rate than six inches a minute, else disintegration will take place. The cells are stated to have a larger storage capacity by this method of forming.—E. T.

*An Improvement in Galvanic Batteries.* C. A. Hitchcock, London. From W. Burnley, North East, Pennsylvania, U.S.A. Eng. Pat. 1110, January 21, 1890. 6d.

THE containing-vessel of this battery is a cylinder of zinc closed at one end and serving as the positive electrode. A core of suitable size and material is placed in position in the zinc cylinder, leaving an annular space around it, and into this space is poured the following mixture in a semi-fluid state:—Sal-ammoniac, 1 part; chloride of zinc, 1 part; plaster of Paris, 3 parts; water, 2 parts; and a vegetable material, such as flour, 1 part. When this mixture has set, the core is withdrawn, and a carbon cylinder inserted, also leaving an annular space around it. This space is filled with sal-ammoniac, 1 part; chloride of zinc,  $\frac{1}{10}$ th part; peroxide of manganese, 3 parts; pulverised carbon,  $3\frac{1}{2}$  parts; water, 2 parts. The top of the cylinder is then sealed in with a compound such as bitumen.—B. T.

*Improvements relating to Storage Batteries or Accumulators.* H. H. Lake, London. From G. A. Johnson, Boston, U.S.A. Eng. Pat. 1495, January 28, 1890. 6d.

EACH electrode is built up of two corrugated plates, so put together that there is a series of horizontal channels formed of somewhat elliptical-shaped section. Into these the active material is packed, being placed in communication with the electrolyte outside by very numerous small perforations in the plates themselves. The electrodes are insulated and supported by vertical rods passing downwards between them.—E. T.

## XII.—FATS, OILS, AND SOAP MANUFACTURE.

*The Constituents of Iceland Moss.* A. Hilger and O. Buchner. Ber. 23, 461—464.

KNOX and SCHNEIDERMAN (Ann. Chem. Pharm. 55, 164) have examined two of the constituents of Iceland moss (*Cetrarea islandica*) (see under XX., page 544.) lichestearic acid and cetraric acid. These have been further studied by the authors. Lichestearic acid is best extracted from the powdered moss by means of petroleum spirit, and forms, when purified by recrystallisation, a voluminous crystalline mass melting at 120°, and soluble in the ordinary reagents with the exception of water. The alkali salts of the acid are soluble, those of the other metals insoluble in water, but none of them could be obtained crystalline. Analyses of the silver, barium and lead salts, of an acid chloride and also of the free acid, are given, from which the formula  $C_{43}H_{76}O_{13}$  appears probable, the acid being dibasic,  $C_{41}H_{74}O_9(CO.OH)_2$ .

Acetyl chloride is without action on the free acid; on oxidation with potassium bichromate and sulphuric acid, carbon dioxide is evolved and capric acid formed.

Cetraric acid forms a white amorphous powder, with a bitter taste, almost insoluble in water. It dissolves in alcohol, but is difficultly soluble both in ether and in petroleum ether. At 200° it decomposes before melting. Analyses of the acid and of its barium and silver salts point to the molecular formula  $C_{30}H_{50}O_{12}$ , and to the dibasicity of the acid.—C. A. K.

## PATENTS.

*Method of Extracting Pure Neutral Grease from Wool and the like.* C. Lahusen and C. Feuerlein, Delmenhorst, Germany. Eng. Pat. 11,383, July 16, 1889. 6d.

PURE neutral wool grease has hitherto been obtained from the sludge thrown down by treating wool washings with sulphuric acid, by more or less troublesome methods. The present plan consists in mixing the sludge (wet or dry) with a "five-fold quantity" of a solvent such as "benzine, bisulphide of carbon, ether, or chloroform, or a mixture of these liquids, at 50°—70° C. A solution of some "water absorbent material" e.g., the chloride of sodium potassium or magnesium or sodium sulphate, is then added in quantity equal to about half the liquid previously used, and the whole well mixed. The mass separates into two layers on standing, the one consisting of a solution of wool grease in carbon, disulphide, ether, &c., the other of the soapy impurities and metallic salts. The former is run off, distilled, and the wool grease obtained, the latter worked up into soap, or otherwise, by known methods.—B. B.

*Improvements in Lubricants.* J. L. Wade, London. Eng. Pat. 540, January 11, 1890. 4d.

A MIXTURE is made of crude glycerin, mineral oil and castor oil in proportions varying with the character of its proposed use, by heating it with close steam in a boiler and causing a rush of air through it when it has reached a suitable temperature by exhausting the air-space above the surface of the liquid, and permitting the entrance of air by an inlet pipe at the bottom, which both mixes and dries the oils.—B. B.



### XIII.—PAINTS, PIGMENTS, VARNISHES, AND RESINS.

#### PATENTS.

*Improvements in the Production of the Compounds for the Manufacture of the Soles and Heels of Boots and Shoes, the same being applicable to other useful Purposes.* A. A. Blandy, London. Eng. Pat. 3591, February 28, 1889. 6d.

THE artificial leather is composed of asphalte, mineral pitch, colophony or black resin, gypsum, gutta-percha, and sulphide of antimony. A harder compound for the heels of boots and shoes is prepared from the same ingredients with the addition of some sulphur, but the proportion of gutta-percha is smaller. The most suitable proportions are given in the specification.—E. E. B.

*The Manufacture or Production of an Improved Material or Compound adapted for use as a Substitute for Ivory, Horn, Whalebone, India-rubber, Gutta-percha, and other Materials, also as a Coating or Varnish for Waterproofing and other Purposes.* F. Greening, Uxbridge. Eng. Pat. 5344, March 28, 1889. 6d.

COTTON combings, paper, esparto grass, or rags, are treated with a mixture of nitric and sulphuric acids. The excess of acid is drained off and the material is placed in a warm atmosphere for one hour. It is then washed with water and transferred to a bath of sodium chloride and ammonia alum. After again washing and drying it is dissolved in a mixture prepared as follows:—Lead acetate and lime are distilled and the product is mixed with fusel oil. The mixture is redistilled after the addition of phosphoric chloride or carbonate of potash. The distillate mixed with absolute alcohol constitutes the solvent.

The plastic mass obtained by the action of the solvent on the nitro-compounds, may be moulded into any desired form. A thin solution can be used as a varnish.—E. J. B.

*Improvements in obtaining Yellow Pigments.* J. Macfarlane, Barnhead. Eng. Pat. 7156, April 30, 1889. 4d.

CARBONATE of lime is mixed with a solution of ferrous sulphate and a yellow precipitate is obtained, consisting of hydrated oxide of iron together with sulphate of lime, and the excess, if any, of carbonate of lime.—E. E. B.

*Improvements in Fabrics.* A. J. Boulton, London. From C. Coste, Paris, France. Eng. Pat. 7967, May 13, 1889. 4d.

“AN improved article or fabric mainly consisting of pure floss silk, which by being coated with india-rubber and like substances of any colour or shade, acquires an appearance similar to that of leather, and is adapted to replace both leather and waterproof india-rubber stuffs imitating leather.”—H. S.

*Improvements in the Manufacture of Artificial Ivory or Ivory Substitutes.* A. and S. de Pont, Paris, France, and H. J. and J. H. Storey, Lancaster. Eng. Pat. 9267, June 4, 1889. 6d.

See under XIX., page 540.

*An Improvement in the Manufacture of Oil Varnish.* H. Noerdlinger, Stuttgart, Germany. Eng. Pat. 9563, June 8, 1889. 4d.

A NON-VOLATILE oil is heated with a saturated salt of resin acid, oleic acid, or another fatty acid. This mixture is allowed to cool, and the supernatant clear liquid mixed with from ten to a hundred volumes of a drying oil, such as linseed oil.—E. E. B.

*Improvements relating to the Covering of various Articles with Xylonite or Celluloid.* E. A. Bluemel, Bow. Eng. Pat. 17,496, November 4, 1889. 4d.

A TUBE of xylonite of smaller diameter than the article to be covered is placed in boiling water or steam and stretched over a heated mandril. It is then plunged in cold water and the mandril removed. The article is then inserted and plunged with its covering of xylonite into boiling water for about 30 seconds, until the tube has contracted and fits closely to the article. They are again plunged into cold water, and, if necessary, the ends are plugged with similar pieces of xylonite.—E. J. B.

### XIV.—TANNING, LEATHER, GLUE, AND SIZE.

#### PATENTS.

*A New or Improved Adhesive Cement and Process of Manufacturing the same.* W. P. Thompson, Liverpool. From K. Korenar and F. Zwickler, Karolinenthal, Austria. Eng. Pat. 2197, February 11, 1889. 4d.

THIS cement, suitable for joining or repairing leather, hemp, or india-rubber hose pipes, is prepared by dissolving about 50 parts by weight of gutta-percha in a liquid mixture of 120 parts by weight of “benzine,” 80 parts “sulphuret of carbon,” and 20 parts of turpentine, removing the surface scum from this solution and mixing it with a second batch of a similar solution whilst in a boiling state. The liquid mixture obtained is drawn off into suitable receptacles, and kept hermetically sealed.—B. H.

*Improved Method of Manufacturing Artificial Leather or Leather Boards made from Hard Sole Leather Cuttings or other Leather Scraps and various Fibrous Substances.* H. R. Mims, Maidenhead. Eng. Pat. 6103, April 9, 1889. 4d.

LEATHER cuttings or scraps are boiled in an alkaline or acid solution till dissolved. In practice a solution of sulphuric acid containing from 5 to 20 per cent. answers well. The fibrous substance used, such as leather, hemp, manilla, or jute, is mixed with the solution of leather cuttings in an ordinary heating engine at a “temperature of 110°–180°.” When all is reduced to a pulp of the required consistency, it is passed through a suitable machine and made into sheets or boards. The sheets or boards are then engraved with any design by suitable machinery.—B. H.

*Improvements in the Manufacture of Cement for joining Wood, Stone, and other Materials.* S. Hindley and G. R. Mackenzie, London. Eng. Pat. 7943, May 11, 1889. 4d.

THIS cement is prepared by soaking glue in water for about 12 hours, and then adding to the glue, after melting it, a mixture of white lead and cement, together with a solution of resin in methylated spirit or mineral oil. The following proportions give good results:—Dissolved glue, 84 lb.; Parisian cement, 9 lb.; white lead, 9 lb.; resin, 6 lb.; methylated spirit, 4 lb.—B. H.

*Improvements in the Process and Means for Dyeing Hides.* J. Kristen, Brunn, Austria. Eng. Pat. 8191, May 16, 1889. 8d.

See under VI., page 500.

*An Improved Glue or Cement.* G. Rothel, London. Eng. Pat. 556, January 11, 1890. 4d.

THE glue is composed of the following ingredients in about the following proportions:—Scotch glue, 6 lb.; rosin, 2 lb.; gutta-percha, 2 oz.; caoutchouc, 1 oz.; wood ashes, 2 oz.; red lead, 4 oz.; bisulphide of carbon, 1 oz.; zinc ash, 2 oz. The glue is first boiled, to which a solution of the rosin in carbon bisulphide is added. The gutta-percha and caoutchouc are then dissolved in carbon bisulphide and added to the glue and rosin, and the mixture is boiled for two hours.

The red lead, wood ashes, and zinc ash are next added, and the whole mixture boiled for three hours with continual stirring. When required for use the glue is liquefied by heat.—B. II.

## XV.—AGRICULTURE, MANURES, Etc.

*Value of Thomas-slag Phosphoric Acid compared with Soluble Phosphate and Bone-Meal impoverished of Lime.* E. Güntz. Ber. u. d. Thätigkeit d. agrikultchem. Versuchsstat. Pommritz, 1888, 11—17; Bied. Centr. 29, 86—87.

ANIMAL charcoal superphosphate (spodium-superphosphat) containing 13·81 per cent. of soluble phosphate; Thomas-slag (93·6 per cent. of fine powder) containing 50·8 per cent. of CaO and 24·11 per cent. of  $P_2O_5$ ; and bone meal, consisting of 92 per cent. of fine bone meal, and containing 30·3 per cent. of  $P_2O_5$ , and 1·49 per cent. of nitrogen, were employed in proportional quantities along with sodium nitrate on potatoes (Saxon onion potato). Thomas-slag gave the best result, then the bone meal, the plots without phosphate manure being poorest. There was much rain in June.—D. A. L.

*Relative Manurial Value of Phosphate, with special reference to Thomas-slag, Bone-Meal, Peruvian Guano, and Coprolite Powder.* Prize Essay by G. Marek. Bied. Centr. 19, 142—144.

Numerous plants, including oats, peas, mangolds, sugar-beet, potatoes, carrots, barley, buckwheat, summer wheat, and beans were cultivated on a great variety of soils, such as clayey, loamy, sandy, humus, marshy and calcareous loamy soils, also rich, poor, and intermediate loams, and a clayey loam, the plant being selected to suit the soil. All received the same quantity of nitrogen, potash, lime, and phosphoric acid, but the form in which the last two were given was varied. During the progress of the experiments records were kept of the temperature of the air, of the cloudiness, of the direction of the upper (cloud) air currents, and of the precipitation. The warmth of the soil at depths of 5, 10, 20, 30, 40, 50, 60, 70, 80, and 100 cm., and the temperature of the original soils from which the experimental ones were taken to a depth of 1 cm. Some of the soils were sifted. The manuring varied as to periods of sowing and the quantity sowed, and for comparison, plots were left without manure. All observations were taken into consideration when discussing the results, and the experience gained in one year's experimenting was made use of in the succeeding year. Results are recorded under many different headings. The following are some of the general data relating to the value of phosphates:—

Crop.	Phosphates in Order of Merit.	Percentage yield above Unmanured.	
		Observed.	Mean.
Winter cereals .....	Thomas-slag .....	..	29
" .....	Bone-meal .....	..	23
" .....	Coprolite powder ..	..	22
" .....	Peru guano .....	..	10
Summer cereals .....	Peru guano .....	0·3—37	21
" .....	Coprolite powder ..	6·0—30	15
" .....	Bone-meal .....	0·5—26	12
" .....	Thomas-slag .....	1·0—19	10
Hoed crops .....	Peru guano .....	9—53	33
" .....	Bone-meal .....	12—47	27
" .....	Thomas-slag .....	8—26	24
" .....	Coprolite powder ..	7—41	23

After-action was investigated on the following kinds of soil:—

1. Calcareous loam.....after sugar beet.
2. Loam....." "
3. Sandy soil....." potatoes.
4. Clay....." mangolds.
5. Marshy soil....." potatoes.
6. Humus soil....." carrots.
7. Rich loam.

The crops selected were potatoes and sugar-beet; they were sown a year after the previous harvest, and were harvested when mature. Potatoes were grown on soils 1—6. No after-action was evident on the clay and marshy soils; three of the soils showed after-action, and one in some instances. The after-action of the different phosphates on potatoes on these soils is shown in the following results:—

—	Observed.	Mean.
	Per Cent.	Per Cent.
Bone-meal .....	0—13·7	9
Coprolite powder .....	2·2—16·2	8
Thomas-slag .....	—18·0—14·2	1
Peruvian guano.....	—12·6—5·6	—2·5

The rich loam served for the sugar-beet experiments and the after-action of the individual phosphates in this case was as follows:—

—	Percentage Yield above Unmanured.	
	Observed.	Mean.
Bone-meal .....	1·3—54·8	30
Thomas-slag .....	1·0—63·1	27
Coprolite powder.....	11·0—49·9	24
Peruvian guano .....	—10·0—51·0	18

The monograph terminates with a summary.—D. A. L.

*Influence of the Degree of Subdivision on the Activity of Thomas-slag on Barley.* Ulbricht. Der Landbote 1889, 833—835.

Pot experiments were conducted:—(1.) In sand to which was added per 4,000 grms. of sand, 0·2235 grm. of potassium chloride, 0·096 grm. of magnesium sulphate, 1·3120 grm. of calcium nitrate, 0·1596 grm. of ferric hydrate, and 11 grms. of humus acid. (2.) In marshy soil containing per cent. dry soil:—N, 3·39;  $K_2O$ , 0·29;  $Na_2O$ , 0·22;  $CaO$ , 4·06;  $MgO$ , 0·15;  $P_2O_5$ , 0·11;  $SO_2$ , 0·5; sand,  $Al_2O_3$ , &c., 13·98; loss on ignition 76·14, and which received 0·4784 grm. of potassium chloride; the moistness during the experiment amounted to 100 to 150 per cent. of the dry marsh soil. The barley seeds were covered with a 2-centimetre layer of glass sand. Basic slag of various degrees of fineness, as indicated in the following table, were added to the pots, the quantities being:—0·1417 of phosphoric acid as monocalcium phosphate, twice that quantity in the other direct experiments, and six times that quantity in the additional C and G experiments.

The weights are in grms. and are in all cases from three pots. The results show the efficiency of the slag in marshy soil for barley, and that slag in fine sub-division is about half as active as monocalcium phosphate (superphosphate). The larger dressings of slag produced no decisive result.

—D. A. L.

For Table see next page.

Manuring and Condition of the Thomas-Slag.	Sand.		Marshy Soil.	
	Grain.	Straw, &c.	Grain.	Straw, &c.
No slag.....	.. ..	.. ..	1.03	20.60
Monocalcium phosphate .....	21.54	30.80	.. ..	.. ..
A. Commercial slag, 22.24 per cent. of $P_2O_5$ , 75.9 per cent. "fine powder" .....	26.35	30.62	51.33	64.
C. Grains of 0.75—1.0 mm. diameter, 20.35 per cent. of $P_2O_5$ , 5.15 per cent. of "fine powder" .....	13.92	17.83	27.41	28.54
F. Grains of 0.25—3 mm. diameter, 20.35 per cent. of $P_2O_5$ , 3.75 of "fine powder" .....	22.11	23.21	39.41	39.15
E. Coarse powder (sieve residues), 22.37 per cent. of $P_2O_5$ , and retaining 49.05 of "fine powder" .....	23.91	30.4	48.95	48.69
H. The coarser portions of the "fine powder," diameter 0.1 to 0.17 mm., with 22.64 per cent. of $P_2O_5$ and 99.3 of "fine powder" .....	25.77	30.15	51.5	52.59
D. Fine powder 99.9 per cent., $P_2O_5$ 22.75 per cent. ....	25.70	32.17	41.4	50.55
G. Finer portions of "fine powder" below 0.1 mm. diameter, 22.96 per cent. of $P_2O_5$ , and 99.55 of "fine powder" .....	25.29	33.79	51.15	50.77
C. Additional .....	19.30	22.70	42.02	41.29
G. Additional .....	23.03	28.78	56.40	57.09

—D. A. L.

*Direct Assimilation of Ammonia by Plants.* A. Muntz.  
Jour. d'agric. prat. 1889, 671–673.

BEANS, field beans, maize, barley, and hemp, were successfully cultivated by the author under the following circumstances:—The soil was washed free from nitrates, mixed with ammonium sulphate, and sterilised at  $100^{\circ}\text{C}$ .; whilst the seeds were dipped into boiling water, just before sowing, to destroy adherent organisms. The plants were reared under covers consisting partly of glass and partly of linen, which whilst it prevented access of germs allowed free circulation of air, and as a further precaution, the walls were smeared with glycerol. The air in the room was moreover purified by water spray. Blank experiments showed that no nitric acid was formed in these sterilised soils, under these conditions; whereas non-sterilised soils produced an energetic nitrification. It is concluded that the plants in question can make use of ammoniacal nitrogen without the intervention of nitrification.—D. A. L.

*Natural and Metallurgical Phosphates.* L. Ravel.  
Jour. d'agric. prat. 1889, 969–973.

SUGAR-beet was cultivated on an acid moorland, and on a fine sandy soil rich in humus and containing per cent. 0.045 of  $P_2O_5$ , 0.06 of  $K_2O$ , 4.0 of  $CaO$ , 0.41 of  $N$ . The results confirm the generally accepted view as regards the special adaptability of Thomas-slag to acid soils and to those deficient in lime: consequently, in comparison with other natural phosphate, the slag showed to advantage on the first soil above mentioned, whilst on the second soil these two phosphates were practically alike financially, although the natural phosphate was more potent when used without other manure and the slag when dung and potash were also supplied. These phosphates yielded lower results when mixed together than when applied individually, and the best results were obtained in conjunction with dung and dung and potash. No benefit was derived from dressings of more than 1,000 kilos. of phosphate per hectare, therefore it is deemed superfluous to employ larger quantities.—D. A. L.

*On the Utilisation and Transformation of certain Alkaloids in the Seed during Germination.* E. Heckel. Compt. Rend. 110, 88–90.

THE author shows that the function of alkaloids in seeds cannot be merely, as has been supposed, to protect them from animals, but that they serve as a nitrogenous reserve for the young plant.

The germination of *Sterculia acuminata* (P. de Beaur) containing caffeine, *Strychnos nux vomica* (strychnine and

brucine), *Datura stramonium* (daturine), and *Physostigma venenosum* (esarine) was examined. In each case the alkaloid in the germinating structures was found to disappear rapidly, and to be replaced by other nitrogenous compounds, e.g., chlorophyll and potassium nitrate.

These results agree with the observations of Réveil (De l'action des poisons sur les plantes, Lyons, 1885) that if plants be watered with solutions of the organic poisons their seeds contain, they die.—P. J. H.

*Researches on the Culture of the Potato for Manufacturing Purposes and Forage.* A. Girard. Compt. Rend. 110, 176–179.

THE author describes experiments on a variety of potato hitherto little known in France, the *Richter's Imperator*, with which remarkable results have been obtained. The average yield of potatoes per hectare in France is 7,500 kilos.; 15,000—18,000 kilos. being regarded as excellent. Experiments carried out on the Government farm at Joinville-le-Pont with the new variety gave (1) in 1888 a yield of 33,000 kilos. per hectare, containing 17.6 per cent. of anhydrous starch; (2) in 1889, with a much better season, 39,000 kilos. per hectare, containing 20.4 per cent. of anhydrous starch.

By permission of the Government 6,000 kilos. were distributed to farmers in different parts of France. For the year 1889, 16 followed the author's prescriptions exactly, and obtained yields varying from 32,000 to 44,000 kilos. per hectare, with the percentage of anhydrous starch varying from 20.4 to 24.2 per cent.; with a mean of 36,000 kilos. of tubers and 7,900 kilos. of starch per hectare.

Seventeen farmers who neglected certain details (i.e., planted too far apart, manured insufficiently, sowed parts of tubers instead of whole tubers, dug up too early) obtained yields varying from 13,300 to 30,000 kilos. per hectare.

A diagram of the yields in different parts of France is given.

The author predicts that this solution of a problem, at which he has been working for six years, will enable France to compete with Germany in its production of potatoes and that potato spirit in France will compete with that obtained from the distillation of grain. Thus the value of the maize for the purpose of producing alcohol equivalent to the yield of this new potatoes per hectare (1) under unfavourable circumstances, (2) under favourable circumstances, would be respectively 1,157 francs and 1,612 francs. Details of the experiments are given in the *Annales Agronomiques*, 16, 5.—P. J. H.

*Experimental Cultures at High Altitudes.* G. Bonnier  
Compt. Rend. **110**, 363—365.

EXPERIMENTS were undertaken to determine by what means the necessary stock of reserve is accumulated by Alpine plants in their subterranean tissues, during the very short Alpine summer.

Since 1884 a series of experimental plantations has been established in the Alps and the Pyrenees, at heights varying from 50 to 2,300 metres. At many of the lower stations the soil used was brought down from the upper stations for the sake of more perfect comparison; and the plants experimented on at different altitudes were cuttings from the same individuals. The number of species investigated was 165.

The conclusions are summed up as follows—

With plants cultivated in Alpine regions under conditions similar, in every respect but that of climate, to those under which plants of the same species were grown at lower altitudes, it was found that—

1. The acrian stalks are more spreading, shorter, and bend down more towards the ground.
2. The flowers are more highly coloured, the leaves thicker, and of a darker green.
3. The protective tissues of the stem are more developed.
4. Owing to the greater thickness of the tissue of palisade cells, and to the abundance of chlorophyll, the assimilation by the leaves per unit of surface is greater.

This last observation shows that the smaller development of the acrian parts of plants is compensated for by the greater rate of nutrition.—P. J. H.

*Observations on the Reactions between Vegetable Soil and Atmospheric Ammonia.* Berthelot. Compt. Rend. **110**, 558—560.

THE soil is constantly giving off ammonia and at the same time absorbing it from the air, which contains, in Paris, about 1 mgrm. in 50,000 litres. It is possible, by altering the composition of the air on the one hand, and the condition of the soil as to amount of surface exposed, the amount of moisture, and the amount of decomposition going on in it, on the other hand, to ensure the nearly complete absorption of the atmospheric ammonia by the soil or the evolution of much of the nitrogen, already in the soil, as ammonia. Thus, conclusions drawn from experiments are only applicable to cases where the conditions are exactly the same, cases which may by accident recur, but can hardly be relied upon as doing so.

The uniform correction which Schloesing makes for ammonia absorbed in his experiments is thus inaccurate; moreover, it is obtained by regarding a surface of dilute sulphuric acid as having the same power of absorbing ammonia as an equal surface of soil which may be absorbing ammonia at one point and giving it up at another. The same *savant* maintains that the absorption of nitrogen in his experiments is due only to absorption of atmospheric ammonia, but he does not prove it directly by determining the amount of ammonia lost by the air passed over the soil.

Fixation of ammonia from the air, if occurring at all must be very small; most *savants* are agreed as to this, and also allow that the atmospheric ammonia must be chiefly derived from the decay of nitrogenous organic matter derived from vegetation.—A. G. B.

*Distribution of Aluminium in Plants.* L. Ricciardi. *Gazetta Chem. Ital.* **19**, 150—159.

ALUMINA is found in the ash of all Italian plants. It is in the most assimilable form in moderately calcareous soils, and less so in clay, highly calcareous soils, and soils from igneous rocks. The ash of vines grown on—

	Per Cent. of Alumina.
Clay soil contained .....	0.850
On highly calcareous soil contained.....	0.810
On medium calcareous soil contained.....	1.140

The alumina is most abundant in the stems and stalks, less so in the seeds and skins, and least in the leaves. The following are a few results obtained:—

	Alumina, per Cent. in the Ash.
Mandarin orange—stem and stalk.....	0.218
Mandarin orange—peel and seed.....	0.093
Mandarin orange—leaves.....	0.022
Figs, Bari.....	0.063
Figs, Catania.....	0.092
Dry almonds—shell.....	0.185
Dry almonds—nut.....	0.138
Ceratonia siliqua, Bari—pulp.....	0.502
Ceratonia siliqua, Bari—seed.....	0.062
Ceratonia siliqua, Catania—pulp.....	0.607
Ceratonia siliqua, Catania—seed.....	0.064
Tobacco leaf.....	2.151
White lupin.....	0.012
Apulian wine, grms. per litre.....	0.022

Besides alumina, manganese was always detected.

—D. A. L.

*Examination of Vines after Treatment with Copper Salts.*  
E. Comboni. *Rassegna di Viticoltura ed enologia*, 1889,  
225—229.

THE author has made various determinations from which the following conclusions are drawn. The quantity of copper retained by the grapes depends on the care taken in the application of the copper salt to the vine, whether the leaves only are covered or not. More copper may be expected in the wine when the copper salt is mixed with lime than when simple copper sulphate solution is used. Grape skins and pulp have a noteworthy amount of copper; the stalks and stones also contain it, the latter in very small quantity, frequently only a trace. Thick wine contains more than clear. Further experiments are required to ascertain if the presence of copper inside the grape has anything to do with the developing processes.—D. A. L.

#### *The Saxmundham Experimental Farm.*

THE East Suffolk Chamber of Agriculture has issued a report of some experiments carried out during the last eight years by Mr. Edward Packard, jun., on his experimental farm at Saxmundham, Suffolk. The soil is poor, and the crops accordingly were not at all heavy where wheat was grown continuously, except in one year out of the eight, when the land was foul and the crop was missed. What is called a complete manure, composed of 33 lb. of lime per acre, 22½ lb. of magnesia, 75 lb. of potash, 47 lb. of phosphoric acid, and 40 lb. of nitrogen (equal to 2 cwt. of sulphate of ammonia), was used on one plot, while another plot had no manure, and from each of the rest one of the ingredients of the complete manure was omitted. The average produce of the seven years' cropping is given below:—

Manures.	Produce per Acre.	
	Bush.	Pecks.
None.....	21	1½
Complete manure.....	30	2
Lime omitted.....	28	3½
Potash omitted.....	29	0½
Nitrogen omitted.....	26	1½
Phosphoric acid omitted.....	26	0½
Magnesia omitted.....	30	1½

There was an increase of about nine bushels an acre from the use of the complete manure; but this was scarcely enough to pay expenses. The omission of the lime from the complete manure reduced the yield by nearly two bushels an acre, and there was a greater reduction when

nitrogen or phosphoric acid was left out; but the benefit of the magnesia was practically nothing, and that of the potash was only a bushel an acre. It is strange that on poor land the omission of the nitrogen did not make more difference in the yield than is shown by the figures. In similar experiments with barley, the average results of seven years show a yield of about 24½ bushels on the unmanured plot, 41 from the complete manure, 36¾ when phosphoric acid was omitted, 34¼ when nitrogen was left out, nearly 38½ without potash, 37¾ without lime, and 37 when magnesia was omitted. All the ingredients, then, were necessary to the production of a full crop of barley. On other barley plots, 4 cwt. of superphosphate and 2 cwt. of kainit per acre were used in each case, and nitrogenous manures in different forms were added to different plots, each dressing containing 53 lb. per acre of nitrogen. Muriate of ammonia gave the greatest yield, or 38 bushels an acre, nitrate of soda coming next with only a quarter of a peck less, and then sulphate of ammonia, with 4½ pecks less. The crops were grown continuously on the same land. For mangolds, the complete manure was made up of the following ingredients, the quantities of the more important substances being those removed by an average crop, excepting that the quantity of nitrogen was only half as much as is removed:—96 lb. of nitrogen, 322 lb. of potash, 51 lb. of phosphoric acid, 36 lb. of lime, 81 lb. of soda, 36 lb. of magnesia, 122½ lb. of sulphuric acid, and 92 lb. of chlorine. The average produce of seven years on the unmanured plot was 24,054 lb. an acre, and that of the plot to which the complete manure was applied was 48,935 lb. When nitrogen alone was left out the yield was only 36,754 lb., and when phosphoric acid was omitted it was no more than 39,570 lb.; but the omission of any of the other ingredients made much less difference, though where magnesia was withheld the yield was 3,422 lb. less than where the complete manure was used, and the absence of lime or potash caused a decrease of about 1,500 lb. Dissolved phosphate (or superphosphate) proved greatly superior to ground coprolite.

#### PATENTS.

*Improved Method of and Means for Preserving Grass, Cereals, Herbage, or other Vegetable Substances, as Eusilage or Food for Cattle and other Animals.* J. Pearson, Wolverhampton. Eng. Pat. 4363, March 13, 1889. 6d.

This invention relates to the application of an exhaust fan worked by hand or other power to silos, and also to providing them with drains, for the emission of liquids, which can be closed air-tight.

The silo, or receptacle, filled with grass or cereals, &c., so as to leave a vacant space at the top, is made air-tight or nearly so; the exhaust fan and pipe are then attached, the latter either penetrating into the mass or preferably only entering the vacant space, and by working the fan the volume of internal air is reduced to effect the preservation of the contents.—D. A. L.

*Improved Manurial Composition or Stimulant for Plants, Bulbs, Trees, and other Vegetable Produce.* A. Booty, High Harrogate. Eng. Pat. 8782, May 27, 1889. 4d.

This invention relates to a manurial composition containing the following ingredients in about the quantities given:—1 ton prepared fuller's earth; 1 ton of clay; 2½ cwt. of calcium sulphate; 2½ cwt. of neutral calcium phosphate; 2½ cwt. of ammonium sulphate; 1½ cwt. of potassium nitrate; ¾ cwt. of fused potassium chloride; ¼ cwt. of ferrous sulphate and a small quantity of gelatin. The proportions may, however, be varied to suit the plant. They are pulverised and mixed dry.—D. A. L.

## XVI.—SUGAR, STARCH, GUM, Etc.

*The General Meeting of the Society of Distillers; and the Society of Starch Manufacturers, in Germany.* February 28 and March 1890. Chem. Zeit. 14, 305–306.

1. *Meeting of Starch Manufacturers.*—Saare discussed the question of the grinding of potatoes, and expressed his opinion that a more complete subdivision than is at present possible by mechanical means is not to be expected. It remains to be seen whether a bacterial and a slightly acid fermentation will more effectually separate the starch cells. By the use of grinding-mills to still further subdivide the pulp from the graters, a larger yield of starch is undoubtedly obtained, but the sieves are easily blocked by the extremely fine pulp thus produced. Of the more modern forms of apparatus the compound and excelsior graters are the most efficient. As regards the feasibility of adding starch to rye and wheat flour to be used in bread making, Delbrück recommends the addition of 10–20 per cent. of starch; and states that there is no difficulty in incorporating the previously gelatinised starch with the flour. Zuntz reports concerning its nutritive value, and shows that bread baked with the addition of skim-milk and 20 per cent. of starch does not contain appreciably less albuminoid matter than bread baked without the addition of starch.

2. *Meeting of Distillers.* See under XVII., page 529.

—H. T. P.

*On the Presence of Sugar-yielding insoluble Carbohydrates in Seeds.* W. Maxwell. Amer. Chem. J. 12, 51–60.

In the chemical analysis of seeds and plants there has generally been admitted a large percentage residue of "undetermined matter." These undetermined matters, excluding nitrogenous substances whose nature is not fully understood, have been summarily expressed as nitrogen-free extract matter (N-freie Extractstoffe). The author has investigated the legumes, *Pisum sativum*, *Faba vulgaris*, and *Vicia sativa*, with especial regard to the non-nitrogenous insoluble residues. As a result of these experiments it appears that the insoluble non-nitrogenous bodies of legumes are, exclusive of cellulose, chiefly carbohydrate bodies more or less convertible by dilute mineral acid into sugars. The sugar most constantly yielded by hydration of these carbohydrates is galactose, indicating that the carbohydrate is paragalactin. About one-fifth of the total matter of the seed, *Pisum sativum*, consists of a carbohydrate wholly convertible into galactose. The seeds of *Faba vulgaris* and *Vicia sativa* contain respectively 14 and 15 per cent. of the non-nitrogenous bodies, but only about one-half of the substance in this case yields sugar with mineral acids. Kramer has determined microscopically that the bodies are secreted in the cells of the endosperm, and must be regarded as a reserve material of the seed and as contributing to the support of the embryo during germination. Chemically these carbohydrates are distinguished from the soluble carbohydrates by their insolubility in water and diastase.—A. J. K.

*Organic Acids in the Juices of the Sorghum Cane.* H. W. Wiley and W. Maxwell. Amer. Chem. J. 12, 216.

PRELIMINARY investigation has revealed in the juices of the sorghum cane the following acids which have been obtained as free crystallised bodies or as salts. The experiments also indicate that the acids are present in the following order by proportion:—Aconitic, citric, malic, oleic, formic, tartaric, and traces of acetic and oxalic. The researches will be continued next season.—A. J. K.

*Note on Xylose and Gum from Straw and other Materials.*  
Allen and Tollens. Ber. 23, 137.

SEEING that many vegetable products give furfural when boiled with acids and a red colouration on warming with phloroglucinol and hydrochloric acid, which reactions are characteristic of the pentaglucooses, arabinose and xylose, the authors deemed it of interest to determine if either of these sugars could be isolated from those substances.

They have examined beechwood, wheat, straw, loofah, cherry-tree wood. On extracting loofah, cherry-tree wood, and straw with soda lye, and precipitating the extract with alcohol, a gum was obtained (15 per cent. from straw), which gave xylose on hydrolysis. The production of furfural is more regular when the substances are distilled with hydrochloric acid instead of sulphuric acid. The authors hope soon to be able to communicate further on these matters.—A. L. S.

*Syntheses of Mannose and Levulose.* E. Fischer. Ber. 23, 370—394.

THE lactone of mannonic acid is so similar in its properties to that of arabinose-carboxylic acid, that it would be impossible to distinguish between them if it were not that the first is dextro-rotatory and the second levo-rotatory, the numbers expressing the rotation being almost the same in the two cases. On mixing solutions of these they unite to form an optically inactive lactone, which yields inactive salts, and this cannot be easily again separated into its two constituents.

All three lactones yield by reduction first a sugar and then a hexahydric alcohol, these reduction products standing in exactly the same relation to one another as the lactones.

As it is advisable to have a systematic notation for these and similar bodies, the author recommends that the letters *l*, *i*, or *d* be prefixed respectively to the names of the bodies according as the sugars from which they are derived are levo-rotatory, inactive, or dextro-rotatory. The following table shows the relation of these bodies to one another:—

<i>d</i> -Mannose (ordinary mannose levo-rotatory).	<i>i</i> -Mannose.	<i>l</i> -Mannose (levo-rotatory).
<i>d</i> -Mannose-phenyl- hydrazone (levo-rotatory).	<i>i</i> -Mannose-phenyl- hydrazone.	<i>l</i> -Mannose-phenyl- hydrazone (dextro-rotatory).
<i>d</i> -Mannonic acid.	<i>i</i> -Mannonic acid.	Arabinose-carboxylic acid ( <i>l</i> -Mannonic acid).
<i>d</i> -Mannonic acid lactone (dextro-rotatory).	<i>i</i> -Mannonic acid lactone.	Arabinose-carboxylic acid lactone (levo-rotatory).
<i>d</i> -Mannitol (dextro-rotatory in presence of borax).	<i>i</i> Mannitol.	<i>l</i> -Mannitol (levo-rotatory in presence of borax).
<i>d</i> -Phenylglucosazone (ordinary glucos- azone) (levo-rotatory in acetic acid solution).	<i>i</i> -Phenylglucosazone	<i>l</i> -Phenylglucosazone (levo-rotatory in acetic acid solution).

*l*-Mannose is obtained by reducing arabinose-carboxylic acid with sodium amalgam. The solution is cooled before the addition of sodium amalgam, and sulphuric acid is added from time to time in sufficient quantity to keep the solution acid. The reduction is complete in about three-quarters of an hour, the neutralised solution is evaporated, and the sodium sulphate allowed to crystallise out. The mother-liquor is mixed with 20 times its volume of alcohol, and the alcoholic filtrate from this on evaporation gives a colourless syrup of *l*-mannose. The yield of sugar is 50 per cent. of the theoretical. It is easily soluble in water, with difficulty in alcohol, and fairly easily in methyl alcohol. It is levo-rotatory, although the exact amount could not be determined as the body was not obtained in a pure state. Its hydrazone is dextro-rotatory, and melts at 195°, with evolution of gas.

*l*-Phenylglucosazone is formed by heating the above hydrazone with twice its weight of phenylhydrazine acetate. It is very similar to *d*-phenylglucosazone (from *d*-mannose, levulose, and dextrose); it melts at 205° with evolution of gas. It is dextro-rotatory. Concentrated hydrochloric acid decomposes it into phenylhydrazine hydrochloride and its osone, and this latter gives by reduction with zinc and acetic acid, *d*-levulose, the formation of which from *l*-levulose is given further on. *l*-Mannose is with great difficulty, if at all, fermentable with beer yeast.

*l*-Mannitol is formed by the reduction of *l* mannose with sodium amalgam, but this reaction takes place much more slowly than the reduction of the lactone to *l*-mannose. The reduction is finished in about 12 hours. The solution is then neutralised, evaporated until sodium sulphate commences to crystallise out, mixed with 20 times its volume of alcohol, filtered, and evaporated, when *l*-mannitol remains as a white crystalline mass; it is purified by recrystallisation from methyl alcohol. It is very similar to ordinary mannitol melting 2° lower (163°—164°), easily soluble in water, with difficulty in alcohol, and much more easily in methyl alcohol. It has a sweet taste, and does not reduce Fehling's solution. In the presence of borax it is strongly levo-rotatory.

*i*-Mannonic Acid.—If equal parts of mannonic acid lactone and arabinose-carboxylic acid lactone be dissolved in water, the solution is optically inactive, and on evaporation yields a colourless radiating mass of crystals of the lactone of *i*-mannonic acid. This is easily soluble in hot water, and on cooling deposits radiating masses of long prisms; it is with difficulty soluble in hot alcohol, and on cooling is similarly deposited in radiating masses of long needles. It melts at a slightly higher temperature than its components, has a sweet taste, and does not reduce Fehling's solution. It is optically inactive, even in strong solutions, and cannot be separated into its components by crystallisation. It forms optically inactive salts, which may be distinguished from those of arabinose-carboxylic acid and of mannonic acid by the difference in their solubilities, &c. It also forms basic salts, which is not the case with its two component acids; but in which behaviour it resembles gluconic acid. Heated with phenylhydrazine acetate it yields a hydrazone.

The author has applied the two methods which Pasteur employed to resolve inactive tartaric acid into its components, so as to obtain *i*-mannonic acid.

By fermentation with *Penicillium glaucum*, little if any separation was effected. By means of the strychnine salts a complete decomposition into dextro- and levo-rotatory acids was effected. The base and lactone are dissolved in molecular proportions by boiling in 70 per cent. alcohol; the salt is obtained on evaporation; it is rubbed down and washed with alcohol. It is soluble in hot water; small quantities added to hot alcohol dissolve at first to a clear solution, but in a very short time a crystallisation of the difficultly soluble arabinose-carboxylic acid begins, the greater portion of this crystallises out, leaving the more easily soluble salt of *d*-mannonic acid in solution. These two salts may then be purified by recrystallisation, &c.

*i*-Mannose is prepared from *i*-mannonic acid lactone in the same way as *l*-mannose is, from the *l*-acid. It is a colourless syrup, easily soluble in water, with difficulty in alcohol, and fairly easily in hot methyl alcohol. Except that it is optically inactive, it exactly resembles *d*- and *l* mannose. It yields a hydrazone in the usual way, which melts with decomposition at 195°.

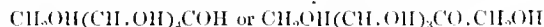
As *l*-mannose is unfermentable and *d*-mannose easily fermentable by beer yeast, it might be expected that *i*-mannose would undergo a partial fermentation under the influence of beer yeast. This is found to be the case, and the unfermentable residue is found to be *l*-mannose.

*i*-Phenylglucosazone is formed by heating *i*-mannose with twice its weight of phenylhydrazine and sufficiently dilute acetic acid. The reaction takes some hours, and the yield is rather smaller than usual. It melts at 217°—218° with decomposition, and in acetic acid solution is optically inactive. This body is identical with  $\alpha$ -erosazone, and the author proposes in future to call it by this name.

*i*-Mannitol ( $\alpha$ -acritol) is formed by the reduction of *i*-mannose, just as *l*-mannitol is obtained from *l*-mannose. The yield is 40 per cent. on the weight of sugar. It is purified by recrystallisation from methyl alcohol. It melts at 170°. It is very soluble in warm water and crystallises out on cooling in small prisms; it is fairly easily soluble in hot glacial acetic acid, and with great difficulty in ethyl and methyl alcohol. It is optically inactive and is identical with  $\alpha$ -acritol. The author points out that in identifying any particular hydrazone or osazone it is not sufficient to show that it agrees in melting point, solubility, &c., with some known hydrazone or osazone, as this would lead one to conclude that the derivatives from *d*-, *i*-, and *l*-mannose were identical; but also that it is necessary to show that their action on polarised light is the same, this being the only satisfactory way of differentiating such bodies as derivatives of these three sugars.

**Constitution of  $\alpha$ -Acrose.**—A sugar has been prepared from acrolein bromide, glycerose (a mixture of glycerol and dioxacetone), and formaldehyde. It can only be isolated as  $\alpha$ -acrosazone; this may be converted into the osone by the action of hydrochloric acid, and the latter yields by reduction with zinc and acetic acid a sugar ( $\alpha$ -acrose) which may or may not be identical with the original one. It yields by further reduction  $\alpha$ -acritol, which is identical with *i*-mannitol;  $\alpha$ -acrosazone is identical with *i*-phenylglucosazone.

The constitution of  $\alpha$ -acrose may be either—



as both would yield the same osazone and the same hexahydric alcohol. The first is the formula of mannose, the second of levulose. *i*-Mannose yields a very characteristic difficultly soluble hydrazone, whereas  $\alpha$ -acrose does not, so that  $\alpha$ -acrose is *i*-levulose, and its constitution is expressed by the second formula. It may be that the originally obtained sugar is not identical with that obtained by reduction of the osone; a solution containing this sugar was prepared from acrolein bromide and formaldehyde. In both cases the reaction with phenylhydrazine did not correspond to that of *i*-mannose, and so in both cases the sugars were *i*-levulose.

**Fermentation of *i*-Levulose ( $\alpha$ -Acrose).**—The synthetically obtained sugar ferments easily with beer-yeast, and after fermentation has a strong dextro-rotation, which is evidently due to *l*-levulose;\* as the solution yields an osazone, which is identical with *i*-phenylglucosazone.

**Conversion of *i*-Mannitol into *i*-Mannonic acid.**—Nitric acid oxidises *i*-mannitol to *i*-mannose just as ordinary mannitol is oxidised to mannose. The *i*-mannose is obtained from the neutralised solution as phenylhydrazone. *i*-Mannitol prepared in one case from *i*-mannonic acid, and in another synthetically from glycerol gave, in both cases, the same sugar as phenylhydrazone; which was identical with *i*-mannose phenylhydrazone; and when decomposed with hydrochloric acid gave *i*-mannose.

Bromine water oxidises the sugar to mannonic acid, which is obtained by heating with phenylhydrazine acetate as a hydrazone. This yields the free acid when decomposed by baryta water, and the baryta removed by sulphuric acid.

The *i*-mannonic acid is then decomposed as described above into *d*-mannonic acid and arabinose-carboxylic acid. The synthesis of all the bodies of the mannose group, except dextrose, has now been made, and may be briefly summarised as follows:—

The sugar,  $\alpha$ -acrose, synthetically obtained from acrolein bromide, glycerose or formaldehyde yields by heating with phenylhydrazine and acetic acid *i*-phenylglucosazone, which with hydrochloric acid yields *i*-glucosone, by reduction with zinc and acetic acid this yields *i*-levulose and with beer yeast it partially ferments, thus leaving *l*-levulose. Returning to *i*-levulose, by reduction with sodium amalgam *i*-mannitol ( $\alpha$ -acritol) is obtained, this oxidised with nitric acid yields *i*-mannose and this oxidised with bromine water yields *i*-mannonic acid; the strychnine or morphine salts of this

may be decomposed into the salts of *l*-mannonic and *d*-mannonic acid; *l*-mannonic acid (arabinose-carboxylic acid) may be reduced first to *l*-mannose and then to *l*-mannitol; *d*-mannonic acid may be reduced to *d*-mannose and *d*-mannitol (ordinary mannitol); *d*-mannose may be converted into *d*-phenylglucosazone by phenylhydrazine acetate, this is decomposed by hydrochloric acid yielding *d*-glucosone and then by reduction yields *d*-levulose (ordinary levulose); or the *d*-phenylglucosazone may be reduced to *iso*-glucosamine which decomposed by hydrochloric acid yields *d*-levulose.

*d*-Mannonic acid and gluconic acid are closely related, the author hopes soon to be able to synthesise the latter, and consequently also dextrose. In the coming year the author proposes to determine if, besides dextrose and other sugars of the *d*-mannitol group, there are also formed by plants derivatives of the *l*- and *i*-mannitol group. He thinks that this is probable, as arabinose belongs to the *l*-mannitol group; and that possibly the formation of the carbohydrates in plants may take place on somewhat similar lines to those of the above syntheses.—A. L. S.

#### On the Preparation of Levulose-carboxylic Acid.

H. Kiliani and G. Düll. Ber. 23, 449—451.

HAVING had to prepare levulose carboxylic acid in quantity, in order to investigate the action of oxidising agents on it, the authors not being satisfied with the previously described methods (Ber. 18, 3070; 19, 221 and 1914), have devised one more satisfactory in every way.

They do not recommend Fischer and Passmore's method (Ber. 22, 2728), in which use is made of the phenylhydrazide of the acid, but recommend the preparation of the pure cyanhydrin and the decomposition of this by hydrochloric acid, particular attention being paid to the two items that: (1) the levulose syrup be prepared according to König (Monatsh. 1883, 562), and so that it contains 25—30 per cent. of water, and that (2) the chief portion of the hydrochloric acid be expelled by evaporation.

The authors give very full directions as to the carrying out of the experiments.

The pure lactone of levulose-carboxylic acid gives with aqueous ammonia a fine crystalline derivative, not yet fully investigated. On oxidation with nitric acid a polybasic acid is formed, which may be isolated as a crystalline calcium salt which will be further investigated by the authors.—A. L. S.

#### Synthesis of Grape Sugar. E. Fischer. Ber. 23, 799—805.

THE author converts *d*-mannonic acid into gluconic acid by heating 20 parts of the former with 40 parts of quinoline and five parts of water for 20 minutes at 150° C., and then for 40 minutes longer at 140° C. A solution of 40 parts of crystallised barium hydrate is added, and the quinoline removed by distillation with steam. To separate from the *d*-mannonic acid the barium is precipitated with the exact amount of sulphuric acid; the filtrate from barium sulphate is evaporated to about 150 cc., and then boiled for half-an-hour with 60 grms. of brucine, which will be entirely dissolved. After standing for some time at ordinary temperature the excess of brucine separates out in crystals. The filtrate is evaporated until barium mamonate begins to crystallise, and then poured into 25 times its volume of absolute alcohol, in which barium mamonate is nearly insoluble. It begins at once to separate out. After filtration the alcoholic filtrate is again evaporated, the resulting syrup dissolved in water and decomposed with 20 grms. of crystallised barium hydrate, when the brucine is partly precipitated and separated in crystals. To remove that part of the base which remained dissolved the aqueous solution is again evaporated to a syrup and then treated with hot alcohol. The brucine is dissolved, whilst the residue contains the barium compounds. This is redissolved in hot water, the barium carefully precipitated by sulphuric acid, and the filtrate from the barium sulphate, containing gluconic acid, treated with animal charcoal. For further

\* The author suggests that if the name *l*-levulose be objected to that it might be called anti-levulose, and that the old name levulose might then be retained for the *d*-sugar.



purification the phenylhydrazide was prepared by heating the liquid, after concentration to 30 cc., with three parts of phenylhydrazine and the corresponding quantity of acetic acid. It melts at 206° C. The calcium, barium, and cinchonine salt of the synthetic gluconic acid were prepared from the hydrazide and proved to be identical with the salts obtained from the ordinary gluconic acid.

**Conversion of Gluconic Acid into Glucose.**—A solution of gluconic acid prepared from the pure calcium salt by precipitation with oxalic acid, is concentrated on the water bath to a syrup to convert the greatest possible amount of the acid into its lactone. It is then redissolved in 9 parts of water, the mixture cooled to 0° C., and a small quantity of sulphuric acid added to it. Sodium amalgam is added in small portions, and is very quickly consumed. It is necessary to add from time to time small quantities of sulphuric acid to keep the mixture acid. About 8 parts of the amalgam are required to 1 of the syrup. To isolate the sugar the solution is neutralised, evaporated until sodium sulphate begins to crystallise out, and poured into an abundant quantity of hot alcohol. Sodium sulphate is precipitated. The alcohol contains, besides glucose, sodium salts of organic acids. On repeating this method of purification several times, pure glucose crystallises from the alcohol after several additions of small quantities of ether. It has all the properties of ordinary glucose.—A. L.

**On a New Inosite called Racemo-Inosite.** Maquenne and C. Tanret. *Compt. Rend.* **110**, 86-88.

The authors discovered independently that the action of hydriodic acid on pinite yields methyl iodide and a dextro-rotatory inosite, the same reaction with quebrachite yielding methyl iodide and a levo-rotatory inosite. (See this Journal, 1890, 202 and 311.) They seek to show in this joint paper that these two inosites are in every way comparable to the dextro- and levo-rotatory tartaric acids.

(1.) They have equal and opposite rotatory powers  $[\alpha_D] = +65^\circ$  for the anhydrous sugars;  $[\alpha_D] = \pm 55^\circ$  in the hydrated state. The rotatory power is the same in 5 per cent. and 10 per cent. solutions, and whether the inosites be dissolved in hot or cold water.

(2.) The rotatory power of the acetic ethers were found to be  $[\alpha_D] = +9.75^\circ$  and  $[\alpha_D] = -10^\circ$ . They are both amorphous and soften when heated by the hand; and they both fuse without decomposition at 247°.

(3.) The two benzoic ethers fuse at 252°, and are but slightly soluble. They both crystallise with 2 H<sub>2</sub>O. The dextro-rotatory inosite has also been obtained anhydrous.

(4.) The two inosites crystallising with 2 H<sub>2</sub>O have about the same solubility; the dextro-rotatory compound dissolves in 2.13 parts of water at 14° and the levo in 2.3 parts at 14°. The anhydrous bodies both dissolve in 1.5 water at 11°.

(5.) They both behave in the same way towards reagents, and both crystallise in hemihedral rhomboidal prisms.

The authors have obtained the inactive inosite (corresponding to racemic acid) *racemo-inosite* by mixing 25 per cent. solutions of these two inosites; from this mixture *racemo-inosite* crystallises out in a short time. Its physical properties are quite different from those of its constituents and from those of ordinary inosite. It is optically inactive; it melts at 233° (30° higher than ordinary inosite) without previous softening. It is soluble in 26 parts of water at 11°, in 22 at 15°. The acetic ether crystallises; its melting point being 111°. The benzoic ether crystallises in cottony crystals, melting point 217°. Unlike ordinary inosite it does not turn Fehling's solution green when heated. Its composition is absolutely identical with that of the other inosites.

Since this inosite is the compound inactive by compensation, it seems certain that the ordinary inosite of muscular tissue, baricot beans, walnut leaves, &c., is the compound inactive by constitution. The authors propose to prove this experimentally. They note that their researches offer the first instance of optical isomerism in the sugars.

—P. J. H.

## PATENT.

*Improvements relating to Apparatus for the Manufacture of Sugar.* G. Adant, Brussels, Belgium. Eng. Pat. 7517, May 4, 1889. 8d.

This invention relates to apparatus for the manufacture of sugar in blocks, slabs, or bars, and is designed to improve the apparatus described by Eng. Pat. 13,084 of 1888 (this Journal, 1889, 810).—A. J. K.

## XVII.—BREWING, WINES, SPIRITS, Etc.

*The General Meeting of the Society of Distillers and the Society of Starch Manufacturers in Germany.* Berlin, February 28 and March 1, 1890. *Chem. Zeit.* **14**, 305-306.

1. *Meeting of Starch Manufacturers.*—See under XVI., page 526.

2. *Meeting of Distillers.*—According to Delbrück, the addition of hydrofluoric acid to worts greatly checks the growth of false ferments. Fermentation is somewhat checked, but the yield of alcohol is undiminished, and appears to be of a better quality. Lintner describes some experiments which he made in conjunction with Irmisch and Gronow to determine the species of yeast most suitable for the fermentation of strong distillers' worts. Thirty-seven pure cultures of (a) brewer's bottom-yeast; (b) brewer's top-yeast; (c) disease-forms of brewer's yeast; (d) white-beer yeast; (e), distiller's yeast; (f) pressed yeast, were placed, partly in wort, partly in 30 per cent. solutions of maltose. The results obtained varied greatly so far as the yield of alcohol, yeast-crop, amount of sediment, and of skimmings are concerned. Some varieties of white-beer yeast yielded very large yeast-crops, and would therefore be suitable for pressed-yeast manufactories. A sample of white-beer yeast also produced the largest amount of alcohol (16 per cent. of alcohol by volume). Delbrück discussed the effect of aeration on yeast and fermentation, and its utilisation in pressed yeast manufactories and in the fermentation of strong worts. Although aeration may cause a loss of alcohol up to 8 per cent. of the whole, yet by this means some species of yeast may be made to go on working till 16 per cent. of alcohol is formed. Delbrück recommends the employment of aeration combined with stirring apparatus for strong distillers' worts. The yeast used should be a pure culture, and the time of fermentation should be curtailed as much as possible. At the beginning, however, after the first aeration, it is well to somewhat check the rapidity of fermentation.—H. T. P.

*Production of Cider in France.* Soc. Arts J. **38**, 538-540.

This article is an abstract of the report of the United States Consul General at Paris. The quality of apples grown on various soils and the nature of the cider produced from them is first considered. The best soil is a mixture of clay and sand with plenty of moisture. It has been decided that to make the best cider the apples should have the following composition:—Water, 80 per cent.; sugar, 17.3 per cent.; tannin, 0.5 per cent.; mucilage, 1.2 per cent.; "fermenting acid," 0.167 per cent.; other substances, 0.833 per cent. Only very few varieties contain the above constituents in the desired proportions; and it is therefore usual to mix different kinds, only those of the same season being used. A list is given of varieties containing the principal constituents in the right proportions; and also of species rich in tannin and sugar respectively. There are three methods in use for reducing the apples to pulp. First, by a machine consisting of a circular trough, around which a stone wheel is made to revolve by horse-power. This machine is being superseded by two new kinds of apparatus. These are the grater—a machine with small saw-like teeth set close

together, and the crusher, a kind of magnified coffee mill, capable of producing 60—80 hectolitres (hectolitre = 2.75 bushels) of pulp per hour. This is the best machine, as it does not crush the pips, the essential oil of which imparts a disagreeable flavour to the cider. The pulp is allowed to macerate in vats for 12—15 hours. Before putting it into the press, the bottom of the latter is lined with a thin layer of straw, a wire netting, or small oak, or beech branches. Straw is objectionable, as it imparts a bitter taste to the juice, while the above named woods yield a little additional tannin, so necessary to preserve the cider. By the hydraulic presses now in use are obtained 65—75 per cent. of the pure juice at first pressure, without drainage. Diffusion processes have been tried in cider making. The older methods were only used in those cases where presses were not obtainable. Recently, however, new diffusion methods have been discovered by means of which the inventors claim to be able to get 85—95 per cent. of the pure juice. The apples are cut into pieces, not crushed, and placed in the diffuser; and the water, which is passed through the apparatus, drives the juice before it. Fermentation is carried on at 12° to 18° C. (54° to 64° F.). If fermentation is slow the juice is stirred vigorously with a bunch of twigs; or in extreme cases, a little yeast is added. This first fermentation is carried on for five to six weeks in open or closed vessels, or in casks with the bung drawn. The bung is then driven in and a small vent-hole made, which is kept open by a straw. After a month or six weeks, during which secondary fermentation goes on, the liquor is drawn off into air-tight casks, which are not quite filled. The dregs that collect in this space may then be removed. If the cider be not quite clear, owing to insufficiency of tannin in the apples, 60 grms. of eachon dissolved in a litre of cold cider, are added to every hectolitre. This precipitates the albuminoids and prevents the cider turning black on exposure to air. Jersey cider has the reputation of being the best. The process employed differs slightly from the one given. The primary fermentation takes place in open vessels at 15°—18° C. The liquor is then drawn into casks which are not quite filled. When sufficient gas has collected in the empty space to extinguish a candle, the juice is drawn off into other casks, and the process is repeated till no more gas forms. Champagne cider is of recent introduction and owes its popularity to its sparkling quality and great stability. A cider is prepared that will keep in cask for five or six years, and can be exported to India or South America. It contains from 10 to 11 per cent. of alcohol. Before fermentation 50 grms. of cream of tartar and 500 grms. of green beech chips are added to every hectolitre. After fermentation the cider is heated by means of an anethiome and instantly cooled again. This apparatus costs about 16*l*. The process, however, is said to injure the flavour and quality of the cider.

The total quantity of cider produced in France in the year 1887 amounted to 295,606,674 gallons, as compared with 182,616,676 gallons in the preceding year.—H. T. P.

#### *Researches on the Germination of some of the Gramineæ.*

Part I. H. T. Brown and G. H. Morris. Proc. Chem. Soc. 1890 (81), 52—57.

In this, the first part of their paper, the authors confine themselves almost entirely to a consideration of the changes which take place in barley during the early periods of its growth. The paper is divided into 21 sections, the heads of which are as follows:—

(1.) Introduction. (2.) Structure of a grain of barley. (3.) The visible changes which occur in the embryo and endosperm during germination. (4.) The relation of the embryo to the endosperm. (5.) Development of excised embryos upon foreign endosperms. (6.) The endosperm to be regarded as a store-house of *dead* reserve material; no residue vitality recognisable in its cells. (7.) Cultivation of excised embryos upon water. (8.) Cultivation of excised embryos upon nutrient solutions. (9.) Growth of excised embryos upon *starch*, and proof that an amylolytic (diastase) is secreted by the growing embryo. (10.) The secretion of diastase is localised in the "absorptive epithelium." (11.) The secretion of diastase is increased

by the presence of a small quantity of acid. (12.) The secretion of diastase not stimulated by the presence of starch. (13.) The secretion of diastase is inhibited by a readily assimilable carbohydrate. (14.) The existence of a *cellulose dissolving* enzyme (cyto-hydrolyst) in the germinating seeds of the Grasses. (15.) The cyto-hydrolyst, like diastase, is secreted by the "absorptive epithelium." (16.) The two varieties of diastase, their genesis and distribution in the resting and germinating seed. (17.) Distribution of diastase in the germinating seed. (18.) The "mother-substance" of the "diastase of secretion" is principally derived from the endosperm. (19.) Action of "diastase of secretion" upon ungelatinised starch. (20.) A consideration of the origin of the hydrolytic enzymes of germinated grain. (21.) The form in which the reserve starch after transformation enters the growing embryo, and the metabolic changes which it there undergoes.

In recording the visible changes which occur in the seed during germination, the authors show that a disintegration and dissolution of the cell-walls of the endosperm always precede any attack upon the cell-contents. This breaking down of the cell-wall is shown to depend on the production during germination of a special cellulose-dissolving ferment, which, like diastase, is soluble. The action of this enzyme on the cell-walls of some kinds of vegetable parenchyma is very energetic. The physiological importance of this soluble ferment is very great, for owing to the non-diffusible nature of diastase, the previous breaking down of the cell-wall is a necessary prelude to the dissolution of the contained starch-granules.

The authors show that the appearance of the cyto- and amylolytic ferments is due to a specialised secretory function of the layer of columnar epithelium, which covers the outer surface of the scutellum. It has hitherto been considered that the function of this epithelium was exclusively that of an absorptive tissue; its absorptive as compared with its secretory functions are, however, of quite secondary importance.

Starch has no special power of stimulating the cells of the epithelium to increased secretion of a diastase, and carbohydrates which are readily assimilable by the embryo, like cane sugar or maltose, check or completely inhibit the secretion of diastase. Carbohydrates, like mannitol or milk-sugar, which are entirely without nutritive value, exercise no such inhibitory action. The authors' experiments point to the secretion of the enzymes being to some extent *starvation phenomena*.

The authors confirm the important generalisation of Sachs, that the relation of the embryo to the endosperm is that of parasite to host, and they have availed themselves of the relation by cultivating the embryo upon suitable media after separating it from its endosperm, and in this way they have obtained information with regard to the secretory powers of the embryo, and the chemical modifications of its absorbed nutriment, which it would have been impossible to obtain by any other means. They show, for instance, that whilst cane-sugar, invert-sugar, dextrose, levulose, maltose, raffinose, galactose, and glycerol have all more or less nutritive value, milk-sugar and mannitol have no such value. Cane-sugar has the greatest nutritive power; maltose is decidedly inferior to cane-sugar in this respect. This is shown to be due to the fact that maltose, directly it is absorbed by the growing embryo, becomes transformed into cane-sugar by the living cells, and in this form is passed from cell to cell.

An examination of the sugars produced during the germination, and of their mode of distribution in the grain, have convinced the authors that the transformed starch of the endosperm is absorbed by the embryo in the form of maltose, and that the seat of production of the cane-sugar which germinated grain contains is the tissue of the embryo itself.

In the discussion that followed the reading of the paper, Professor Marshall Ward drew attention to the recent paper of Haberlandt, in which he maintained that the aleurone layer in the seeds of the Gramineæ and other families of plants, is the seat of the production of diastase. He also mentioned that the observations of Wortmann on the

behaviour of bacteria in a mixture of starch and proteids tended to confirm the authors' views regarding the secretion of enzymes.

Professor Green said that his observations led him to believe that in the case of the date the enzyme was probably located in the epithelial layer and not in the endosperm, whilst in castor-oil seeds both the endosperm and embryo appeared to be possessed of vitality.

Dr. Lauder Brunton commented on the resemblance between the embryo in highly organised plants and in animals. The embryo in both cases appeared to be purely parasitic.

Dr. Armstrong compared the action of protoplasm in converting maltose into cane sugar to that of phenylhydrazine in effecting the conversion of dextrose into levulose through the agency of the osazone.

In reply, Mr. Horace Brown said that the diastase developed during germination appeared to differ in important particulars from ordinary diastase. He also said that the experiments of Dr. Morris and himself did not favour the conclusion that the aleurone-layer had any marked diastatic power.—G. H. M.

#### Some Experiments on the Numerical Increase of Yeast Cells. A. J. Brown. Trans. Lab. Club, 3, 64—80.

THE author first states that in the experiments about to be described, he adopted the method of directly counting under the microscope the number of yeast cells, both before and after fermentation.

Having briefly mentioned the work of Pedersen, Delbrück, Durst, Hansen, Hayduck, and Foth, on the increase of "bottom fermentation" yeast under various conditions, the author mentions that no work has been done with "top fermentation" yeast, with the Burton type of which the whole of the experiments about to be described were performed.

The first experiments were made with a view of ascertaining what influence different strengths of ordinary hopped ale wort had upon the total increase of yeast cells when fermenting it. The experiments were carried out as follows:—

Three flasks were taken of a capacity of about 250 cc. To No. 1 flask was added 150 cc. of a filtered hopped ale wort, of the specific gravity of 1076.70. To No. 2 was added the same volume of the same wort which had previously been diluted with distilled water to the specific gravity of 1051.14. To No. 3 was added the same volume of the same wort but diluted to the specific gravity of 1025.57. To each of these flasks was added an equal volume of yeast. On well shaking the flasks and transferring a drop of the liquid to the haematimeter, it was found, on counting under the microscope, that each standard volume contained on the average 1.04 cells of yeast. The flasks were loosely plugged with cotton wool, immersed in a water-bath and kept at a temperature of 68° F. for three days, when all signs of fermentation were quite over. The flasks were removed from the bath, and, after being well shaken, a sample from each was placed in the haematimeter and the cells present again counted. It was now found that in each standard volume in No. 1 flask there were 21.52 cells; in No. 2, 20.24 cells; and, in No. 3, 14.80 cells. If from each of these numbers is subtracted the number of mother cells with which the fermentation was commenced (that is to say, 1.04), we find that in No. 1, 20.48 new cells have grown, in No. 2, 19.20, and in No. 3, 13.76. Hence it appears that, although the wort in No. 2 was only two-thirds the strength of that in No. 1 flask, yet the number of new cells formed is almost exactly the same; and further, although the wort in No. 3 flask was only one-third the strength of that in No. 1, yet the new cells formed are two-thirds the number of those in No. 1.

A second experiment was made with a different wort, in which No. 1 contained 100 cc. of the wort at the specific gravity 1106, and No. 2, 100 cc. of the same wort diluted with distilled water to the specific gravity of 1079.5. This experiment was conducted in the same manner as the previous one, with the result that at the conclusion of the fermentation No. 1 contained 34.10 new cells per standard

volume, and No. 2, 33.83. Here, again, we find two worts of rather high but different specific gravities, in which the total increase of the yeast had been the same. These experiments, and others which were made, all point to the conclusion that under the conditions of the experiments, worts exceeding some point near 1060 in specific gravity nourish the same number of yeast cells, and that below this specific gravity the number of cells formed is not in proportion to the specific gravity, but in a much higher one. These remarks, of course, only apply to a comparison of the same wort when diluted to different strengths. Worts brewed from different materials undoubtedly differ considerably from each other, as is seen when the results of the first two experiments are compared.

The above experiments having been made with malt wort, which contains both carbohydrate and nitrogenous yeast-food, it appeared desirable to conduct experiments in solutions in which either of these foods could be altered at will. In the first place experiments were made with yeast-water and varying amounts of dextrose, as follows:—

#### EXPERIMENT I.

No. 1.—100 cc. yeast-water containing 20 per cent. dextrose.....	Each flask yeasted with 1.23 cells pure yeast per standard volume.
No. 2.—100 cc. yeast-water containing 10 per cent. dextrose.....	
No. 3.—100 cc. yeast-water containing 5 per cent. dextrose.....	
No. 4.—100 cc. yeast-water containing 2 per cent. of dextrose.....	
No. 5.—100 cc. yeast-water containing no dextrose.....	

At the end of four days the fermentation was complete, and counting of the cells gave the following results:—

	Cells.
No. 1 contained.....	9.29
No. 2 ".....	10.02
No. 3 ".....	7.32
No. 4 ".....	5.40
No. 5 ".....	1.37

A second experiment was made with a stronger yeast-water and the following amounts of sugar:—

#### EXPERIMENT II.

	Per Cent.
No. 1.—100 cc. yeast-water, containing 30 dextrose.	
No. 2. " " " " 20 " "	
No. 3. " " " " 10 " "	

Each flask was yeasted with 1.92 cells pure yeast per standard volume. After fermentation, the total number of cells found was:—

	Cells.
No. 1.....	14.10
No. 2.....	14.02
No. 3.....	14.34

From these experiments it appears that the amount of the carbohydrate food here used does not influence the multiplication of the yeast at all when it exceeds 5 per cent.; and further, that below 5 per cent. the multiplication of the cells is not in proportion to the sugar present, but in a much higher one. The result in flask 5, Experiment I., shows that yeast-water alone does not encourage yeast increase at all.

The influence of nitrogenous food upon yeast increase was next examined. In the first experiment, varying strengths of the same preparation of yeast-water were used as the nitrogenous food, and the carbohydrate food was maintained a constant quantity.

#### EXPERIMENT I.

Flask 1 contained 100 cc. yeast-water.
Flask 2 contained 50 cc. yeast-water and 50 cc. distilled water.
Flask 3 contained 25 cc. yeast-water and 75 cc. distilled water.
Flask 4 contained 100 cc. distilled water alone.

The solution in each flask contained 10 per cent. dextrose, and was yeasted with 2.20 cells pure yeast. Fermented

four days at 72° F., and afterwards the total cells were counted, with the following results:—

	Total Cells.
Flask No. 1 .....	14'44
" 2 .....	12'01
" 3 .....	7'97
" 4 .....	3'57

A second experiment was made in which the nitrogenous food was represented by asparagin, which was dissolved in varying amounts in a 10 per cent. solution of dextrose in pure water.

#### EXPERIMENT II.

##### Per Cent.

Flask No. 1 contained 2'0 asparagin and 150 cc. sugar solution.

" 2	"	1'0	"	"	"
" 3	"	0'5	"	"	"
" 4	"	0'25	"	"	"
" 5	"	no asparagin	"	"	"

Each solution was yeasted with 3'05 yeast cells per standard volume. The total number of cells found after fermentation was—

	Total Cells.
Flask No. 1 .....	5'37
" 2 .....	6'13
" 3 .....	7'47
" 4 .....	6'80
" 5 .....	3'00

A third experiment was made in a similar manner to the preceding one, but in which meat peptone replaced the asparagin as nitrogenous food, all the other conditions being the same.

##### Per Cent.

Flask No. 1 contained .....	0'333 peptone.
" 2 .....	0'166 "
" 3 .....	0'083 "

The solution in each flask was yeasted with 3'22 cells per standard volume. After completion of fermentation, the following results were obtained:—

	Total Cells.
Flask No. 1 .....	6'33
" 2 .....	5'96
" 3 .....	5'41

It is evident from the above experiments that an increase in the amount of nitrogenous food does not cause a proportional increase in the number of yeast-cells formed, but points to the conclusion that beyond a certain concentration there is no further increase. Neither asparagin nor meat peptone can be regarded as favourable nitrogenous food for yeast.

An experiment made to ascertain what effect the amount of yeast introduced into a wort had upon the total number of cells found at the conclusion of the fermentation showed that, within reasonable limits, the amount of yeast originally introduced to start a fermentation in malt wort, has little or no influence upon the number of new cells at the conclusion of fermentation, but that the number will be the same in the same volume of wort.

An experiment made to determine if different volumes of the same solution nourished the increase of yeast cells in the same proportion as their bulk showed that the increase of yeast was almost exactly *directly as the volume of the solution*.

The following experiment was made to ascertain, at intervals during a fermentation, the rate of increase of the yeast cells, and also the amount of sugar fermented by them. For this purpose a flask, containing 250 cc. of yeast-water, in which was dissolved 56'35 grms. of dextrose, was sterilised by heat in the usual way and inoculated with 1'39 cells of pure yeast per standard volume. At intervals of 24 hours for the first five days of fermentation, and afterwards at intervals of 48 hours, the flask was well shaken and a sample withdrawn, in which was determined the number of yeast cells, and also the amount of sugar unfermented. The following table gives the results obtained:—

Fermented at 68° F.	Total Cells Found.	Dextrose Fermented.
Started—		
May 30th, 11 a.m....	1'39	None.
May 31st, 11 a.m....	8'46 (3'70)	3'70 grms. per 100 cc.
June 1st, 11 a.m....	15'02 (3'35)	7'05 " "
June 2nd, 11 a.m....	16'79 (3'98)	16'98 " "
June 3rd, 11 a.m. ..	18'06 (4'47)	15'45 " "
June 4th, 11 a.m....	17'92 (1'82)	17'27 " "
June 6th, 11 a.m....	18'45	20'38 " "
June 8th, 11 a.m....	18'04	21'84 " "
June 10th, 11 a.m....	18'72	22'01 " "

In the above table the figures in brackets represent the weight of sugar fermented each 24 hours.

It is very generally supposed that during fermentation the yeast cells split up sugar into alcohol and carbonic acid most rapidly during a late period of their growth, or when their growth has already ceased, and that during the very early stages of growth but very little sugar is fermented; but the results of the experiments given in the above table appear to contradict this idea, for it will be noticed that at the end of the first 24 hours, when 8'46 cells are found in the liquid, 3'70 grms. of sugar had been fermented away. At no other time during the fermentation was anything like this proportion of sugar fermented by a similar number of cells. For instance, at the end of the fourth day of fermentation, we find that 4'47 grms. of sugar have been decomposed in the previous 24 hours; but we find that 18'06 cells have been in the liquid during this time; obviously, therefore, the 8'46 cells present during the first 24 hours have done far more work in decomposing 3'70 grms. of sugar, than 18'06 cells in decomposing 4'47 grms.

The above experiment is open to the objection that the flask was shaken every 24 hours in order to distribute the yeast before taking a sample, and as agitation has a great influence in promoting yeast growth, the experiment was repeated as follows:—

Six flasks were taken similar in size and shape, and to each was added 150 cc. of the same yeast-water solution, containing 18 per cent. dextrose. To each flask was added an equal amount of yeast, amounting to 0'65 yeast-cell per unit volume. All the flasks were then fermented under precisely the same conditions, and one flask withdrawn every 24 hours for examination, leaving the remaining flasks at rest. The results obtained are given in the following table:—

	Total Number of Cells.	Total Grms. Alcohol in 100 cc.	Mean Number of Cells present during each Interval of Time.	Grms. Alcohol in 100 cc. formed during each Interval of Time.	Proportion of Grms. Alcohol per 100 cc. to a Single Cell, in each Interval of Time.	Interval of Experiment.  Hours.
January 9, 11 p.m. ....	0'65	" "	" "	" "	" "	" "
January 10, 11 a.m. ....	4'87	0'654	2'76	0'654	0'237	12
January 10, 11 p.m. ....	12'03	1'933	8'45	1'279	0'1513	12
January 11, 11 a.m. ....	15'38	2'975	13'70	1'042	0'0760	12
January 12, 11 a.m. ....	15'88	4'237	15'63	1'262	0'0807	24
January 13, 11 a.m. ....	15'80	6'187	15'80	1'350	0'1234	24

It will be noticed, on examining the table, that the results found quite bear out the conclusion arrived at from the first experiment, viz., that yeast cells at an early period of their growth ferment sugar much more rapidly than at a later period.

An experiment made upon malt wort and carried on in a similar manner to the above, gave results which quite confirm this conclusion.

The author also quotes an experiment taken from a paper by Hansen, which also confirms this result.

A. J. Brown draws the following conclusions from his work :—

1. Similar volumes of the same malt wort, varying in strength, but exceeding the specific gravity of 1060°, encourage the growth of equal numbers of yeast cells; in the same malt wort, below the specific gravity of 1060°, the increase in number of yeast cells is not in proportion to the specific gravity, but in a much higher proportion.

2. An increase of sugar (dextrose) in a fermenting liquid beyond 5 per cent. does not increase the number of yeast cells formed; below 5 per cent. of dextrose the increase in number of cells is not in proportion to the strength of the sugar solution, but in a much higher proportion.

3. Nitrogenous food, when beyond a certain concentration in a fermenting liquid, appears not to increase the multiplication of yeast cells; below this concentration the multiplication is not in proportion to the nitrogenous food present, but in a much higher proportion.

4. The quality of yeast food determines a greater or less increase of yeast cells.

5. The number of new cells formed in a fermenting liquid is independent of the number of mother cells originally introduced, when these do not exceed certain limits.

6. The total number of yeast cells found in different volumes of the same fermenting liquid is *directly in proportion to the volume*.

7. Fermentation of sugar is most rapid during the earliest stages of yeast increase, and least rapid when the increase has ceased.

8. Fermentation is carried on by yeast cells for a long time after their increase in number has ceased.—G. H. M.

*On the Analysis of a Beer of the last Century, with some Remarks upon the Estimation of Amyloins (Maltodextrins) in Beer.* H. T. Brown and G. H. Morris. Trans. Lab. Club, 3, 81—98.

THE beer in question was brewed at Burton-on-Trent in or about the year 1798.

It is a strong ale, and although still perfectly sound, has to a great extent lost the general character of a beer during the long years it has been in bottle. Its chief characteristic is the extraordinary bouquet it possesses, which strongly resembles that of old Madeira. This bouquet is due to compound ethers which have been developed in considerable amount. When the sediment is examined under the microscope, it is seen to be made up for the most part of finely-divided amorphous matter, but here and there undoubted evidence of yeast cells have been obtained. These are very shrivelled in appearance, but on one or two occasions the authors have suspected the presence of a few yeast cells still retaining vitality, and in these cases the beer was still charged with gas. No bacterial growth has ever been observed.

The following is an analysis of the old beer, and also, for purposes of comparison, that of a strong ale of about the same gravity, but of an age of only 18 months. The results are expressed in percentages on the total solids present before fermentation, deduced from the original gravity by the 3.86 divisor.

	Old Ale. (90 Years Old).	New Strong Ale. (18 Months Old).
Specific gravity at 60° F. .	1030.45	1030.47
Original specific gravity .	1110.38	1103.58
Solid matter per 100 cc. .	28.595 grms.	26.782 grms.
Alcohol by weight . . . . .	8.70 per cent.	7.85 per cent.
Total acid (calculated as acetic acid) . . . . .	0.6605 „	0.3635 „
Volatile acid (calculated as acetic acid) . . . . .	0.1101 „	0.2095 „
Fixed acid (calculated as lactic acid) . . . . .	0.7038 „	0.2400 „
Volatile compound ethers (calculated as acetic acid) . . . . .	0.0598 „	0.0334 „
Percentages on original wort solids:—		
Fermented matter . . . . .	59.98	58.04
Free maltose . . . . .	0.00	0.00
Free dextrin . . . . .	5.52	14.04
Amyloins . . . . .	17.57	17.08
Containing maltose . . . . .	14.58	10.81
„ dextrin . . . . .	2.90	6.27
Albuminoids (N. × 6.5) . . . . .	4.63	10.86
Undetermined . . . . .	12.30	
Total . . . . .	100.00	100.00

In these analyses the authors have expressed a portion of the carbohydrate constituents as *amyloins*, and it is with the determination of these substances that the new method has to do. Before describing the details of the method, the authors furnish the following explanation of the term, and of the position which the substances occupy with regard to the other transformation products of starch.

In a paper read by them before the Chemical Society in 1885 (this Journal, 1885, 682), they described a substance, under the name of *maltodextrin*, which occurs in all starch transformations to a greater or less extent; and in a later paper (this Journal, 1889, 716) they described a substance of a similar nature under the name of *amylogdextrin*. The distinguishing characteristics of these substances are:—

1. That they give numbers on analysis which allow their composition to be expressed in terms of a mixture of maltose and dextrin.

2. That they cannot be separated by any known means into maltose and dextrin, and are, therefore, compound bodies.

3. That they are completely converted into maltose by the action of malt-extract or diastase.

4. That they are unfermentable during the primary fermentation.

A closer study of the products of the transformation of starch has convinced the authors that there are a considerable number of these bodies, and in their latest paper dealing with the subject (this Journal, 1889, 717) they recognise the existence of “maltodextrins, whose number is only limited by the size of the original amylin group,” from which they are derived. It is these substances which Brown and Morris now propose to name *amyloins*, a term suggested by Armstrong, and one which expresses their truly compound nature as distinguished from a mixture of maltose and dextrin.

In considering the position which these bodies hold with regard to the other transformation-products of starch, the authors state that in their last paper they expressed an opinion, based upon their more recent experiments, that the dextrins can no longer be regarded as constituting a polymeric series, but that the starch molecule, in the first moment of

hydrolysis, breaks down into five amylin groups of equal magnitude. One of these amylin groups resists, to a much greater extent than the other four groups, the hydrolysing influence of diastase—this constitutes the stable dextrin which has been so often described as that of the No. 8 equation. The remaining four amylin groups are capable of gradual and complete hydrolysis to maltose, a series of intermediate amyloins of varying degrees of complexity being produced, the general tendency being for the molecular aggregations to become less as the hydrolysis becomes more complete, until the final stage of maltose is reached.

It follows from this that in a starch conversion which has proceeded to any extent—such a one, for instance, as takes place in the mash-tun—the products of the conversion consist therefore of free maltose, of free dextrin, which is not further hydrolysed, and of a series of amyloins of varying constitution.

When a beer which has undergone a normal fermentation reaches the racking stage, it is found on examination that, although it still apparently contains a very considerable amount of maltose, judging from the cupric reducing power, yet it undergoes but little or no further fermentative change until the secondary fermentation commences, or until malt-extract is added. In a word, a normal beer, when racked, contains little or no free maltose, the apparent maltose in the beer existing for the most part as amyloins, which require to be degraded before they become fermentable. On storage, degradation gradually takes place under the influence of the secondary yeast-forms, free maltose is liberated and fermentation starts once more.

From what has been stated above as to the properties of amyloins, it is evident that if the free maltose can be removed from a beer, the unfermentable maltose will represent the amylin portion of the amyloin, and further, that if the beer residue is degraded with diastase under favourable conditions, the increase in maltose so produced will be a measure of the amylin portion of the amyloins.

The details of the method which Brown and Morris propose for the determination of amyloins, and which they employed in the analyses given above, are as follows:—

The original gravity of the beer, the optical activity, and the cupric-reducing power are determined in the usual way (see Heron, this Journal, 1888, 272).

250 cc. of the beer are then taken, the greater portion of the alcohol is distilled off, and the residue is made up to the original volume. The results of the subsequent determinations can then be calculated on the original gravity of the beer.

100 cc. of this residue are set to ferment at 80°–85° F. with a very little pressed and washed yeast. When the fermentation, if any, is complete, the liquid is cooled, made up to the original volume, and the optical activity and cupric-reducing power determined. The difference between these numbers and those obtained for the original beer represents the amount of free maltose originally present in the beer.

A second 100 cc. are degraded at 120°–125° F. for one hour with a sufficient quantity of malt-extract or precipitated diastase, preferably the latter, in order to degrade the amyloins. The degraded residue is then boiled, cooled, and made up to the original volume; the optical activity and reducing power are again determined, and the difference between these numbers (after correcting, if necessary, for malt-extract) and those obtained before degradation, is a measure of the conversion of the amylin or dextrin portion of the amyloins into maltose.

The method of calculating these results will be best seen in the following example, which shows the results obtained by this method with a pale ale undergoing cask fermentation:—

#### Original gravity—

Residue .....	1026.74
Degrees of gravity lost, deduced from spirit indication .....	34.30
Original gravity .....	1061.04

Original wort solids per 100 cc. ....	15.813 grms.
Solid matter fermented .....	8.886 "

#### Analysis of residue—

Opt. act. in 200 mm. tube .....	43.4 divs.
Copper oxide reduced per 100 cc. 2.924 grms. = 1.610 grms. maltose.	

#### Residue after fermentation—

Opt. act. in 200 mm. tube .....	39.2 divs.
Copper oxide reduced per 100 cc. 2.171 grms. = 1.610 grms. maltose.	

#### Residue after degradation—

Opt. act. in 200 mm. tube .....	39.6 divs.
Copper oxide reduced per 100 cc. 3.814 grms. = 2.830 grms. maltose.	

The solid matter fermented divided by the original wort-solids gives the matter fermented, which expressed as a percentage on the original wort-solids is  $\frac{8.886 \times 100}{15.813} = 56.19$  per cent.

The free maltose is found by subtracting the maltose found after fermentation from that in the original beer, thus:— $2.170 - 1.610 = 0.560$  grm. free maltose per 100 cc., expressed as a percentage on the wort-solids this is  $\frac{0.560 \times 100}{15.813} = 3.54$  per cent.

The maltose found after fermentation represents that present in combination as amyloins, the percentage in this case being  $\frac{1.610 \times 100}{15.813} = 10.18$  per cent maltose as amyloins.

The amylin or dextrin portion of the amyloins is found by subtracting the maltose in the original beer from that found after degradation; the small difference due to fixation of water being neglected as falling within the errors of experiment, thus:  $2.830 - 2.170 = 0.660$  grm. dextrin, or as a percentage  $\frac{0.660 \times 100}{15.813} = 4.17$  per cent.

In order to obtain the free dextrin, the deflection due to the maltose found after degradation is calculated and subtracted from the total deflection, the amount of dextrin corresponding to the residual deflection is then calculated in the usual way, thus:  $2.830 \times 7.813 = 22.11$  divs. deflection due to maltose; then  $39.6 - 22.1 = 17.5$  divs. deflection due to dextrin; and  $17.5 \div 11.25 = 1.555$  grm. of dextrin; or, expressed as a percentage,  $\frac{1.555 \times 100}{15.813} = 9.83$  per cent.

The total amount of amyloins is of course obtained by adding together the maltose and dextrin present in this form.

#### The complete results are therefore:—

Fermented matter .....	56.19
Free maltose .....	3.54
Free dextrin .....	9.83
Amyloins:—	
Containing maltose .....	10.18
dextrin .....	4.17
Undetermined matter .....	14.35
	16.09
	100.00

It will be noticed that in the above, the results are calculated from the copper oxide determinations, as far as the original beer and residue after fermentation are concerned, and not from the polariscope readings. The authors consider that in this case the former determinations are capable of being made with greater accuracy than the latter, owing to the dilution required to obtain a good reading, and also to the comparatively small differences in reading which are caused by the conversion of dextrin into maltose. The opticity determinations may be used as a control to the numbers obtained from the copper oxide determinations.

Brown and Morris state that the method may be employed for the analysis of malt-worts, and also for the comparison of different malts mashed under identical conditions; in these cases the amount of free maltose will be large, and will require to be corrected for the ready formed sugars of the malt. It is also advisable to take the residue after complete fermentation of the free maltose for the determination of the amyloins.

In conclusion, they draw attention to the very different types of amyloins which are present in the three beers, the analyses of which are given above. In the old strong ale the mean ratio of  $\frac{M}{D}$  is 4.9:1 (dextrin being taken as unity); in the new strong ale the ratio is 1.7:1; and in the pale ale the ratio is 2.4:1. The authors consider that it is probable the closer study of the ratio of maltose to dextrin in the amyloins in beer will throw much light on the conditions of after-fermentation.

They state that there can be no doubt that the amyloins play a most important rôle in the manufacture of beer, forming, as they do, a store of carbohydrate material, which only slowly becomes available for fermentation as it is gradually hydrolysed. The recognition of their presence also explains much that was formerly obscure in the conditioning of beer; in fact, it may be said, with a tolerable amount of certainty, that if it were not for the existence of such bodies, the long-continued after-fermentation of beer would be an impossibility, and our popular beverage would be something very different from what it now is.

—G. H. M.

*The Influence of Germination upon the Constituents of Barley.* J. O'Sullivan. Trans. Lab. Club, 3 (5), 1890.

THE author divides the constituents of barley and of germinated barley into two groups, those that are soluble in cold water and those that are insoluble in that medium.

In barley the following bodies comprise the first or soluble group:—

- Cane sugar.
- Dextrose.
- Levulose.
- Albuminoids.
- $\beta$ -amylan and other carbohydrates.
- Mineral matter.

To these, in the case of germinated barley, must be added maltose, whilst in the ungerminated grain, raffinose is sometimes found (C. O'Sullivan, Jour. Chem. Soc. 1886, 70).

The second, or insoluble group, in each case consists of—

- Fat.
- Starch.
- Cellular matter.
- Casein (vegetable).
- Cellulose.
- $\alpha$ -amylan.
- Mineral matter.

The chief change involved in the germination of barley is that of diminishing the constituents that are insoluble in cold water, and, at their expense, increasing those that are soluble. This is the direction in which we would naturally expect the change to take place, and not the other way about, for we could not imagine insoluble bodies circulating in the cells, or from cell to cell of the embryo plant. The insoluble bodies in the endosperm, or starchy portion of the grain, which are meant by nature to nourish the young plant, must be made soluble before it can make use of them, or, in other words, they must be brought into circulation by what is in reality a digestion process. The quantity of insoluble substance of barley is considerably diminished by germination; a part of this appears as an increase in the soluble substance of the germinated barley, another appears in the form of rootlets, and the remaining part is consumed during the respiration of the embryo to produce water and carbonic anhydride.

The ratio the carbon oxidised bears to the water liberated points to the oxidation of a glucose; it is not, however, improbable, judging from the bodies that are altered in quantity at an early stage of the germination, that the carbon oxidised is obtained from other sources.

The cane-sugar, or saccharose, is considerably increased during the germination, and as its inversion products, levulose and dextrose, are essential food matter for the young plant, the production of cane-sugar, and its inversion, together with the consumption of the products of its inversion, must be going on at the same time during the

growth of the barley. The aqueous extract of green malt contains no cane-sugar, but the extract of dried malt, if quickly made (two to three hours' extraction), contains cane-sugar; this, however, is not the case if the extraction extends to 10 or 12 hours.

The invertive ferment, or invertase, as already observed by Kjeldahl, occurs chiefly in the rootlets of germinated barley, and from experiments made to determine in what part of the germinating grain, besides the rootlets, this ferment is most abundant, it is found to be exclusively confined to the germ end. The quantity of invertase either in the rootlets or in the germ end is very variable. The following experiments show this.

Digestions were carried on in the cold, with a cane-sugar solution for 24 hours. The rootlets were obtained from samples of the same barley grown at different times, but apparently under like conditions and at the same age of growth.

- 1.7 grms. of moist rootlet (removed from 35 grms. barley) inverted 1.22 grms. cane-sugar.
- 0.5 grm. of moist rootlet (removed from 10 grms. barley) inverted 1.37 grms. cane-sugar.
- 0.5 grm. of moist rootlet (removed from 9.1 grms. barley) inverted 0.15 grm. cane-sugar.

The following results will show pretty forcibly the greater invertive power of the rootlets when in contact with the cane-sugar solution.

The extract obtained from 0.5 grm. of rootlets, by digesting with water in the cold for two hours, when further digested with cane-sugar solution for 24 hours, gave no inversion. Under similar conditions, but in contact with the sugar, 0.5 grm. inverted 1.37 grms. of cane-sugar.

Invertase being more active at about 55° C. than in the cold, experiments were made to determine the invertive power of rootlets at that temperature, and also to learn if, after extraction with cold water, they retained their invertive power when placed in contact with cane-sugar solution: (1) 0.75 grm. of rootlets was digested in the cold for two hours with about 10 cc. of water; then filtered out and washed on filter till filtrate and washings amounted to 50 cc. To 46 cc. of this extract 25 cc. of a 10 per cent. cane-sugar solution were added. (2.) The residual rootlets on the filter were added to another 25 cc. of the sugar solution. (3.) 0.75 grm. of the same rootlets was added to a third 25 cc. of the sugar solution; digested at 55°—57° C. for eight hours, and after standing for eight hours longer in the cold made up to 100 cc. The cane-sugar inverted, and calculated from the cupric oxide reducing power of each 100 cc., is shown as follows:—

Experiment I.—

Extract of 0.75 grm. rootlets inverted 0.19 grm. sugar.

Experiment II.—

Residual rootlets of Experiment I. inverted 1.90 grms. sugar.

Experiment III.—

Original 0.75 grm. rootlets inverted 1.69 grms. sugar.

The cupric oxide reducing power of the extract of the rootlets was allowed for in I. and III. experiments, and was obtained from the 10 cc. kept out of the 50 cc. extract.

The results of a repetition of these experiments, with a different growth of rootlets, but allowing the original rootlets to remain in contact with a little water for the same time as that digested and extracted in the cold, are as follows:—

Experiment I.—

Extract of 0.106 grm. rootlets inverted only a trace cane-sugar.

Experiment II.—

Residual rootlets of Experiment I. inverted 0.45 grm. cane-sugar.

Experiment III.—

Original rootlets 0.106 grm. inverted 0.35 grm. cane-sugar.

The results show that the extracted rootlets retain their inverting power, in fact they come out here more active than the original rootlets; however, much cannot be said about this difference, as it may be due to the difficulty of proper sampling.

It is clear, from these experiments, that nature has arranged that the invertase, which must be of vital impor-



tance to the young plant, should not be easily dissolved by water, and it is more than probable that no invertase is dissolved from the rootlets of the growing grain in any appreciable time.

The following results show that the invertase was exclusively confined to the germ end:—

A number of grains, weighing 9.4 grms., of the same air-dried germinated barley as that from which the rootlets in the last-mentioned experiments were obtained, were cut in two. The germ ends, weighing 4.21 grms., were very finely pulverised, and 1.82 grms. of this was added to 25 cc. of a 2 per cent. cane-sugar solution, the remainder being added to about 25 cc. of water. The acrospires were carefully removed (avoiding any starchy matter) from 80 grains, weighing 3.7 grms., of the same growth of barley. These weighed 0.188 gm. They were placed in a 25 cc. flask, which was then filled with the 2 per cent. sugar solution.

All three were digested for 16 hours at 45°—50° C. In the first two experiments the residue was filtered out, and washed on filter, till filtrate and washings amounted to 50 cc.; in the last experiment the solution was made up to the original volume.

A determination, by the optical and cupric oxide reducing method of analysis, gave the following results for the cane-sugar inverted in the digestions with the germ end and acrospire. The results obtained in both experiments were corrected by those of blank or duplicate experiments with water:—

1.82 grms. germ end (4.0 grms. original grain) inverted 0.2 gm. cane-sugar.

0.118 gm. acrospire (3.7 grms. original grain) inverted 0.21 gm. cane-sugar.

The quantity of dextrose in germinated barley was always found to be in excess of the levulose, but it was not uncommon to meet with some which showed no evidence of the presence of maltose, yet cane-sugar was never absent.

The insoluble nitrogenous matter of the barley was considerably diminished, and found to be an important property imparted either to the nitrogenous matter already soluble in the barley or to that rendered soluble by the germination—namely, high diastatic power.

Rootlets contain a very high amount of nitrogen, as much as 3.5 to 4.0 per cent. having been found in them, which, if calculated as albuminoid, amounts to 21.8 to 25.0 per cent. Asparagin has been isolated from rootlets, and if the nitrogen be taken to be due to this body, we shall find from 16 to 19 per cent. are present in the dry rootlet. The quantity of nitrogen in the rootlets shows the amount that diffuses in a soluble form from the endosperm through the scutellum during germination. About half of the nitrogenous matter of rootlets is soluble in water, and none of this is coagulable. This soluble non-coagulable portion is probably asparagin, and the insoluble portion is most likely vegetable casein.

The quantities of  $\alpha$ - and  $\beta$ -amylase are diminished during germination. So are also the inorganic or mineral constituents of barley soluble in water, and which are chiefly composed of potassium phosphate.

The fat of barley is diminished during germination, and evidently being a suitable body to take part in the early respiration of the embryo, its diminution is observed at an early stage of the germination.

Starch supplies the largest amount of food of any of the constituents to the embryo. It is diminished and it gives rise to the soluble sugar maltose which is produced from the starch by the diastase developed in the seed by germination. Dextrin is not present in germinated barley (C. O'Sullivan, Jour. Soc. Arts, 1876). It is probable that in the solution of the starch dextrin is produced; however, at the period of germination, when the starch begins to be dissolved, there is present in the germinating grain an excess of diastase, which converts the dextrin immediately into maltose.

Besides barley, wheat, rye, maize, and oats were found to possess a diastatic power as well.

In order to determine if this power increases in that part of the endosperm farthest removed from the embryo, the following experiments were made:—

Some sound barley, after being steeped, was allowed to germinate. At the end of the first day 20 grains which

showed signs of germination were taken and cut transversely in two, leaving about two-thirds of the grain with the germ end, which was neglected. From the other one-third, which I shall call the endosperm end, the starchy portion was removed (avoiding that near the husk) and pulverised in a mortar. The pasty mass thus obtained was mixed with about 25 cc. of cold water; this mixture was gradually heated to 65°—68° C., and kept at that temperature for 12 hours. It was observed that although the starch was not dissolved, yet there was an undoubted tendency in this direction, as compared with a similar experiment by its side made with the original barley. This experiment was repeated from day to day during germination, with the following results:—

Days of Germination.	Appearance of Solution.	Result.
First	Inclination to dissolve; no "break" in solution.	Soluble starch present in filtrate of a portion cooled.
Second	Decided solution, but no "break."	Ditto.
Third	Decided solution and "break" or precipitation of flocculent albuminoid and cellular matter.	No soluble starch, but $\alpha$ -dextrin, &c.
Fourth	Complete solution and "break," leaving solution quite bright.	In 20 minutes neither soluble starch nor $\alpha$ -dextrin.

Similar results were obtained for germinating oats.

The time of digestion for the third day's experiment was only three hours. The rootlets, even on the fourth day of growth, were only 1 to 2 mm. in length, and the acrospire had scarcely started.

The production and presence of naked diastase in the endosperm, stimulated by the growing embryo, was considered by the author to be the most reasonable explanation for all the facts relating to the germination of barley.

By the cellular matter mentioned in the insoluble group, it is meant the constituent of the cells, which is pretty easily acted upon by boiling dilute acids this constitutes a portion of the endosperm of barley as may be seen if the endosperm is dissolved in the usual manner by malt extract; the starch goes into solution leaving the insoluble cellular matter as a flocculent structureless body. It is not, however, confined to the endosperm. It is diminished during germination, becoming soluble, and its metamorphosed form supplies the embryo with nutrient matter probably in the form of a sugar.

The casein is diminished during germination. This body is not dissolved by malt-extract, it is soluble in dilute alkalies, and is precipitated by acids from its alkaline solutions; it has, in fact, the properties of the casein of milk.

The cellulose proper of barley is chiefly located in the husk or palea; it is the most insoluble of the organic constituents. It is not altered in quantity during the period of germination, and probably is not touched even when the plant has developed to the stage when the whole of the contents of the endosperm are exhausted, and it is capable of assimilating for itself.

The results arrived at by the author are (1) that the diastase ferment originates in the endosperm, and (2) that the ferment capable of inverting cane-sugar is situated, where it is wanted, in the acrospire and rootlets.

The author is of opinion that the soluble metamorphosed contents of the endosperm, being produced in the cells of the endosperm of the growing barley-corn, are repelled, and diffuse through the scutellum towards the point of least concentration, which must be in the vicinity of the embryo or point of their consumption.

*Denaturation of Spirit.* G. Lunge. Zeits. f. angew. Chem. 1890, 71—72.

It has been stated in an article by A. W. von Hofmann, Krämer, and Löwenherz (Chem. Ind. 1889, 521) that the Swiss are using a "pyridine mixture" for denaturising spirit. As by this is understood a mixture of wood spirit and pyridine bases, the author takes the opportunity to assert that wood spirit is not used. Work on the subject is in progress.—T. L. B.

*The Denaturation of Spirit.* A. W. von Hofmann, G. Krämer, and L. Loewenherz. Chem. Ind. 13, 119—120.

A REPLY to the statement of Lunge (preceding abstract) that the Swiss Commission have rejected the German denaturation mixture. It is stated that the Swiss Administration up to the end of 1889 purchased large quantities of pyridine bases.—T. L. B.

*Recent Methods of Spirit Purification.* Chem. Ind. 13, 106—108.

THE older methods were mainly useless as being unscientific and unreliable. More recently has been brought out, however, the capillarimetric and stalagmometric method of Tranbe (see this Journal 1889, 42).

The method of the "*Société Française des Alcools purs*" (this Journal, 1889, 57) has reference rather to the working up of first runnings. The first runnings obtained in the rectification of spirit contain chiefly aldehyde and acetone. They are treated in vats with a solution of an alkali bisulphite of 32° B., in such quantity that, after conversion of the whole of the aldehyde and acetone into their sulphites, a slight excess of free bisulphite is present. When butyl aldehyde is proved to be present, a little neutral sulphite is used, as forming a more stable compound than the bisulphite. The whole mass is then distilled without any separation being previously performed. The alcohol so obtained contains sulphurous acid, and is therefore distilled again in presence of a base. Thus from a cheap source aldehyde and acetone may be obtained, as well as a tolerably pure alcohol.

A process which has been much used is that of Bang and Ruffin (this Journal 1885, 506; 1887, 147; and 1888, 224). It depends on the taking up of the impurities from the diluted alcohol by petroleum ether.

Schmitt diminished the capability of alcohol to dissolve fusel oil by addition of potassium carbonate. Potassium carbonate solution was added as long as no "layering" was visible, and then the whole was treated with petroleum ether. In his experiments Schmitt obtained the following results:—

Strength of Alcohol in Vols. per Cent.	Fusel Oil in Vols. per Cent.	Volume of Spirit for Treatment.	Volume of Petroleum Ether used.	Potassium Carbonate Solution. (Sp. gr. 1.3.)	Fusel Oil in Vols. per Cent. after Treatment.	Decrease of Fusel Oil, in Vols. per Cent.
50	0.5	cc. 300	cc. 150	cc. 24	0.29	0.21
30	0.3	300	150	110	0.17	0.13
50	0.2	300	150	110	0.11	0.09
30	0.2	300	300	110	0.05	0.15
30	0.2	300	600	110	0.00	0.20

After removal of the petroleum ether more potassium carbonate is added to bring about "layering," and the upper layer of alcohol is then syphoned off, neutralised, and distilled.

Before Schmitt, however, Traube made use of potashes (this Journal, 1888, 449, 513, and 762; 1889, 559 and 995), and very good results have been obtained. Tranbe mixes, at a given temperature, the spirit with a potassium carbonate solution, whose specific gravity varies according to the strength of the spirit; for 80 per cent. spirit he uses a potassium carbonate solution of sp. gr. 1.24 at 70° C. By using 4—5 parts of the solution to one part of spirit a layer separates, which contains three-quarters of the fusel oil. By replacing the spirit and alkali taken up by the first layer, by pure spirit and potassium carbonate, another similar layer is obtained, and the quantity of fusel oil is still further reduced. The first layer obtained in each treatment contains most fusel oil, and consequently the first layers of the various batches are kept by themselves, and finally worked up like the raw spirit itself. The spirit, purified by this "layering" process, is finally distilled from the potassium carbonate.—T. L. B.

constituents, next in importance came isobutyl alcohol, and after that isopropyl and ethyl alcohols and traces of normal propyl and butyl alcohols.—A. L. S.

## PATENTS.

*A Method of and Appliances for the Collection and Utilization of the Carbonic Acid and other Products given off during the Process of all Fermentations.* C. R. C. Tichbourne, A. E. Darley, and M. F. Purcell, Dublin. Eng. Pat. 3707, March 2, 1889. *sd.*

THE fermenting vessel is hermetically closed and the gas from the atmosphere above the fermenting liquid is collected by a pipe with a funnel-shaped opening; the opening is capable of adjustment so as to collect from any height above the surface of the liquid; any other suitable contrivance may be used. The gas contains, besides carbonic acid, vapours of alcohol and ethers; the amount of these is much larger in distilleries than in breweries, as the fermentation in the former is carried out at a higher temperature; it is led through a scrubber fed with a spray of water, or in special cases, sulphuric acid, and the purified carbonic acid condensed by suitable pumps. When, besides carbonic acid, other volatile bodies are present in the purified gas they may be separated by successive partial condensations, and the products obtained at various pressures collected separately. The alcohols and ethers recovered in any of the above methods may be either returned to the fermented liquid or utilised in any other way. A sheet of drawings accompanies the specification.

—A. L. S.

*An Examination of Fusel Oil.* J. H. Long and C. E. Linebarger. J. Analytical Chem. 4, January 1890. (Reprint.)

THE fusel oil was from American distilleries who chiefly use corn and other raw grain. It had a specific gravity of 0.810 at 20° C. It was dehydrated and separated by fractional distillation as follows:—1 litre gave 17 cc., 80°—90°; 8 cc., 90—95°; 15 cc., 95°—105°; 30 cc., 105°—115°; 55 cc., 115°—120°; 95 cc., 120°—125°; 240 cc., 125°—130°; 515 cc., 130°—135°; 25 cc. loss and residue.

A careful examination of these fractions showed that active and inactive amyl alcohol were the most important

*Improvements in and Apparatus for the Purification of Alcohol or Aleoholic Liquors.* E. Luck, London. Eng. Pat. 9181, June 3, 1889. 8d.

THE alcohol vapour as it rises from the distillation of the mash is purified by being drawn by pumps through one or more vessels containing liquid hydrocarbon. These vessels may be conveniently of the shape commonly used for washing gases, and furnished with a jacket, which may be supplied with steam or water, so that the hydrocarbon may be heated or cooled to any convenient temperature. The washed vapours are then cooled by a suitable condenser. A sheet of drawings accompanies the specification.—A. L. S.

*Improvements in the Manufacture of Yeast.* J. Bruce, Glasgow. Eng. Pat. 15,772, October 8, 1889. 4d.

THIS invention principally relates to the manufacture of yeast for baking purposes. A solution of albumen is prepared by extracting raw grain with cold or slightly warm water; this solution is then peptonised, either after filtration from the raw grain or without doing so, by allowing to sour in the presence of a very little malt, or mineral acid and malt. The solution or mixture is kept at 100°–120° F. for about 15 hours, and then the heat brought up to 175° F. This solution is then added to a distillers' wort prepared in the usual way; when the raw grain has not been separated before peptonising it forms part of the mash; otherwise it is used in making a prior mash. If, after saccharification, the acidity is less than 0.25 per cent. lactic acid, a small quantity of spent wash is added. The liquid is fermented by adding to it a mash of barley and rye, which has been allowed to sour and is in active fermentation, the amount being about 5 per cent. of the principal wort. The yeast is skimmed off as it rises to the surface, and the alcohol distilled from the fermented mash in the usual way.

—A. L. S.

separated from the milk by means of one of the well-known forms of centrifugal separators; thus delivered it passes over a cooling apparatus, where it is reduced to a suitable temperature and conducted by a pipe into the churning apparatus. This consists of a jacketed cylinder, kept at a suitable temperature by the circulation of a refrigerating fluid; the internal periphery is provided with straight or curved ledges. A high-speed beater revolves in the axis of the churn, and the butter globules are thus separated from the milk and delivered into a tube, which forms a prolongation of the churning cylinder, and here by centrifugal force the lumps of butter are collected into a mass; the mixture of butter lumps and milk are delivered from the end of a receiving chamber.—C. C. H.

*Improvements in Preserving Fish, Birds, Poultry, or other Analogous Articles of Food.* W. Douglas, Glasgow, and J. Donald, Kilwinning. Eng. Pat. 20,614, December 23, 1889. 4d.

FISH, poultry, or other analogous articles of food are enveloped in a suitable covering placed in an ice-making machine, and frozen into a block of ice. So prepared they can be preserved for an indefinite period.—C. C. H.

*Improvements in the Method of and Apparatus for the Production and Preservation of Sterilised Milk.* A. Schmidt-Mulheim, Wiesbaden, Germany. Eng. Pat. 1326, January 25, 1890. 6d.

THE patentee describes four forms of vessels or flasks suitable for the sterilisation of milk; these are shown in the accompanying figures. The milk is heated by means of steam jets and kept boiling for about 15 minutes after it begins to bubble. Fig. 1 shows a flask fitted with a

## XVIII.—CHEMISTRY OF FOODS, SANITARY CHEMISTRY, AND DISINFECTANTS.

### (A.)—CHEMISTRY OF FOOD.

#### PATENTS.

*Improvements in Apparatus for the Manufacture of Bread, specially relating to Bakers' Ovens and Sponge-Blenders.* J. Adair, Waterford. Eng. Pat. 18,771, November 23, 1889. 8d.

HEAT collecting chambers are constructed underneath the ordinary flues of an outside heating oven, and "metals" may be placed in any of the chambers in which heat is specially required.

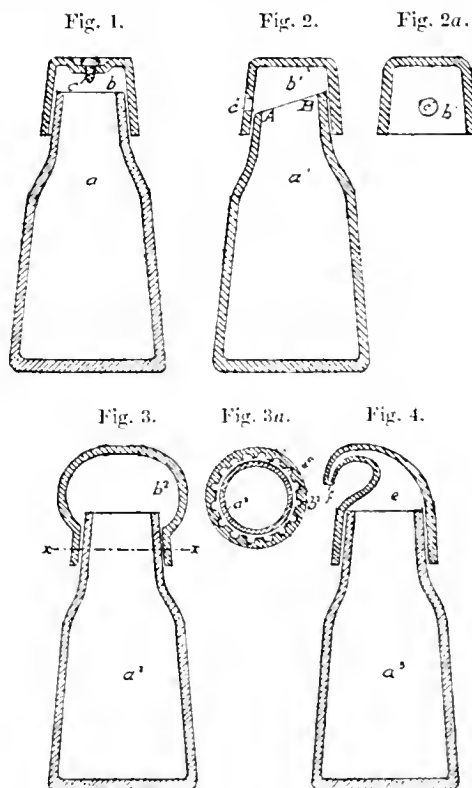
To blend the sponge a moveable sponge-blender made of strained wires, rods or hoops, may be thrown into the dough machine. Drawings accompany the specification.—E. E. B.

*An Improved Bread.* H. Fairlie, Newton Stewart. Eng. Pat. 19,042, November 27, 1889. 4d.

THE ingredients employed are:—Minnesota straight flour, broad-leaved English bran, "baking soda," small bread dough, loaf sugar, American hard, extract of malt and salt. These are mixed in specified proportions. The dough thus obtained is allowed to ferment for at least two hours before it is baked.—E. E. B.

*Improvements in Apparatus for Producing Butter.* H. H. Leigh, London. From C. G. P. de Laval, Stockholm, Sweden. Eng. Pat. 20,232, December 16, 1889. 6d.

THE object of this specification is the continuous production of butter from new milk, so as to combine the processes of skimming and churning. The cream is



ground cap having a cotton plug *c* tightly pressed in after boiling. Fig. 2 shows a flask with the neck cut off slanting, the ground plug *b* is provided with a hole *c*, so that communication with the air can be cut off by turning the cap round. In Fig. 3 the cap *b* is provided as shown

with narrow flutings small enough in section to retain the water driven off by ebullition round the neck of the flask, and so form a seal. In Fig. 4 the ground cap is drawn out to a very fine orifice, and though not tightly sealed to the air it has been found that after sterilisation in such a flask milk will remain quite good for several days even in summer.—C. C. H.

*Improved Sterilising Apparatus.* J. F. H. Gronwald, and E. H. C. Oehlmann, Berlin, Germany. Eng. Pat. 1910, February 5, 1890. 8d.

THIS invention relates to apparatus in which the fluid is sterilised in the vessel used for storage, and which can be closed without admitting air, whilst it also allows the vessel to be filled to any desired height without any risk of the fluid being deteriorated, or of the bottle being burst through any increase of temperature. To effect this an air-tight expansion chamber is detachably connected with the storage vessel in such a manner that the air in one, and the fluid in the other are sterilised at the same time, the liquid during the process partly entering the chamber and afterwards flowing back on cooling. Arrangements are provided for closing the vessel without admitting external air. The space above the liquid being filled with sterilised air acts as a cushion, and prevents any damage to the vessel from the expansion of the liquid on an increase of temperature. (See also this Journal, 1890, 405.)—E. S.

*Improvements in the Manufacture of Whole-meal Bread, Biscuits, and other like Articles of Food.* H. W. Hart, London. Eng. Pat. 2658, February 14, 1889. 6d.

THE wheat is washed, then crushed (*not* ground), soaked in water for about two hours and then baked.—E. E. B.

*Improvements in Processes and Apparatus for the Preparation of an Extract of Coffee, and in the Utilisation of the By-Products.* G. Sonstadt, Cheshunt. Eng. Pat. 3172, February 22, 1889. 8d.

THE apparatus described herein consists of a boiler, and a digester or extractor, fitted with suitable condensing column connected with vessels packed with wool for the absorption of the more volatile products. The boiler is connected with the digester by means of a pipe reaching to the bottom of the latter vessel and provided at the end with a rose or strainer. The ground coffee, sometimes mixed with chicory, is placed on a perforated plate near the bottom of the condenser, and boiling water run in from a subsidiary boiler. The principal boiler is then started, charged with sugar and water, the steam passing into the digester; the volatile products are arrested in the condensing column whilst the aromatic compounds are arrested in the vessels packed with wool. The boiler is then allowed to cool, consequently the decoction contained in the digester passes back thereto. The operation may be repeated as often as is requisite. The decoction is then evaporated to dryness after being mixed with sugar, and the aromatic bodies arrested in the condensing column and by the wool. It is requisite that the evaporator as well as the digester should be provided with an agitating apparatus which can be used for breaking up the material operated on.—C. C. H.

*Improvements in the Manufacture of Aerated Bread and in Apparatus to be used in the Manufacture.* J. Childs, London. Eng. Pat. 3607, February 28, 1889. 8d.

SEPARATED milk is aerated and used for making the dough. The mixing apparatus is constructed so that the aerated liquid can be added to it first and the flour afterwards.—E. E. B.

*Improvements in the Manufacture of Aerated Bread.* J. Childs, London. Eng. Pat. 5728, April 3, 1889. 4d.

THE aerated liquid for making the dough is obtained by adding some ferment to a mixture of flour and water, and keeping the mixture at 80° F. for 10 to 12 hours, or by adding to water either hydrochloric acid and bicarbonate of soda or tartaric acid and bicarbonate of soda.—E. E. B.

## (B.)—SANITARY CHEMISTRY.

*Phosphorous Oxide.* T. E. Thorpe and A. E. Sutton. Proc. Chem. Soc. (82), 1890, 61—64.

PHOSPHOROUS oxide has a well marked physiological effect, and it is not improbable that the action hitherto attributed to phosphorus, especially as regards its influence on the glycogenic functions of the liver and on tissue change, may be really due to this substance. It is well known that persons employed in the manufacture of lucifer-matches are occasionally attacked by caries of the lower jaw; this is not due to the action of the phosphorus after absorption into the circulation, but to the direct effect of the fumes upon the bone itself; for it has been found that when a bone of an animal fed by phosphorus was exposed, no carious change took place; but if one were exposed to the fumes caries was produced, and amongst lucifer-match makers it has been noticed that only those who have carious teeth suffer from necrosis of the jaw (T. Lauder Brunton, *Pharmacology*, &c., 771). The fumes from phosphorus consist largely of phosphorous oxide; by drawing air over phosphorus without allowing it to ignite and passing the fumes through a narrow strongly cooled tube a deposit is obtained which melts with the warmth of the hand and gives the reactions for phosphorous oxide. Moreover, the smell of the product is identical with that of pure phosphorous oxide, and it is also identical with the peculiar smell noticed in a lucifer-match manufactory during the making and handling of the "composition" with which the splints are "tipped," and which hangs about the benches where the "boxers" are at work. It is highly probable, as Schönbein long ago surmised, that phosphorus vapour as such is odourless, and that the smell which phosphorus ordinarily possesses is a mixture of that of ozone and of phosphorous oxide.

*Sewage Decomposition in Streams.* J. H. Long. J. Analytical Chem. 4, January 1890. (Reprint.)

THE author has examined the rate and manner in which sewage is oxidised in a highly contaminated stream (the Illinois) flowing for over 30 miles without dilution, and then for a further total of 160 miles, making allowance for the amount and character of the dilution by tributaries. He finds that the oxidation of the organic matter, as determined by estimations of free and albuminoid ammonia, oxygen consumed, nitrates and chlorides, is very much more rapid in summer than winter, and that in a dry hot summer it is quickest. Nitrates and nitrites did not appear until after a flow of many miles.—A. L. S.

## PATENT.

*An Improved Process for Deodorising, Disinfecting, Precipitating, Drying, and Pulverising the Solid Portions of Sewage.* W. Astrop, London. Eng. Pat. 6219, April 11, 1889. 4d.

THIS specification described improvements on Eng. Pats. 11,901 of 1884 and 10,047 of 1886 (this Journal, 1885, 463), and their object is, after extracting the greater part of the water from sewage sludge or excreta, to render the manipulation of the remaining solid matter easier. Larger vacuum boxes than formerly specified are employed, and the solid matter yielded by the operation is passed between rollers, reduced to a cake-like form, and in its passage through the rollers mixed with sifted dry material, e.g., cinders, road sweepings, bone dust, &c.—C. C. H.

## (C)—DISINFECTANTS.

## PATENT.

*Improvements in Disinfectants, their Manufacture, and their use for General Purposes, for Treating Sewage, and for Protecting Vines against Mildew and Phylloxera.* F. W. A. Hille, London. Eng. Pat. 6411, April 15, 1889. 6d.

MAGNESIUM or calcium chloride is dissolved in boiling water, and to this solution is added and thoroughly incorporated therewith a solution of camphor made by dissolving 1 oz. of camphor in 2 oz. of alcohol. The proportions recommended are 5 lb. of the metallic salt to half a gallon of boiling water, to which is finally added  $\frac{1}{2}$  oz. of the camphor solution. Thorough incorporation is effected by agitation. When used for disinfecting drains or sewers one part of the solution is diluted with four parts of water; when used for dressing vines for the purpose set forth it is diluted with from five to ten times its volume of water.—C. C. H.

## XIX.—PAPER, PASTEBOARD, Etc.

*Sulphite Pulp in America.* Chemical Trade Journal, 1890, 303—304.

THE following abstract is from a letter on the above subject by Mr. Waldemar Thilmann, Kaukauna, Wisconsin, U.S.A., on March 13th, to the *Papier Zeitung*, No. 35, 1st May, 1890.

At the Thilmann Pulp and Paper Works, the Mitscherlich sulphite process is at work. The first mill using this process was started at Alpena, Michigan, in November 1886, with a daily output of 20 tons of 2,000 lb. per ton.

It interested him much to find out how the different kinds of wood would behave in the sulphite process, and he therefore allowed a boiler full of each of the following different woods to be digested, viz.:—Beech (*Fagus Syleatica* L), birch (*Betula Alba*), maple (*Acer Pseudoplatanus* and *Acer Platanoides* L), American larch (*Pinus Larix* L), aspen (*Populus tremula* L), black poplar (*Populus Nigra* L), red fir (*Pinus Picea*), spruce or white pine (*Pinus Abies*), yellow pine (*Pinus Sylvestris*).

The wood was freshly felled, and had a diameter varying from 5 to 8 in. It was cut into discs of  $1\frac{1}{4}$  in. thick, and then digested whole with a solution of  $5\frac{1}{2}$  B. by the Mitscherlich methods. All of these woods with the exception of the *Pinus Sylvestris* (which remained hard in the centre), were well-boiled and completely soft.

Beech, birch, and maple yielded snow-white sulphite pulp, whose fibre, however, was considerably shorter than that from the red or white pine (*Pinus Picea* and *Pinus Abies*), and consequently they did not yield so strong a paper.

Afterwards, some boilers full of larchwood ("Tanræck") were digested, and the pulp run off on a cylinder machine in the same way as it is sent to the paper mills. The fibre appeared good and strong, but the colour was mottled, i.e., it varied from dark brown to citron yellow.

At present the seven mills in America, working the Mitscherlich system, produce daily about 70 tons pulp, and use exclusively spruce or white pine wood (*Pinus Abies*).

Two larger mills, situated in the Fox River Valley, with eight digesters, each 14 ft. diameter and 42 ft. long, produce daily 20 tons of pulp; whilst another mill at Madison, Maine, has 20 digesters of the above size, equal to a daily output of 20 tons pulp.

Sulphite pulp prepared by the Mitscherlich process is clean if the wood be properly sorted. There is apparently a difference in the strength of the fibre obtained from American spruce by the sulphite method as compared with imported sulphite pulps, even those made by the Mitscherlich system in Europe. Tests of raw American-made pulp and newspaper made from it, in a paper mill belonging to

Mr. Thilmann's brother, revealed the fact that from 10 to 15 per cent. more imported pulp had to be used to obtain the same results as regards strength.

The pulps imported to America are frequently mixed, so that the paper-maker is not always provided with the same make. How this occurs the importer only knows. This he keeps secret, and makes many untrue assertions regarding it. The paper-maker who has hitherto made good newspaper from 20 per cent. sulphite and 80 per cent. mechanical wood pulp, suddenly finds that his paper is much weaker, and consequently many complaints pour in upon him. He then investigates his pulps, and finds among them the most diverse qualities.

All sulphite pulp made in America and manufactured into newspaper is used for this purpose in the unbleached state.

## PATENTS.

*Transparent Printed Paper having the Appearance of Stained Glass and Means or Apparatus for its Manufacture.* W. L. Wise, London. Eng. Pat. 5418, March 29, 1889. 8d.

THE inventor claims "the manufacture in the ways and by the means described of transparent or translucent printed paper having the appearance of stained glass, using blocks and cylinders of wood or metal such as zinc, either bare or faced with caoutchouc or gutta-percha or other soft and elastic substance, and covered or not covered with a coating of gelatin." After printing, the paper is coated on both sides with varnish; and when this is dry, with another coating of gelatin or other adhesive substance.—E. J. B.

*Improvements in or relating to the Prevention of the Corrosion of Vessels, Tubes, Valves, and the like used in the Manufacture of Sulphite Cellulose.* A. J. Boulton, London. From C. H. Jung and J. P. Lindig, Freiberg, Germany. Eng. Pat. 5643, April 2, 1889, 4d.

THE vessels, thoroughly cleaned by boiling in alkali and subsequent treatment with dilute acids, are coated with a solution of acid calcium sulphite. When dry the surface is covered with a solution of silicate of soda. On to this coating a layer of a pulpy mixture of calcium sulphite and silicate of soda or potash is laid to a depth of from 1—5 centimetres. When this coating is dry and hard, water containing acid calcium sulphite is boiled in the vessel.

In case cracks should form in the coating, the boiler is connected with a dynamo and forms the cathode of an electrical circuit, while a lead plate inside the boiler forms the anode. If a crack occurs the exposed part is coated with an electrolytically formed deposit.—E. J. B.

*Improvements in the Manufacture of Artificial Ivory or Ivory Substitutes.* A. and S. de Pont, Paris, France, and H. L. and J. H. Storey, Lancaster. Eng. Pat. 9267, June 4, 1889. 6d.

THE improved artificial ivory is obtained by kneading together lime, phosphoric acid, calcium carbonate, magnesia, alumina, gelatin, and albumen, in such proportions that the resulting mixture has the same chemical composition as natural ivory. The material is subjected while hot to great pressure, and after maturing for some weeks it can be cut, turned, and polished in the ordinary way.—E. J. B.

*Improvements in Extracting Substances Dissolved in the Waste Water of Paper Manufacture.* F. C. Altkier, Wieselburg, Austria. Eng. Pat. 1514, January 28, 1890. 4d.

IN the manufacture of paper from steamed ground wood pulp, the waste water contains methyl alcohol and acetic

acid. In this process these products are recovered. Instead of using fresh water, water from previous operations is used; in this way it gradually becomes sufficiently concentrated to render the recovery profitable. To neutralise the acetic acid, lime or soda is added. The liquid is distilled to drive off the alcohol and the residue is evaporated to dryness and distilled with hydrochloric acid.—E. J. B.

*Improvements in and relating to the Construction of Digesters or Boilers for use in the Manufacture of Paper Pulp.* W. W. Keys, Bridgeport, U.S.A. Eng. Pat. 1517, January 28, 1890. *8d.*

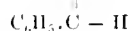
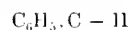
THE boilers are cast in sections in a material known as de-oxidised bronze. It is obtained in the following manner. The surface of melted copper is covered for an hour to the depth of an inch or two with small pieces of animal horn. Tin is then added in the proportion of one to nine of copper. This bronze completely resists the action of the acid sulphite solutions used for preparing wood pulp.—E. J. B.

## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

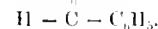
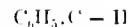
*Iso-Cinnamic Acid, an Acid found in the Alkaloids accompanying Cocaine.* C. Liebermann. Ber. **23**, 141—156 and 512—516.

THE mixture of acids obtained by treating the alkaloids accompanying cocaine with hydrochloric acid according to the author's process (D. P. 47,602) for preparing ecgonine therefrom, contains chiefly  $\alpha$  and  $\beta$  truxillic and cinnamic acids. The residue after the separation of these acids, leaves a thick oily mass behind after extraction with ether and distilling off the latter, which does not solidify in the cold. When extracted with petroleum spirit (b.pt.  $60^{\circ}$ — $70^{\circ}$ ) a small portion of the residue dissolves and the solution deposits crystals of the new acid on standing. Special precautions were taken to obtain this acid perfectly free from ordinary cinnamic acid, advantage being taken of the greater solubility the free iso-acid in petroleum ether and of its calcium salt in water. Iso-cinnamic acid melts at  $57^{\circ}$  (ordinary cinnamic acid at  $133^{\circ}$ ), and forms characteristic monoclinic crystals quite distinct from those of either cinnamic, hydrocinnamic or atropic acid; further the non-identity of the new acid with any of the above is fully borne out by its physical and chemical characters. Analyses and a molecular weight determination by Raoult's method point to the formula  $C_9H_8O_2$ . On oxidation with potassium permanganate in alkaline solution benzaldehyde is formed. The calcium, barium, cobalt, manganese and zinc salts of the acid are soluble, the copper and mercury salts insoluble in water; the silver salt is unacted on by light, and when treated with methyl iodide yields the methylester of isocinnamic acid. Isocinnamic acid begins to boil at  $265^{\circ}$ , and is converted quantitatively into ordinary cinnamic acid, but on continued heating carbon dioxide is evolved and styrene  $C_8H_8$  formed. This conversion into cinnamic acid is also effected by treatment with sulphuric acid or with iodine in carbon bisulphide solution. With many reagents derivatives of ordinary cinnamic acid result, thus, nascent hydrogen gives ordinary hydrocinnamic acid; bromine, —dibromcinnamic acid, hydrobromic acid,—phenyl- $\beta$ -bromopropionic acid and hydrochloric acid,—phenyl- $\beta$ -chloropropionic acid. The author also found a small quantity of iso-cinnamic acid in ordinary cinnamic acid prepared from storax, but failed to detect it in cinnamic acid from benzaldehyde.

The following constitutional formula is assigned to the acid—



Ordinary cinnamic acid being—



The less stable nature of the iso-acid as compared with the normal acid agrees with the formula; this is further confirmed by its conductivity when dissolved, for electricity, which is about five times as great as that of the ordinary acid. —C. A. K.

*On a Compound of Calcium Chloride and Normal Propyl Alcohol.* C. Göttig. Ber. **23**, 181—182.

THIS compound was first obtained as a by-product when drying commercial propyl alcohol by calcium chloride.

When a portion of the propyl alcohol had been distilled off from the calcium chloride, the residue on standing deposited two bodies, the bottom one was opaque and consisted of calcium chloride, containing variable quantities of alcohol and water, the upper one was transparent, and consisted of needle-shaped crystals; it was dried between blotting paper, and on analysis gave numbers corresponding with the formula  $CaCl_2 \cdot 3 C_3H_7O$ . To determine whether this was an accidental mixture, the substance was recrystallised from pure propyl alcohol and gave the same analytical number.

The body forms short transparent needle-shaped crystals, of a slightly greater density than water. When small pieces are thrown into water they move about on the surface whilst dissolving, and are decomposed into propyl alcohol and calcium chloride; larger pieces sink to the bottom and dissolve more slowly. On exposure to the air it absorbs water, and in a desiccator, over sulphuric acid, loses alcohol.—A. L. S.

*Hydrastine.* M. Freund and A. Rosenberg. Ber. **23**, 404—415. (Compare this Journal, 1889, 1006.)

THE near relationship which exists between narcotine and hydrastine would lead to the expectation that the halogen-alkyl addition products of the two alkaloids would also show a similar behaviour. Experiments instituted some time ago with the object of preparing a compound from hydrastine, analogous to the pseudonarcine  $C_{21}H_{23}NO_6 + 3H_2O$  obtained by Roser (Annalen **247**, 167) from narcotinemethyliodide, gave such abnormal results that a more complete investigation has now been carried out.

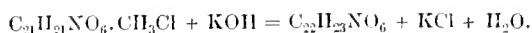
*Hydrastinemethyliodide*,  $C_{21}H_{21}NO_6 \cdot CH_3I$ , melting at  $208^{\circ}$  is the sole product of the action of methyl iodide on hydrastine. When digested with silver chloride and water it is converted into the corresponding *methylechloride*.

*Hydrastinemethylhydroxide*,  $C_{21}H_{21}NO_6 \cdot CH_3OH + H_2O$ , is formed, together with *methylehydrastine* when this methylechloride is treated with potash in the cold. The reaction which takes place may be represented by the equations—

I.



II.



The crystals of methylhydrastine are separated by filtration, and the alkaline filtrate evaporated, whereon the hydroxide is obtained in colourless prisms. It melts at  $242^{\circ}$  and is readily soluble in hot water and dilute alcohol; it is identical with the compound (m.p.  $236^{\circ}$ — $238^{\circ}$ ) previously described (Ber. **19**, 2799), obtained by treating the methyl iodide with moist silver oxide.

*Methylhydrastine*,  $C_{22}H_{23}NO_6$  (see above), is the principal product of the action of potash on hydrastinemethylchloride. It crystallises in yellow needles, melts at  $156^\circ$  and is almost insoluble in water but readily soluble in chloroform, benzene, carbon bisulphide, ether and dilute alcohol. It dissolves in Fröhde's reagent, yielding a violet solution which quickly turns blue, and then slowly becomes green. The *hydrochloride*  $C_{22}H_{23}NO_6 \cdot HCl$  melts at about  $241^\circ$  with decomposition, and is moderately easily soluble in hot water. The *platinochloride*, *nitrate* and *sulphate* are more sparingly soluble.

*Methylhydrastinemethyliodide*,  $C_{22}H_{23}NO_6 \cdot CH_3I$ , is formed when methylhydrastine is treated with methyl iodide in cold alcoholic solution. It crystallises in yellowish needles, decomposes at  $250^\circ$ , and is more readily soluble in hot water than in alcohol; it is readily decomposed by alkalis with liberation of trimethylamine and formation of a compound of the composition  $C_{20}H_{20}O_8$ . This reaction may be represented by the equation:  $-C_{22}H_{23}NO_6 \cdot CH_3I + KOH + H_2O = KI + N(CH_3)_3 + (C_{20}H_{20}O_8)_x$ .

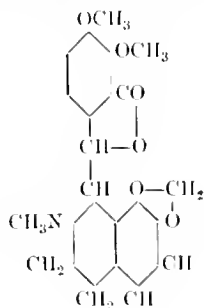
*Methylhydrastine*,  $C_{22}H_{27}NO_4 + H_2O$ , is obtained by warming hydrastinemethylchloride with excess of potash; on neutralising the concentrated solution with acetic acid the product separates in crystals. It is more conveniently prepared by boiling methylhydrastine with concentrated potash until an oil begins to separate, then evaporating, dissolving the oil in water and neutralising with acetic acid. The new compound then separates in crystals and can be purified by recrystallisation from water. It melts at  $150-151^\circ$ , loses its water at  $100^\circ$ , and is readily soluble in hot water and alcohol; it dissolves in mineral acids and in alkalis, but it is insoluble in sodium carbonate and dilute acetic acid. It gives the same colouration with Fröhde's reagent as methylhydrastine, and it is coloured red by ammonia and chlorine water, but it is not acted on by iodine. It is optically inactive and resembles pseudonarcotine in many respects. It is a tertiary base, combines with methyl iodide in the cold, and is reconverted into methylhydrastine by hot concentrated hydrobromic acid. The salts are colourless. The *hydrochloride*  $C_{22}H_{27}NO_4 \cdot HCl + H_2O$  melts at  $290^\circ$  and is soluble in water.

*Hydrastine-ethylhydrastine*  $C_{21}H_{21}NO_6 \cdot C_2H_5OH + 2\frac{1}{2}H_2O$  and *ethylhydrastine* were prepared from hydrastine ethyl-iodide exactly as described in the case of the corresponding methyl compounds. The hydroxide crystallises from dilute alcohol, melts at  $225^\circ$ , and is identical with the compound obtained by Wilhelm (Arch. Pharm. 1888, 341).

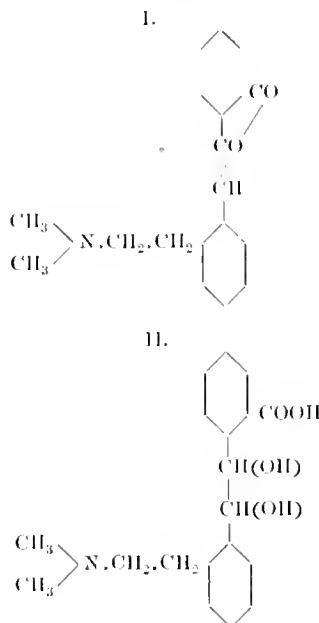
*Ethylhydrastine*  $C_{20}H_{25}NO_6$  melts at  $126-127^\circ$ , and forms yellow, crystalline salts with hydrochloric and with nitric acid; it combines with ethyl iodide yielding *ethylhydrastine ethylchloride*  $C_{22}H_{27}NO_6 \cdot C_2H_5I$ . This compound melts at  $241^\circ$ , and is decomposed by alkalis in the same way as the corresponding methyl derivative.

*Ethylhydrastine*,  $C_{22}H_{29}NO_3 + 2H_2O$ , obtained from ethylhydrastine as described in the case of the methyl derivative, crystallises from hot water in needles and melts at  $130^\circ$ .

The tertiary nature of hydrastine having now been established, and bearing in mind the analogy existing between narcotine and hydrastine, the constitution of the latter may be represented by the formula—



*Methylhydrastine* has probably the constitution (I.), whilst that of methylhydrastine may be represented by the formula (II.).



—F. S. K.

*The History of Hydrastine.* M. Fremd. Ber. **23**, 416—417.

A CLAIM for priority.—F. S. K.

*Taxin, the Alkaloid of the Yew Tree (Saxus Baccata).* A. Hilger and F. Brande. Ber. **23**, 464—468.

BEJARDIN, Schroff, and Lucas have pointed out the presence of a narcotic substance in the leaves, shoots, and fruit of the yew tree, whilst Marmé isolated the alkaloid in question by extracting the leaves, &c. with ether, treating the residue, after distilling off the ether, with dilute acid, and then precipitating the base by means of ammonia. The authors have adopted this method of Marmé's for the extraction of the alkaloid, to which the name of *taxin* is given. The acid extract (sulphuric acid was used) is coloured at first, but on repeating the treatment with acid and ammonia two or three times a perfectly colourless product is obtained in the form of a white amorphous powder, melting at  $82^\circ C$ . All attempts to obtain the alkaloid in a crystalline condition failed. Taxin when heated in a glass tube forms a white cloud, which condenses in oily drops on the cool parts of the tube. These drops solidify on cooling. At the same time it gives off a characteristic aromatic odour. It dissolves readily in alcohol and ether, less readily in chloroform, very slightly in water, and is insoluble in benzene. With concentrated sulphuric acid an intense purple-violet colouration results, whilst the slightly acidified aqueous solution of the base gives yellow precipitates with solutions of iodine and of bismuth iodide in potassium iodide, a pale yellow precipitate with sodium phospho-molybdate, and a white precipitate with the fixed alkalis and with ammonia, insoluble in excess. Gold chloride, platinum chloride and picric acid also produce characteristic precipitates of double salts. The alkaloid possesses a very bitter taste. Taxin forms a series of very soluble salts, of which the acetate, oxalate, tartrate, hydrochloride, and sulphate have been prepared. It is difficult to obtain them in a crystalline form, the hydrochloride being prepared as a crystalline precipitate by passing dry hydrochloric acid gas into an anhydrous ethereal solution of the base. Analyses of the free base, its hydrochloride, sulphate,



and also of its difficultly soluble double salts with the chlorides of gold and of platinum are given, which point to the empirical formula  $C_{37}H_{52}O_{10}N$  for the alkaloid. It is mon-acid. Heated with ethyl iodide in a closed tube to  $100^\circ$ , a crystalline product  $C_{37}H_{52}O_{10}N.C_2H_5I$  results, pointing to the fact that taxin is a nitrile-base.—C. A. K.

*Dextro-rotatory Cocaine.* A. Einhorn and A. Marquardt. Ber. **23**, 468—474.

THE experiments of Ladenburg and others have shown that certain atropine derivatives are capable of being converted into molecular isomers which differ in their action on polarised light and in their physical properties. Since cocaine like atropine contains two asymmetric carbon atoms, the authors have tried similar experiments with cocaine derivatives, and find that eegonine, eegonine methylester, benzoyl eegonine, cocaine, and the alkaloids that are found with cocaine in the coca leaves, are all converted, by warming with caustic potash, into a dextro-rotatory eegonine. This body differs from ordinary eegonine in its physical properties; it melts at  $254^\circ$ , ordinary eegonine at  $198^\circ$ . The rotatory power of a 4.4 per cent. solution in a 2 dem. tube is  $+1.6$ . Its hydrochloride and double salt with gold chloride both differ somewhat from the corresponding salts of ordinary eegonine. Heated with glacial acetic and hydrochloric acids, anhydro-eegonine, identical with the product derived from ordinary eegonine results. The methylester of dextro-rotatory eegonine is readily prepared, and is a crystalline body melting at  $115^\circ$ . When heated with benzoylchloride it is converted into a dextro-rotatory cocaine, the hydrochloride of which can be obtained from aqueous or alcoholic solution in beautiful crystals, melting at  $205^\circ$ . Ordinary cocaine hydrochloride melts at  $181^\circ.5$ . A 1.9 per cent. solution of this salt in dilute alcohol in a 2 dem. tube has a rotatory power of  $+1^\circ.5$ .

The physiological action of this isomer of cocaine differs from that of ordinary cocaine only inasmuch as that its local anæsthetic action takes effect and disappears more rapidly. The authors hope to be able to prepare dextro-rotatory compounds in a similar way from the other levo-rotatory cocaine derivatives, and to convert ordinary cocaine into this new isomer by an analogous reaction.—C. A. K.

*A By-product in the Commercial Synthesis of Cocaine.* C. Liebermann and F. Giesel. Ber. **23**, 508-512.

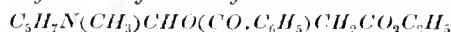
WHAT appears to be a methyl cocaine has been obtained by the authors in small quantities from the mother liquors in the synthetical method of preparation of cocaine from eegonine. The free base when separated from a salt by means of an alkaline carbonate forms an oil which solidifies on standing, and which is readily soluble in ether, alcohol, benzene, and petroleum spirit. Its salts crystallise well, the difficultly soluble nitrate  $C_{18}H_{29}NO_3.HNO_3$  being especially characteristic. The hydrochloride, sulphate, gold and platinum double chlorides are also described. Methyl cocaine is less readily decomposed by water or by hydrochloric acid than ordinary cocaine is. With acid, benzoyl-methyl eegonine hydrochloride ( $C_{17}H_{23}NO_3.HCl$ ) first results, and then methyl-eegonine,  $C_{16}H_{21}NO_3$ , which latter melts with decomposition at  $264^\circ$  and forms a characteristic hydrochloride and gold double salt. The analytical data on which the determination of the composition of this methyl cocaine rest are not sufficiently accurate to decide between the formulæ for cocaine and methyl cocaine, and the authors refer to the isomeride of cocaine described by Einhorn and Marquardt (this Journal, 1889, 135,) which agrees in some of its properties, especially as regards the melting points of its hydrochloride and gold double salt with this methyl cocaine. Further, the latter, as well as the "methyl eegonine," have a dextro-rotatory power equivalent to that of the corresponding derivatives described by Einhorn and Marquardt. On the other hand there are several differences to be noticed in the two series of compounds, so that their identity or otherwise still remains to be proved.—C. A. K.

*On Dextrococaine and its Homologues.* A. Einhorn and A. Marquardt. Ber. **23**, 979-988.

THIS body was obtained in crystals, melting at  $43-45^\circ C$ , by treating its solution with a crystal of benzoyldextro-eegonineethylester. Its gold and platinum double salts, the hydrobromide, hydriodide, nitrate and sulphate are described. Whether dextrococaine is identical with Liebermann and Giesel's methylecocaine is not yet finally decided, though the bases are very much alike; there are however still slight differences, and further researches on this point require to be made.

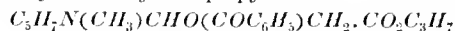
*Benzoyldextro-eegonine hydrochloride* is obtained by heating dextrococaine for 48 hours with water. After removing some benzoic acid with ether, the aqueous solution is concentrated, hydrochloric acid added, and the salt recrystallised from alcohol or water. It forms needles or prisms melting at  $244-245^\circ C$ . The dextro-eegonine esters are prepared by covering the hydrochloride with the particular alcohol, and passing gaseous hydrochloric acid into the mixture, until the salt is dissolved, the excess of alcohol is removed by evaporation, and from the residue the ether is liberated by potassium carbonate. All these compounds prepared hitherto, consist, with the exception of the amylester, of oils; the amylester forms yellow prisms melting at  $152^\circ C$ . If these esters be mixed with double their volume of benzoylchloride and heated in the oil bath to  $160-165^\circ$ , removing benzoic acid, and addition of potassium carbonate, new alkaloids were obtained.

*Benzoyldextro-eegonine ethylester—*



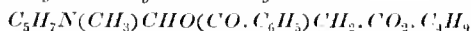
White hard prisms melting at  $57^\circ C$ ; its hydrochloride melts at  $215^\circ C$ .

*Benzoyldextro-eegonine propylester—*



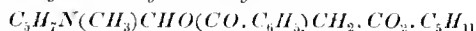
The hydrochloride melts at  $220^\circ C$ . The free base is an oil.

*Benzoyldextro-eegonine isobutylester—*



The hydrochloride melts at  $201^\circ C$ . The free base is an oil.

*Benzoyldextro-eegonine amylester—*



is an oil. The hydrochloride melts at  $217^\circ C$ .—A. L.

*The Physiological action of Selenious Acid.* C. Chabrie and L. Lapieque. Compt. Rend. **110**, 152—154.

CHABRIÉ has shown (Bull. Soc. Chim. **50**, 133) that selenious acid is reduced to selenium by a glucose solution containing yeast, but not by a sterilised solution. It is now shown that (1) the proportion of selenious acid necessary to prevent fermentation of *bouillon* exposed to the ordinary microbes present in air slightly exceeds 2 per thousand; (2) that with a smaller percentage of  $SeO_2$ , fermentation takes place and selenium is deposited on the micro-organisms.

Sodium selenite (perfectly neutral) injected into the blood of dogs in the proportion of 3 per 1,000 of the total weight of the animals, was found to be fatal. The physiological phenomena induced are described in detail, and it is concluded that sodium selenite is an irritant poison. The authors failed to discover the acicular crystals which Rabateau found after similar experiments. They contrast the toxicity of selenites with the innocuity of sulphites, these being immediately after injection transformed into sulphates.

—P. J. H.

*Report on New Drugs and Fine Chemicals.* Merck's Bull. 1890 [3], 17—24.

*Borates of Alkaloids.*—Petit and Galezowsky (Les nouv. Remèdes, 1889, 143) especially recommend these for the preparation of eye washes, since boric acid, even if present

in excess, will exert no deleterious action in such application, whilst any other acid which might be combined with the alkaloids used would produce irritant effects. The following borates of mydriatic and myotic alkaloids have so far been placed on the market:—Atropine-, cocaine-, physostigmine- (eserine), morphine-, and pilocarpine-borates.

**Cetraric Acid (Cetrarin),**  $C_{14}H_{10}O_8$ , the bitter principle contained in *Cetraria islandica* (Iceland moss), forms white needles with a bitter taste and readily soluble in boiling alcohol. Physiological and therapeutic researches on cetraric acid were first made by Köhler and Müller (Pflanzenstoffe. Husemann and Hilgers, 318), and these were supplemented by the more recent investigations of A. Fortunatow (Zur Frage über die Wirkung der Bittermittel, Inaug. Dissert. St. Petersburg, 1884), which developed new and interesting facts.

Fortunatow showed that intravenous injections of cetraric acid augment the secretions of the pancreas, and still more so those of the salivary glands. Further, that doses of 2–4 centigrams per kilo. of the animal's weight (2–4 parts per 100,000) injected into the blood increase the biliary secretion by 2–4-fold its usual quantity.

Intravenously injected, cetraric acid has no effect on the gastric digestion, whereas, administered by the mouth, it exerts a disturbing influence thereon. Professor Kobert, of Dorpat University, promises further experimental results.

**Codeine Phosphate**, soluble in 4 parts of water, is used hypodermically in cases of insomnia, insane excitation, and hallucinations. It acts less energetically than hyoscyne, and is useful when the use of the latter is accompanied by hallucinations.

Codeine phosphate is also useful for the treatment of morbinism, as demonstrated by Schmidt (Wiener Med. Presse, 1889, 43). A 10 per cent. solution was found best for internal use.

**Di-iodosalicylic Acid,**  $C_6H_3I_2(OH).CO_2H$ .—This is a white powder, consisting of microscopic needles, but little soluble in cold water, rather more soluble in hot water, and readily soluble in alcohol and ether. It acts as an analgetic and antithermic at the same time, retarding the action of the heart. It is excellent as an antiseptic.

Di-iodosalicylic acid was especially valuable in certain cases where salicylic acid and its salts had remained void of effect.

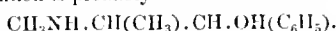
**Lobeline, Alkaloid and Sulphates.**—The alkaloid is obtained from the leaf or seeds of the plant "*Lobelia inflata*," and it takes the form of a yellow syrupy liquid which darkens on keeping. The sulphate is best suited for medicinal use. Two sulphates are obtainable, viz.:—

**Lobeline Sulphate** from the Lobelia seeds. Yellowish, crumb like, little granules, extremely hygroscopic, and hence difficult of exhibition in powder form.

**Lobeline Sulphate** from the leaves.—Yellowish white powder, less hygroscopic than the sulphate from the seeds. The physiological actions of these sulphates have not been observed to differ from each other.

Dreser states (Archiv für exp. Path. and Pharm. 1889, 237) that *Lobeline* is the only medicinally active principle peculiar to the leaves and seeds of *Lobelia inflata*. It kills warm-blooded animals by paralyzing the respiration. Silva Nunez has recently most warmly recommended the use of Lobeline in the treatment of bronchitical dyspnea and spasmodical asthma. (Brit. Med. J. 1889, 1051.) This alkaloid is immensely superior to the old Galenic preparation of lobelia.

**Pseudo-Ephedrine,**  $C_{10}H_{15}ON$ .—The pure alkaloid is useful as a mydriatic. It is a crystalline substance and has a faint but agreeable odour. It is easily soluble in alcohol, with difficulty in cold water, somewhat more readily in hot water. Melting point (uncorrected) about  $114^{\circ}$ – $115^{\circ}$  C. The constitution is probably—



The physiological action on the eye has been investigated by A. de Vries (Ann. Oculist du Warlomont, 1889, iii.

and iv., 183). The practical advantages in its use over other mydriatics are summed up thus:—"It occasions neither inflammation nor disturbances of refraction; it does not modify the intra-ocular pressure, and it can be employed for a long time without causing any injury." (This Journal, 1889, 724; also *ibid.*, 304).

**Theobromine and Sodium Salicylate ("Diuretin").**—This is a white crystalline powder, but slightly soluble in cold water. It is soluble in hot water and in warm dilute alcohol, but insoluble in ether and in chloroform. The salt has been clinically investigated by Ch. Gram (Therapeut. Monatsh. 1890, 10), who states that it is much more easily absorbed than simple theobromine. Its action is strongly diuretic, whilst it is devoid of any toxic properties and free from the peculiar excitant influence on the central nervous system possessed by Caffeine. It was first prepared by Merck at the instance of Dr. Riegel of Giesseur.

Merck's Bull. 1890 [4], 25–32.

**Abrin** is obtained from the seeds of *Abrus precatorius* ("Indian liquorice" or "*Jequirity*"). It is a brownish-yellow powder soluble in water, and consists of an exceedingly poisonous albuminoid or proteid substance belonging to the class of unorganised ferments. In these respects it much resembles "Ricin" (page 545). The lethal dose, according to Kobert, is only 0.00001 gram per kilo. of the animal's weight, i.e. 1 to 100,000,000. Hence the enormous toxicity of the substance calls for extreme caution in its preservation, handling, and exhibition. Of high therapeutic interest is the fact that Kobert has succeeded in inducing the well known "*Jequirity ophthalmia*" both by "*Abrin*" and "*Ricin*." (Brit. Med. J. 1889, 1053).

**Ethylbromide,  $C_2H_5Br$ .**—This is becoming more and more recognised as superior, especially in small operations, to chloroform, and Gilles of Cologne, after employing it in over 450 painful dental operations says:—"Ethylbromide, when chemically pure, is a most excellent general anæsthetic, because it acts promptly and certainly, and when employed with due care, without danger. Its advantages over chloroform, nitrous oxide, cocaine, and other anæsthetics in general use, are so vast that with the exception of some rare and very complicated cases, it must soon come to be recognised as the sole standard anæsthetic in dental surgery." Both C. E. Diehl (Les nouveaux Remèdes, 1889, 361), and O. Eschricht (Deutsch. med. Wochenschr. 1889, 15, 31), testify in similar terms to the superiority of ethylbromide. Careful experiments instituted on the occasion of the Sixty-second Annual Meeting of German Naturalists and Physicians at Heidelberg, brought forth the unanimous verdict from the meeting that ethylbromide is *not a heart poison*. The pure substance is a colourless, limpid, very mobile and not readily inflammable liquid, scarcely soluble in water, though miscible in all proportions with alcohol, ether, chloroform, or fatty and ethereal oils. Sp. gr. at  $15^{\circ}$ , 1.45. Boiling point,  $38^{\circ}$ – $39^{\circ}$  C. Shaken with an equal volume of distilled water, pure ethylbromide must not impart to the latter any acid reaction (Absence of  $HBr$ ). Mixed with an equal volume of pure conc. sulphuric acid, no colouration must arise in 24 hours. (Absence of ethylene, amyl, or sulphur compounds.) A few drops of ethylbromide thrown into a stratum of KI solution about 3 cm. deep, must not, in settling, show any violet colour. (Absence of free  $Br$ .) The drug must be kept in a cool, dark place, and also in small dark coloured, entirely filled and well stoppered bottles, since decomposition ensues otherwise under the influences of air and light.

**Iron Succinate.**—This is an amorphous, reddish-brown powder, insoluble in water and alcohol.

In conjunction with chloroform, it is very efficacious in biliary calculi (Buckler, Therapeutic notes in the Deutsch. med. Zeit. 1889, 79).

**Lanolin (Anhydrous).**—Consists essentially of the esters of various fat acids with cholesterol. Lanolin is quite indifferent to solutions of salts of which it is capable of absorbing and retaining almost double its own weight. At ordinary temperatures it is not attacked either by acids or aqueous alkaline solutions and its absorbability by the skin has been already referred to. (This Journal, 1886, 578—

580; 1890, 356--358.) To this property it owes its extensive use as an ointment-basis. Anhydrous lanolin has a melting point of about  $45^{\circ}\text{C}$ ., it is insoluble in ether, and it yields homogeneous mixtures with aqueous and alcoholic liquids as well as with fatty and ethereal oils. Anhydrous lanolin does not become rancid, and will keep for any length of time. Hence it is the only really eligible ointment-base for hot climates. It gives Liebermann's cholesterol reaction, and also that known as Salkowsky and Vulpinus's chloroform and sulphuric acid reaction. The full series of reactions to be fulfilled by pure lanolin have been enumerated by Bernhard Fischer (*Die neueren Arzneimittel*, 1888, 63). The use of anhydrous lanolin is especially indicated when solutions of salts or extracts in large proportions, are to be incorporated with a fatty substance. When large proportions of powdered medicaments are to be mixed directly with anhydrous lanolin without the intervention of water, it is advisable to mix 30 per cent. of olive oil, or, better still, vaselin.

**Ricin.**—This is an albuminoid or proteid substance. It is obtained from the seeds of the castor-oil plant (*Ricinus communis*), and is one of the unorganised ferments similar to *Abrin* (page ), and like the latter has enormous toxic properties and produces "*Jequivity ophthalmia*." Ricin is a white amorphous powder, most readily soluble in a 10 per cent. solution of sodium chloride. Pure ricin in aqueous solution has a neutral reaction. The high toxic powers of ricin hold no relation to the familiar effects of castor oil. Intravenously administered ricin gives lethal effects in doses even below 0.00003 grm. per kilo. of the animal's weight (3 to 100,000,000). The lethal dose by mouth, for a human being (132 lb.) would be about 0.18 grm. ( $2\frac{3}{4}$  grains). Solutions of ricin on being boiled entirely lose their active properties, whilst dry ricin sustains a temperature of  $100^{\circ}\text{C}$ . intact. Robert and Stillmark, who have investigated its physiological action (*Arbeiten des Pharm. Inst. zu Dorpat*, 1889, 3, 59), also found other and similar poisonous albuminoids in the seeds of many species of *Ricinus*, in those of *Croton Tiglium* (croton-oil plant), Barbadoes nuts (seeds of *Jatropha Curcas*), and *Jequivity*.

**Synthetic Carbolic Acid.** A. Schneider. Pharm. C. H. N. F., 1890, 68.

In order to determine whether synthetic carbolic acid turns red under the same conditions as coal-tar carbolic acid, the author has investigated the action of various metals, wood, cork, &c., and finds that the colouration is due to the presence of traces of copper, which are probably derived from the vessels in which the substance is distilled.—J. B. C.

**The Action of Saccharin.** F. Jessen. Arch. f. Hygiene, 1890, 64.

The easily soluble saccharin is without action on the malting of starch by means of ptyalin, and has a slightly retarding action in the peptonising of albumen. The presence of large quantities of saccharin has no effect on the digestion of milk or on the digestive organs themselves. By the daily use of 1—2 grm. over a period of three months the author has observed no injurious action in several cases, and the author has himself taken 5 grms. per diem without any unpleasant effects. In 27 hours the last trace of saccharin is discharged in the urine. Pure saccharin has a slightly retarding action upon bacterial ferments; but none in the case of pathogenic ferments.

—J. B. C.

**The Action of Light on Phosphorus and on some of the Properties of "Amorphous" Phosphorus.** A. Pedler. Proc. Chem. Soc. (82), 1890, 66—67.

As part of an extensive series of experiments on the action of strong light on substances, the author has studied its effect on phosphorus, and has thereby been led also to

examine and compare the several forms of allotropic phosphorus, viz., that produced by the action of light only, that produced at moderate temperatures, the commercial form, and also rhombohedral or metallic phosphorus prepared by dissolving phosphorus in lead at high temperatures.

The conclusion is arrived at that the term "amorphous phosphorus" is a distinct misnomer, and that, so far from commercial "amorphous" phosphorus constituting a separate allotropic modification of the element, it is in reality the same substance as the form called rhombohedral or metallic phosphorus, the very slight differences in character noticed between the substances in question being explained by the difference in the state of division and the slight variations conditioned by their mode of formation. Whether the term amorphous phosphorus can be truly applied to the forms made by the action of light is open to grave doubt; even in this case there appears to be distinct evidence of crystalline form, although in some instances a form which appeared to be amorphous was obtained. It is suggested that it would be better to altogether discard the use of the term *amorphous phosphorus*.

The author finds that when phosphorus is exposed to light in contact with liquids containing oxygen, such as alcohol, it tends to enter into action with them. He arrives at the conclusion that neither *in vacuo* nor at ordinary pressures is there any change whatever of red into ordinary phosphorus at  $260^{\circ}$  as ordinarily stated; and that, practically, no change occurs up to temperatures of nearly  $358^{\circ}$ , above which, *in vacuo*, change takes place, but exceedingly slowly, even up to  $445^{\circ}$ . He also describes experiments which tend to show that red phosphorus is not permanent in air, as commonly supposed.

#### PATENTS.

**A Method of producing a Substitute for Musk.** A. Baur, Gispersleben, Germany. Eng. Pat. 4963, March 21, 1889. 4d.

TOLUENE is heated with the chloride, bromide, or iodide of butyl in presence of aluminium chloride or bromide, and the resulting hydrocarbon  $\text{C}_{11}\text{H}_{16}$  distilled off from the products of the reaction by means of steam. The distillate is fractionated, and the product of boiling point  $170^{\circ}$ — $200^{\circ}$  nitrated with a mixture of fuming nitric and sulphuric acids. It is this nitro-product, which is separated by precipitation with water and recrystallisation from alcohol, that possesses a strong smell of musk.—C. A. K.

**Improvements in the Manufacture of Ethyl Mercaptan.** O. J. Steinhart, London. Eng. Pat. 6494, April 16, 1889. 4d.

THE object of the invention is to prepare pure ethyl mercaptan from ordinary methylated spirits. One part by weight of ordinary methylated spirits is mixed with two parts of concentrated sulphuric acid, and the mixture gently heated over the water-bath for an hour. The solution is then poured into cold water, neutralised with an alkaline carbonate and evaporated, whereby the greater part of the alkaline sulphate as well as resinous matters, due to impurities in the spirit, are removed. The impure sulphovinate solution is then heated with a strong aqueous solution of potassium sulphhydrate and the product of the reaction distilled from the water-bath, when the bulk of the methyl mercaptan escapes owing to its low boiling point, whilst the ethyl mercaptan is condensed and collected. The product is further purified by careful fractional distillation.

The patentee also converts the methylated spirits into crude ethyl chloride, and treats this product with an alcoholic solution of potassium sulph-hydrate, the resulting mercaptan being purified as described above.—C. A. K.

*Process of Manufacturing Coumarone.* A. A. Vale, London. From The Chemische Fabriken Actien Gesellschaft, Hamburg, Germany. Eng. Pat. 1422, January 27, 1890. 4d.

NAPHTHA, or the light tar oils freed from pyridine bases and phenols are well fractionated so as to separate the portion boiling between  $165^{\circ}$ — $175^{\circ}$ , and the contained coumarone precipitated as the picrate,  $C_8H_6O.C_6H_5(NO_2)_3.OH$ , by the addition of the requisite amount of picric acid. The crystals are dried and decomposed by hot water or dilute alkali, and the liberated coumarone removed by distillation with steam, the picric acid employed being recovered from the residue. (See this Journal, 1890, 275.)—C. A. K.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

*Intensification of Negatives.* C. Jones. Phot. J. 14, 40—45.

THE author proved by direct experiment that when mercuric chloride acts on the metallic silver composing the image on an ordinary gelatin plate, silver chloride and mercurous chloride are formed in strict accordance with the equation  $Ag + HgCl_2 = AgCl + HgCl$ . The properties of the product lead to the conclusion that it is a definite double chloride and not merely a mixture. When sodium sulphite comes in contact with silver chloride the latter is dissolved and probably silver sulphite is formed. When mercurous chloride is treated with sodium sulphite half the mercury is reduced to the metallic state and half dissolves in the form of mercuric sodium sulphite. The sulphite is not oxidised and does not act as a reducing agent. When mercurous silver chloride formed in accordance with the first equation is treated with sodium sulphite, all the chlorine, three-fourths of the mercury, and half the silver are dissolved, the remaining mercury and silver being reduced and forming the image. The reaction is represented by the equation  $4 HgAgCl_2 = 3 HgCl_2 + 2 AgCl + Ag_2Hg$ , the two chlorides being dissolved by the sulphite. Whether this reaction results in an increase in the opacity of the image depends mainly on the physical character of the film and of the original deposit. The author is unable to confirm the statements that the opacity of the image can be indefinitely increased by repeating the process several times or can be reduced by treating with sodium thiosulphate.

Ferrous oxalate reduces mercurous chloride and silver chloride separately to the metallic state, and it likewise reduces mercurous silver chloride completely. A method of intensification which enables the operator to control the opacity of the resulting image is to immerse the negative in a saturated solution of mercuric chloride mixed with 0.2 to 0.3 per cent. by volume of strong hydrochloric acid. When all the silver has been converted into silver chloride the plate is well washed and treated with a ferrous oxalate developer made by mixing 6 parts of a saturated solution of potassium oxalate with 1 part of a saturated solution of ferrous sulphate. All the mercuric chloride must be washed out of the film before applying the ferrous oxalate, and the latter, which acts somewhat slowly, must also be thoroughly washed away. If the resulting image be again treated with mercuric chloride, followed by ferrous oxalate, a further increase in opacity will result. A third treatment gives still more opacity, and thus the image can be gradually built up to any required density.—C. H. B.

*Properties of the Acid Fixing-Bath.* A. Lainer. Phot. Corr. 1890, 16—18. (Compare this Journal, 1889, 637; 1890, 102.)

A STRONG acid fixing-bath, made by mixing equal parts of sodium thiosulphate solution and sodium hydrogen sulphite solution, gradually removes fog from gelatin negatives without any sensible loss of detail and without injuring the film. The reducing action is greatly accelerated by addition of a very small quantity of potassium ferrieyanide. The acid fixing-bath also reduces the intensity of prints on albumen paper, gelatino-chloride paper, &c., but at the same time alters their colour; it is also superior to sodium thiosulphate alone as an accelerator in conjunction with the ferrous oxalate developer, as Belitski has already stated. It will also remove brown stains resulting from mercurial intensification or from contact of the moist film with paper containing silver nitrate. Silver sulphide is precipitated much more readily from waste acid fixing-bath than from the ordinary neutral bath.—C. H. B.

*Silver in the Fixing Bath used for Gelatin Negatives.* A. Lainer. Phot. Corr. 1890, 70.

AN old fixing bath was found to contain 184 grms. of silver per hectolitre. In order to obtain this quantity of silver about 1,230 plates of cabinet size must have been fixed.

—C. H. B.

*Formaldehyde Sodium Bisulphite as an Addition to Gelatino-Bromide Emulsion and to Developers.* J. M. Eder. Phot. Corr. 1890, 105—107.

ADDITION of formaldehyde-sodium bisulphite to an emulsion does not materially accelerate the process of ripening, but tends to produce fog, and gives no appreciable increase in sensitiveness. When added to the ordinary pyrogallol developer its effect is uncertain, although in some cases a greater amount of detail is obtained in the resulting image. Immersion of an exposed plate in a solution of 1 part of the salt in 1,000 to 2,000 parts of water previous to development with ferrous oxalate reduced the time required for development and gave an image with a greater amount of detail, in this respect behaving like a very dilute solution of sodium thiosulphate. In order to avoid fog, however, it is desirable to wash the plate after removing it from the preliminary bath and before placing it in the ferrous oxalate solution.

—C. H. B.

*Magnesium Chloride as a Fixing Agent.* Liesegang. Phot. Arch. 1890, 76.

MAGNESIUM chloride dissolves silver chloride from paper prints as completely as sodium thiosulphate does. Fixing proceeds somewhat rapidly, the colour of the image is much less affected than when thiosulphate is used, and the excess of the fixing agent is readily washed out of the paper.

—C. H. B.

## PATENTS.

*Improvements in the Production of Developers for Photographic Use.* H. W. Jones, Coventry. Eng. Pat. 8085, May 15, 1889. 4d.

AN alkaline sulphite, preferably crystallised sodium sulphite, is heated until all water of crystallisation is expelled, when it is ground with quinol (hydroquinone) preferably in equal proportion, sodium bromide being added in quantity amounting to one-tenth of the whole mixture. The powder is moistened with dilute sulphurous acid, partially dried, forced through a sieve with 40 meshes to the inch, dried at a gentle heat, and the granular product compressed into discs, tablets, or pellets by means of a powerful press.

—C. H. B.

*Improvements in Apparatus for the Manufacture of Photographic Films.* A. J. Boulton, London. From G. Eastman, Rochester, U.S.A. Eng. Pat. 19,896, December 10, 1889. 8d.

AN apparatus for the manufacture of flexible transparent films from a solution of nitrocellulose and camphor in volatile solvents. It consists of a long bed-plate of polished plate glass or any other suitable material having a polished or dead surface. Over this travels, in a longitudinal direction, the spreading apparatus, which consists of a hopper holding the solution and a transverse spreading blade made in sections and fitted with automatic adjusting mechanism which compensates for any deviation of the bed-plate from a true level. Over the bed-plate slides a light cover which protects the film from dust during evaporation and also enables part of the solvent to be recovered, the vapours being drawn off from under the cover by means of an exhaust fan placed at one end. The machine is also provided with an apparatus for cutting the film into narrower strips and reeling the latter off on spools. When the film is to be used as a support for gelatin-bromide emulsion, the film is coated whilst still on the bed-plate, to which it adheres somewhat firmly. After the emulsion has set and dried the coated film is cut and reeled off.—C. H. B.

## XXII.—EXPLOSIVES, MATCHES, Etc.

### PATENTS.

*A Machine for the Continuous Manufacture and Boxing of Matches.* L. Cobbaert, Grammont, Belgium. Eng. Pat. 6595, April 17, 1889. 1s. 7d.

"THIS invention has for its object the construction of a machine by means of which the manufacture of matches is made to constitute a single continuous operation, that is to say, all the operations of this manufacture, such as the placing in presses frames of the stems of the matches, the coating of the same with paraffin, the dipping thereof into the chemical igniting compound, the drying, removing from the frames, and packing into boxes, which up to the present have been carried out by hand by means of separate apparatus, are all effected by means of one and the same machine." In order to understand this long and complex specification it is necessary to consult the numerous drawings which accompany it, and which are fully described in the text.—W. M.

*Improvements in Solidified Pyrotechnic Powders, and in the Method of Converting the same into Solid Form for Convenience in Transportation, Storage, and Combustion.* P. Jensen, London. From H. G. Underwood, Milwaukee, U.S.A. Eng. Pat. 1884, February 4, 1890. 4d.

THE object of this invention is to convert pyrotechnic powders, such as "red fire," into the solid form, and this is attained by "adding a small quantity of alcohol to the powdered ingredients, and mixing the whole thoroughly together in a water bath at a temperature of 200° F. until the mass is in a soft pasty condition, and then, while still warm, pressing the mass into moulds previously coated with vaseline or other analogous substance, and leaving it there until cool and hardened."—W. M.

## XXIII.—ANALYTICAL CHEMISTRY.

*Research on the Action of Metals upon Sulphuric Acid.* A. Ditte. Ann. Chim. Phys. 1890, 19, 68.

THE author divides the metals which are attacked by sulphuric acid into two groups.

First, those which are acted on only with warm and concentrated acid. There is a regular production of sulphurous acid and no secondary reaction. In this group are silver, mercury, copper, lead, and bismuth.

The second group consists of the more easily attacked metals which are affected by acid of any strength, more or less energetically. The main product of the reaction is hydrogen, which is produced in the cold, and seldom ceases on heating. Sulphurous acid is only formed when warm concentrated acid is used, the temperature at which it is produced varying with the metal employed. The quantity increases with the temperature. The amount of hydrogen varies inversely, and at a high temperature it ceases entirely, and only pure sulphurous acid is formed. The production of the latter decreases on diluting, and a point is reached at which it ceases, and hydrogen is alone produced.

By regulating the concentration of the acid and the temperature a known mixture of the gases can be obtained.

This reaction does not hold good with certain metals, such as magnesium, which, by acting on sulphurous acid, produce hydrogen. Here the secondary reaction assists the chief one by increasing the amount of hydrogen, and the gas contains no foreign elements. In other cases, when by the action of sulphurous acid a sulphide is formed, the main reaction is interfered with. Sulphuretted hydrogen is produced, and reacts with the sulphurous acid; both are decomposed and sulphur is deposited. If the metals and acid be brought together in such a way as to prevent the formation of sulphurous acid, then no sulphuretted hydrogen will be produced, and pure hydrogen will be obtained. To this second group belong:—Magnesium, manganese, nickel, cobalt, iron, zinc, cadmium, aluminium, tin, and thallium, and probably also the alkaline metals. (See page 549.)

—D. A. S.

*The "Gasobrometer," an Apparatus for dispensing with Reduction Calculations in Measuring Gas Volumes.* G. Lunge. Zeits. f. angew. Chem. 1890, 139—144.

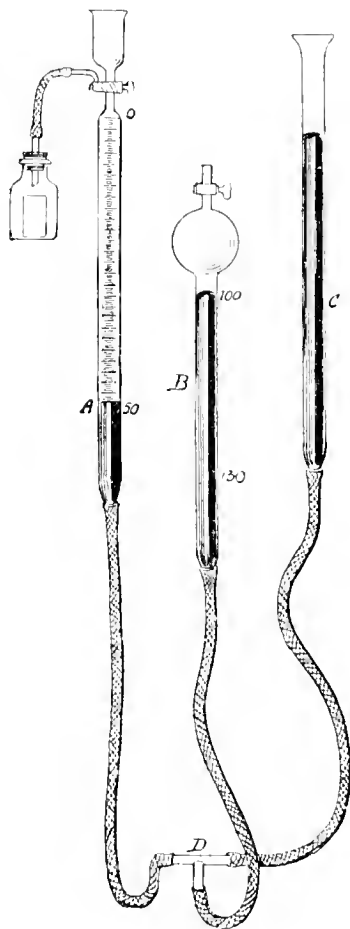
IN technical gas analysis a considerable amount of time is taken up by calculations for reducing gas volumes to standard temperature and pressure. In pure gas analysis the inconvenience is not so great; for technical purposes the initial and end temperature and pressure may be taken as the same, owing to the short duration of the experiment, and for more accurate purposes "compensators" have been devised. Where, however, the gas to be measured is evolved from a weighed quantity of a liquid or solid (so that volume and weight have finally to be connected) the matter is different, and readings of thermometer and barometer have to be made, and then the necessary calculations are to be gone through. Tables of reduction have certainly been compiled for reduction of gases at various temperatures and pressures (Dingl. Polyt. J. 231, 522), but still readings of thermometer and barometer have to be made, and part of the time only is saved. To further reduce the time occupied and to render the technical chemist in this department to a great extent independent of temperature and atmospheric pressure the present apparatus has been constructed.

By means of a T-tube, D (Fig. 1), and thick-walled rubber tubing, are connected the three tubes A, B, C. A is for measuring the gas; it may be any form of nitrometer, a Bunte's burette or other convenient burette. B is the "reduction tube," which has at its upper end a spherical or cylindrical bulb. The volume to the first mark is 100 cc., the remaining narrow portion of the tube being calibrated up to 130—140 cc. in divisions representing  $\frac{1}{100}$  cc. This "reduction tube" is set once for all at the beginning of work by observing thermometer and barometer, calculating the volume which 100 cc. of perfectly dry air, measured at 0° C. and 760 mm., would occupy under the existing

conditions. This quantity of air is then introduced, and the tube closed by means of the stop-cock shown, or by fusing up the inlet (having in place of the inlet tube shown in the Figure a tube of capillary bore). If it be necessary to measure the gas moist a drop of water is introduced into this tube, and of course in the calculation necessary the barometric pressure must be reduced by the vapour tension of water; if the gases are to be measured perfectly dry (as, for instance, when using the nitrometer with sulphuric acid), a drop of sulphuric acid takes the place of the water.

C is the pressure or levelling tube.

Fig. 1.



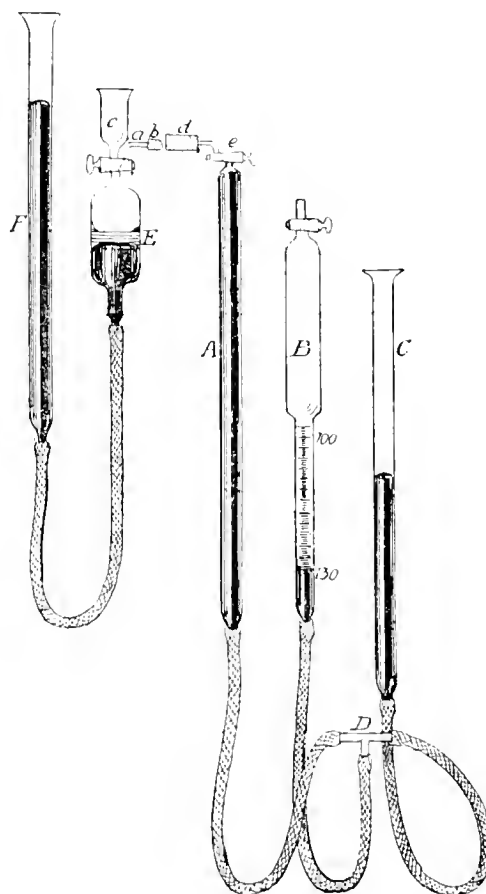
If necessary for the purpose of regulating the temperature A and B may be surrounded with water-jackets. A, B, and C are supported by spring clamps. It is easily seen that when by raising C the level of the mercury in B has been forced up to the mark, 100, exactly the amount of pressure is exerted by C as will compress the gas in B to its volume under standard conditions.

In taking a reading A and B must be levelled and the mercury level in B must have been brought up to 100. The volume shown on A is then the volume reduced to standard temperature and pressure. In cases where the gas is generated in A itself, or where the gas is transferred to A, this is all that need be done. If, however, the gas is generated in a side apparatus, as shown in Fig. 1, A and C must first be levelled and the stop-cock of A then closed so that the gas in A is collected at atmospheric pressure. After this reduction may be effected as already explained.

In nitrogen determinations by Dumas' method, A contains caustic potash as well as mercury; this is compensated by having on the reduction tube, B, a mark at a distance below

the 100 mark equal to one-tenth of the height of the caustic potash column (sp. gr. of the caustic potash equals one-tenth sp. gr. of mercury); when taking a reading the mercury in B must be at 100, and that in A must be on a level with this new lower mark of B. Similar allowance may be made in nitrometric determinations, but the ease is here more difficult, owing to the variations in the quality and specific gravity of the sulphuric acid used. It is better in such cases to liberate the gas in a separate vessel and transfer subsequently to the burette for reduction and measurement. Fig. 2 shows a convenient form of apparatus. Of course the working part E, F need not be graduated. Before beginning the operation the mercury is made to fill E with the side tube *a*, which side tube is then capped with a caoutchouc stopper to prevent escape of the mercury during subsequent shaking. A, with its side tube *e*, is also completely filled with mercury. The substance under examination, and subsequently the acid, are added through C as usual. To transfer the gas from E to A, the cap *b* is removed and *a* is fitted to *e* by means of the rubber connection *d*. F is then raised and C lowered, the taps are carefully opened, and transference effected until the acid in E just fills *e*.

Fig. 2.



A further saving of time may be effected in works, where the instrument is to be used for always one and the same object, by marking on the gas-burette or nitrometer the weight in milligrams corresponding to certain volumes; this may be done either instead of or alongside the cc. divisions; or by using a fixed quantity of substance, percentages may be marked off directly. For nitrogen determinations by Dumas' method 1 cc. of nitrogen under normal conditions weighs 1.254 mgrm. In the case of azotometric determinations of ammoniacal nitrogen (by

sodium hypobromite) the graduations may be made to represent ammonia. Correction must be made in graduating, however, for the incompleteness of the reaction. Tables giving the corrections have been introduced, but the author has shown (Chem. Ind. 1885, 165) that these may

be dispensed with, and that it is sufficient to make a correction of 2·5 per cent. For urea, however, the correction is 9 per cent.

The following table shows substances for which gasometric methods are used:—

Substance.	Basis to which Percentages are Calculated.	Method Employed.	Gas Evolved.	1 cc. of Gas = n.gram. of Basis. (Col. II.)
Organic substances .....	Nitrogen	Dumas'	N	1·254
Ammonia salts .....	Do.	Hypobromite	N	1·285*
Do. ....	Ammonia	Do.	N	1·561*
Urine .....	Urea	Do.	N	2·952*
Bone-charcoal, &c. ....	Carbon dioxide	Decomposed with HCl	CO <sub>2</sub>	1·965
Do. ....	Calcium carbonate	Do.	CO <sub>2</sub>	4·468
Pyrolusite .....	Manganese dioxide	By H <sub>2</sub> O <sub>2</sub>	O	3·882
Bleaching powder .....	Chlorine	Do.	O	1·5835
Potassium permanganate .....	Oxygen	Do.	O	0·715
Chili saltpetre .....	Sodium nitrate	Nitrometer	NO	3·805
Nitrous bodies .....	N <sub>2</sub> O <sub>3</sub>	Do.	NO	1·701
Do. ....	HNO <sub>3</sub>	Do.	NO	2·820
Do. ....	Nitric acid 36° B.	Do.	NO	5·330
Do. ....	Sodium nitrate	Do.	NO	3·805
Nitroglycerol, dynamite, &c. ....	Trinitroglycerol	Do.	NO	3·387
Do. do. ....	Nitrogen	Do.	NO	0·6267
Nitrocellulose, pyroxylin .....	Do.	Do.	NO	0·6267

\* The corrections above referred to have here already been made.

—T. L. B.

#### Action of Sulphuric Acid on Aluminium. A. Ditte.

Compt. Rend. **110**, 573—576.

As the heat of formation of aluminium hydrate is 195·8 calories, aluminium should decompose dilute sulphuric acid, and even water, at the ordinary temperature.

The author finds that when a plate of aluminium is immersed in dilute sulphuric acid (2·5 in 100 of water) there appears to be no action; but this is due to the fact that an immediate action does take place, but is at once checked by the smooth surface of the metal becoming covered with a continuous layer of hydrogen, which prevents further contact of the acid and metal. In the course of days bubbles of hydrogen detach themselves from the metal, the surface of which thus becomes roughened, giving points from which bubbles can detach themselves with increasing rapidity till all the metal dissolves; in a vacuum the action is more rapid as the hydrogen detaches itself more quickly. The behaviour of aluminium is thus exactly similar to that of amalgamated zinc.

The presence of a trace of the chloride of any metal which can be easily reduced by the aluminium, and is insoluble in dilute sulphuric acid, will hasten the dissolution of the aluminium in the acid by forming a deposit on its surface, thus breaking the continuity of the layer of hydrogen.

The behaviour of aluminium in a solution of aluminium sulphate is exactly similar to its behaviour in dilute acid, a basic aluminium sulphate being formed in this case; the action is accelerated by removing the pressure on the surface of the liquid, or by adding an easily reducible chloride.

It is for the same reason that aluminium does not appear to reduce copper sulphate in dilute sulphuric acid; the presence of a trace of reducible chloride immediately brings about the reduction by breaking the continuity of the layer of hydrogen; time produces the same result. (See page 547.)—A. G. B.

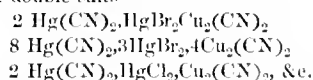
On the State of Chemical Equilibrium assumed by a Gaseous Solution originally Homogeneous, of which One Part is kept at a higher Temperature than the Rest. P. van Berchem. Compt. Rend. **110**, 82—84.

A CONTINUATION of the researches of Ludwig (Wiener Ber. 1856, **20**, 339) and Soret (Archiv. des Sciences Phys. et Nat. 1879, **2**, 48, and 1880, **4**, 209; and Compt. Rend. **91**, 289) on salt solutions. The author has studied solutions of hydrochloric acid and ammonia contained in tubes, of which one part was kept at a temperature of 50°, the other at 14°, for intervals varying from 14 to 42 days. He finds that, as with the salt solutions examined by Soret, concentration of the body dissolved takes place in the colder portion of the solution, the difference of composition varying from about  $\frac{1}{3}$  to 3 per cent. for hydrochloric acid solutions, from ·2 to 2 per cent. for ammonia solutions. Between these limits the results obtained were very inconstant.—P. J. H.

#### The action of Copper Salts on Metallic Cyanides.

R. Varet. Compt. Rend. **110**, 147—149.

WHEN cupric halogen salts are heated in solution with mercuric cyanide, they are reduced and cyanogen is evolved, and a series of double salts—



is obtained, varying with the relative proportions of mercury and copper salts present. When the copper salt is in large excess, cuprous cyanide only is formed.

The author concludes from results in this and former papers that—

- (1) the halogen salts of copper act on all cyanides;
- (2) that the oxysalts of copper act on all cyanides but those of mercury and silver.

Hence these two last salts seem to have a peculiar constitution, and to possess, as Berthelot suggests, the formulae



$\text{Ag}_2(\text{CN})_2$  and  $\text{Hg}(\text{CN})_2$ . The author considers that the cyanogen radicles are linked together in these salts and are independent in all other cyanides (including that of zinc, which has been regarded by some authors as anomalous).

—P. J. II.

*On the Strength of Reagents.* R. Blochmann.

Ber. 23, 31—35.

THE author suggests that in order to introduce some uniformity into the strength of laboratory reagents, the solutions should have a relation to the molecular weight of the substance dissolved, and has drawn up tables giving a convenient strength whilst keeping to the principle of equivalent weights. The following series are made up so as to be of twice the normal strength:—Hydrochloric acid, nitric acid, sulphuric acid, acetic acid, oxalic acid, tartaric acid, caustic potash and soda, and ammonia, ammonium sulphide, chloride and carbonate, sodium carbonate, and acetate. The following are recommended to be of normal strength:—Barium, calcium and ferric chloride, potassium, magnesium, and copper sulphate, sodium phosphate, lead acetate, potassium chromate, and potassium ferrocyanide. Half-normal solutions are to be made with platonic and mercuric chloride, silver and barium nitrate. Reagents used for oxidation and reduction are to be normal, i.e., equivalent to 8 grms. of oxygen in the litre. Such substances which do not readily dissolve in water are to be used in saturated solutions.—J. B. C.

*Preparation of Platinum Black.* O. Loew. Ber. 23,

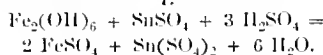
289—290.

THE catalytic action of platinum black differs considerably with the method employed for preparing the metal in this peculiar form, the product obtained by decomposing a hot solution of platonic sulphate with alcohol exhibiting this property in a greater degree than that prepared by boiling a solution of platonic chloride with sodium carbonate and sugar, or by precipitating the metal with zinc and hydrochloric acid. The author had occasion to require a very powerful catalytic agent, and after making a number of experiments succeeded in obtaining a suitable product by proceeding in the following manner:—50 grms. of platonic chloride in 50 to 60 cc. of water are mixed with 70 cc. of 40 to 45 per cent. formaldehyde, and treated gradually and with constant cooling with 50 grms. of caustic soda in 50 cc. of water. After standing for 12 hours the mixture is filtered and the precipitate washed. The filtrate, which at first has a yellow colour, assumes a black colour as soon as the greater portion of the salts (sodium chloride and sodium formate) has been removed. This is due to the fact that part of the precipitate dissolves in water at this stage; if, however, the black mass is allowed to drain on the filter it rapidly undergoes oxidation and becomes insoluble, after which the washing can be completed without further loss. The precipitate is then pressed and dried over sulphuric acid.—D. B.

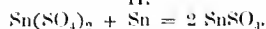
*The Precipitation of Tin from Acid Solutions by means of Metallic Iron.* B. Schultze. Ber. 23, 974—976.

THE precipitation of tin by iron occurs in neutral solutions when the tin is in the form of stannous salt. When a solution of tin is in contact with a mixture of iron rust, metallic tin, and metallic iron, any free acid present is neutralised, forming ferrous sulphate and stannic sulphate, the latter again taking up tin and passing to the condition of stannous sulphate.

I.



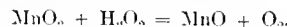
II.



So long as there is iron oxide present the stannous salt is oxidised to stannic salt. When the acid is taken up no further oxidation occurs. Though the precipitation of tin in this manner is a very slow process, the whole of the metal is precipitated.—J. B. C.

*Analysis of Pyrolusite by means of Hydrogen Peroxide.* A. Baumann. Zeits. f. angew. Chem. 1890, 72—79.

IF powdered pyrolusite and a neutral or alkaline solution of hydrogen peroxide be brought together oxygen is given off until the whole of the hydrogen peroxide is decomposed. The manganese dioxide remains unchanged, and the action is therefore "catalytic." If a strongly acid solution of hydrogen peroxide be used, then both manganese dioxide and hydrogen peroxide give up oxygen, and the reaction is quantitatively represented by the equation—



This reaction may be used for examining samples of pyrolusite in three different ways, viz., (a) gravimetrically, (b) volumetrically, and (c) gasometrically.

(a.) *The gravimetric method* may be quite easily carried out in any approved carbonic acid apparatus consisting of feed bulb for the hydrogen peroxide, generating flask, and drying tube. The weighed sample is introduced into the flask together with 30 cc. of sulphuric acid (1 of sulphuric acid to 3 of water), a sufficient quantity of hydrogen peroxide introduced into the feed bulb, and the whole apparatus then weighed. The hydrogen peroxide is next allowed to run slowly into the flask, the whole apparatus being gently shaken. When the reaction is at an end and after the whole of the evolved oxygen has been displaced by air, the apparatus is again weighed. Difference in weight is due to escape of oxygen, as shown by the equation above. It is to be noted that the manganese ore must be powdered extremely fine. According to the equation, 100 parts of oxygen correspond to 271.68 parts of manganese dioxide, hence if 2.7168 grms. of the manganese dioxide be weighed out, the number of mgrms. of loss shown give the percentage of manganese dioxide in tenths. Hence at least 2 to 3 grms. of ore are required. Where the ore contains only 30—60 per cent. of dioxide, double the quantity should be taken. For decomposition, about 40 cc. of hydrogen peroxide, as ordinarily used, are required.

(b.) *The volumetric method.* A measured quantity of standardised hydrogen peroxide is added to the finely-powdered pyrolusite, and the excess ascertained by titration with standard potassium permanganate solution. The details as recommended are as follows:—The permanganate solution is made by dissolving 6—10 grms. of potassium permanganate in a litre of water and titrating by any known method. The hydrogen peroxide generally brought into the market is too concentrated; 5 cc. should therefore be titrated with the permanganate solution, and then so much dilute sulphuric acid (1:10) should be added to the hydrogen peroxide as will make this solution approximately equivalent to the permanganate solution. For analysis 0.4—1 gm. of the finely-powdered ore is introduced into a flask or tall beaker; to this exactly 50 cc. of the prepared hydrogen peroxide solution are added, and the whole is then allowed to remain for about half an hour, being shaken from time to time. At the end of this time the excess of hydrogen peroxide is ascertained by means of the permanganate solution. In the case of ores giving a turbid brown liquid, titration is difficult. Hence to obtain accurate results the weighed sample is introduced into a 100 cc. flask, the 50 cc. of hydrogen peroxide are run in, and the whole is allowed to stand for half an hour, after which the flask is filled to the containing mark, and the contents are filtered through a double filter; 50 cc. of the filtrate are then titrated. This method admits of quite a number of determinations being carried on at the same time. Ores containing ferrous oxide may not be examined by this method, it is obvious.

(c.) *The gasometric method.*

At 0° C. and 760 mm. barometric pressure 100 cc. of oxygen are obtained, according to the equation already given, from 0.38825 gm. of manganese dioxide; hence 0.1 cc. represents 0.1 per cent. Thus it will not do to use a collecting vessel of only 50 cc. capacity, using half the quantity of pyrolusite; the errors of experiment become much too high. The endiometer or burette used (of whatever kind it be) should be capable of measuring up to 100 cc., and should be provided with a water-jacket. The decomposition apparatus consists of a test-tube provided

with doubly-bored stopper, through one hole of which passes a glass rod with, fused to its inner end, a small vessel capable of holding 10 cc. of hydrogen peroxide; through the other hole of the stopper passes a glass delivery tube provided with a tap, and connected with the gas burette by means of rubber tubing. The ore is accurately weighed into the test-tube, and to it are added 20 cc. of dilute sulphuric acid; 10 cc. of hydrogen peroxide are filled into the small vessel fastened to the stopper, and the stopper is then fixed into position. Before connecting with the gas burette the

decomposition apparatus is brought to the temperature of the gas burette. The reaction is easily brought about by tilting the decomposition apparatus, whereby the hydrogen peroxide is caused to run from its containing vessel. Water is used in the burette. As regards calculation of results, it is first necessary to reduce to 0° C. and 760 mm. barometric pressure, and then to multiply by the factor for converting oxygen to manganese dioxide. Both these may be performed, with the aid of the accompanying table, by a single multiplication.

WEIGHT OF MANGANESE DIOXIDE IN MGMS. CORRESPONDING TO 1 CC. OF OXYGEN AT TEMPERATURES VARYING FROM 10° C. to 25° C., AND PRESSURES VARYING FROM 700 to 770 MM.

$$\text{Calculated from } \frac{(p - w) 3.8825}{760 (1 + 0.00366t)}$$

(Between 10° and 12° subtract 1 mm. from observed barometric height; between 13° and 19°, 2 mm.; between 20° and 25°, 3 mm., for reduction of the mercury column to 0° C.)

Baro- metric Height.	10°	11°	12°	13°	14°	15°	16°	17°	18°	19°	20°	21°	22°	23°	24°	25°	Baro- metric Height.
mm.	mg.	mg.	mg.	mg.	mg.	mg.	mg.	mg.	mg.	mg.	mg.	mg.	mg.	mg.	mg.	mg.	mm.
700	3.405	3.390	3.374	3.359	3.344	3.329	3.313	3.297	3.282	3.265	3.250	3.233	3.217	3.200	3.183	3.166	700
702	3.415	3.400	3.384	3.369	3.354	3.338	3.323	3.307	3.291	3.275	3.259	3.243	3.226	3.210	3.193	3.175	702
704	3.424	3.409	3.394	3.379	3.364	3.348	3.332	3.317	3.301	3.285	3.269	3.252	3.236	3.219	3.202	3.185	704
706	3.434	3.419	3.404	3.389	3.373	3.358	3.342	3.326	3.310	3.294	3.278	3.262	3.245	3.228	3.211	3.194	706
708	3.444	3.429	3.414	3.399	3.383	3.367	3.352	3.336	3.320	3.304	3.288	3.271	3.255	3.238	3.221	3.204	708
710	3.454	3.439	3.423	3.408	3.393	3.377	3.361	3.345	3.329	3.313	3.297	3.281	3.264	3.247	3.230	3.213	710
712	3.464	3.449	3.433	3.418	3.402	3.387	3.371	3.355	3.339	3.323	3.307	3.290	3.274	3.257	3.240	3.222	712
714	3.474	3.459	3.443	3.428	3.412	3.396	3.381	3.365	3.349	3.333	3.316	3.300	3.283	3.266	3.249	3.232	714
716	3.484	3.468	3.453	3.437	3.422	3.406	3.390	3.374	3.358	3.342	3.326	3.309	3.293	3.276	3.258	3.241	716
718	3.494	3.478	3.463	3.447	3.431	3.416	3.400	3.384	3.368	3.352	3.335	3.319	3.302	3.285	3.268	3.250	718
720	3.503	3.488	3.473	3.457	3.441	3.426	3.410	3.394	3.378	3.361	3.345	3.328	3.311	3.294	3.277	3.260	720
722	3.513	3.498	3.482	3.467	3.451	3.435	3.419	3.403	3.387	3.371	3.354	3.338	3.321	3.304	3.287	3.269	722
724	3.523	3.507	3.492	3.476	3.461	3.445	3.429	3.413	3.397	3.380	3.364	3.347	3.330	3.313	3.296	3.279	724
726	3.533	3.517	3.502	3.486	3.470	3.454	3.439	3.422	3.406	3.390	3.373	3.357	3.340	3.323	3.305	3.288	726
728	3.543	3.527	3.512	3.496	3.480	3.464	3.448	3.432	3.416	3.399	3.383	3.366	3.349	3.332	3.315	3.297	728
730	3.553	3.537	3.521	3.506	3.490	3.474	3.458	3.442	3.425	3.409	3.392	3.376	3.359	3.342	3.324	3.307	730
732	3.563	3.547	3.531	3.516	3.499	3.483	3.468	3.451	3.435	3.418	3.402	3.385	3.368	3.351	3.334	3.316	732
734	3.572	3.557	3.541	3.525	3.509	3.493	3.477	3.461	3.444	3.428	3.411	3.395	3.378	3.360	3.343	3.325	734
736	3.582	3.566	3.551	3.535	3.519	3.503	3.487	3.470	3.454	3.437	3.421	3.404	3.387	3.370	3.352	3.335	736
738	3.592	3.576	3.560	3.545	3.529	3.513	3.497	3.480	3.464	3.447	3.430	3.413	3.396	3.379	3.362	3.344	738
740	3.602	3.586	3.570	3.554	3.538	3.522	3.506	3.490	3.473	3.456	3.440	3.423	3.406	3.389	3.371	3.353	740
742	3.612	3.596	3.580	3.564	3.548	3.532	3.516	3.499	3.483	3.466	3.449	3.432	3.415	3.398	3.381	3.363	742
744	3.622	3.606	3.590	3.574	3.558	3.542	3.525	3.509	3.492	3.476	3.459	3.442	3.425	3.408	3.390	3.372	744
746	3.631	3.616	3.600	3.583	3.567	3.551	3.535	3.519	3.502	3.485	3.469	3.451	3.434	3.417	3.399	3.382	746
748	3.641	3.625	3.609	3.593	3.577	3.561	3.545	3.528	3.511	3.495	3.478	3.461	3.444	3.426	3.409	3.391	748
750	3.651	3.635	3.619	3.603	3.587	3.571	3.554	3.538	3.521	3.504	3.488	3.470	3.453	3.436	3.418	3.400	750
752	3.661	3.645	3.629	3.613	3.596	3.581	3.564	3.548	3.531	3.514	3.497	3.480	3.463	3.445	3.428	3.410	752
754	3.671	3.655	3.639	3.623	3.606	3.590	3.574	3.557	3.540	3.523	3.507	3.489	3.472	3.455	3.437	3.419	754
756	3.681	3.665	3.648	3.632	3.616	3.600	3.583	3.567	3.550	3.533	3.516	3.499	3.482	3.464	3.446	3.429	756
758	3.691	3.675	3.658	3.642	3.626	3.610	3.593	3.576	3.560	3.542	3.526	3.508	3.491	3.474	3.456	3.438	758
760	3.700	3.685	3.668	3.652	3.635	3.619	3.603	3.586	3.569	3.552	3.535	3.518	3.500	3.483	3.465	3.447	760
762	3.710	3.694	3.678	3.662	3.645	3.629	3.612	3.595	3.579	3.562	3.545	3.527	3.510	3.492	3.475	3.457	762
764	3.720	3.704	3.688	3.671	3.655	3.639	3.622	3.605	3.588	3.571	3.554	3.537	3.519	3.502	3.484	3.466	764
766	3.730	3.714	3.697	3.681	3.665	3.648	3.632	3.615	3.598	3.581	3.564	3.546	3.529	3.511	3.493	3.475	766
768	3.740	3.724	3.707	3.691	3.674	3.658	3.641	3.624	3.607	3.590	3.573	3.556	3.538	3.521	3.503	3.484	768
770	3.750	3.733	3.717	3.701	3.684	3.667	3.651	3.634	3.617	3.600	3.583	3.565	3.548	3.530	3.512	3.494	770

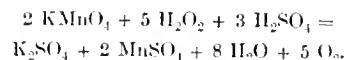
The author maintains that for accurate work a reduction apparatus should not be used. He also asserts that in calculating the results the factor of Liebig and Lunge ought not to be used, as being too high.

The accompanying table may also be so used that much trouble in calculating results is saved, thus:—By putting the decimal point in the table two figures to the right, the weight of manganese dioxide is ascertained, which, at the particular temperature and pressure specified, gives 100 cc. of oxygen. It is only necessary, therefore, before the experiment, to read the temperature of the jackets and the barometric pressure, and to weigh out exactly the amount of ore ascertained as explained; the oxygen evolved then shows directly the percentage of manganese dioxide in the sample. All the methods described are said to give good results.

—T. L. B.

*Tables for Strength of Potassium Permanganate Solution, and for use in the Valuation of Bleach.* L. Vanino. Zeits. f. angew. Chem. 1890, 80—83.

1. *Potassium Permanganate.*—The reaction with hydrogen peroxide is represented by the equation—



According to concentration 10 to 50 cc. of permanganate solution are treated with hydrogen peroxide and the oxygen evolved is measured in a Wagner's "azotometer." The calculation of results is simplified by making use of the accompanying table.

TABLE I.—WEIGHT OF 1 CC. OF OXYGEN IN MGMS. AT TEMPERATURES VARYING FROM 10° C. TO 25° C., AND PRESSURES VARYING FROM 700 TO 770 MM.

Calculated from $(p - w) \cdot 1.42908$ 760 (1 + 0.00366t)								(Barometric reading to be reduced as indicated in Table on p. 551.)									
Barometric Height.	10°	11°	12°	13°	14°	15°	16°	17°	18°	19°	20°	21°	22°	23°	24°	25°	Barometric Height.
mm. 700	mg. 1.253	mg. 1.248	mg. 1.242	mg. 1.237	mg. 1.231	mg. 1.225	mg. 1.219	mg. 1.214	mg. 1.208	mg. 1.202	mg. 1.196	mg. 1.190	mg. 1.184	mg. 1.178	mg. 1.172	mg. 1.165	mm. 700
702	1.257	1.251	1.245	1.240	1.234	1.229	1.223	1.217	1.211	1.205	1.200	1.194	1.188	1.181	1.175	1.169	702
704	1.260	1.255	1.249	1.244	1.238	1.232	1.227	1.221	1.215	1.209	1.203	1.197	1.191	1.185	1.179	1.172	704
706	1.264	1.259	1.253	1.247	1.241	1.235	1.230	1.224	1.218	1.212	1.207	1.201	1.194	1.188	1.182	1.176	706
708	1.268	1.262	1.256	1.251	1.245	1.239	1.234	1.228	1.222	1.216	1.210	1.204	1.198	1.192	1.186	1.179	708
710	1.271	1.266	1.260	1.254	1.249	1.243	1.237	1.231	1.225	1.219	1.214	1.208	1.202	1.195	1.189	1.183	710
712	1.275	1.269	1.264	1.258	1.252	1.247	1.241	1.235	1.229	1.223	1.217	1.211	1.205	1.199	1.192	1.186	712
714	1.279	1.273	1.267	1.262	1.256	1.250	1.244	1.239	1.233	1.227	1.221	1.215	1.208	1.202	1.196	1.190	714
716	1.282	1.277	1.271	1.265	1.259	1.254	1.248	1.242	1.236	1.230	1.224	1.218	1.212	1.206	1.199	1.193	716
718	1.286	1.280	1.274	1.269	1.263	1.257	1.251	1.245	1.240	1.234	1.228	1.221	1.215	1.209	1.203	1.196	718
720	1.289	1.284	1.278	1.272	1.267	1.261	1.255	1.249	1.243	1.237	1.231	1.225	1.219	1.213	1.206	1.200	720
722	1.293	1.287	1.281	1.276	1.270	1.264	1.259	1.253	1.247	1.241	1.235	1.229	1.222	1.216	1.210	1.203	722
724	1.297	1.291	1.285	1.280	1.274	1.268	1.262	1.256	1.250	1.244	1.238	1.232	1.226	1.220	1.213	1.207	724
726	1.300	1.295	1.289	1.283	1.277	1.271	1.266	1.260	1.254	1.248	1.242	1.236	1.229	1.223	1.217	1.210	726
728	1.304	1.298	1.292	1.287	1.281	1.275	1.269	1.263	1.257	1.251	1.245	1.239	1.233	1.226	1.220	1.213	728
730	1.308	1.302	1.296	1.290	1.284	1.279	1.273	1.267	1.261	1.255	1.249	1.243	1.236	1.230	1.224	1.217	730
732	1.311	1.306	1.300	1.294	1.288	1.282	1.276	1.270	1.264	1.258	1.252	1.246	1.240	1.233	1.227	1.221	732
734	1.315	1.309	1.303	1.298	1.292	1.286	1.280	1.274	1.268	1.262	1.256	1.250	1.243	1.237	1.231	1.224	734
736	1.318	1.313	1.307	1.301	1.295	1.289	1.283	1.277	1.271	1.265	1.259	1.253	1.247	1.240	1.234	1.227	736
738	1.322	1.316	1.310	1.305	1.299	1.293	1.287	1.281	1.275	1.269	1.263	1.256	1.250	1.244	1.237	1.231	738
740	1.326	1.320	1.314	1.308	1.302	1.296	1.290	1.285	1.278	1.272	1.266	1.260	1.254	1.247	1.241	1.234	740
742	1.329	1.324	1.318	1.312	1.306	1.300	1.294	1.289	1.281	1.276	1.270	1.263	1.257	1.251	1.244	1.238	742
744	1.333	1.327	1.321	1.315	1.309	1.304	1.298	1.292	1.285	1.279	1.273	1.266	1.261	1.254	1.248	1.241	744
746	1.337	1.331	1.325	1.319	1.313	1.307	1.301	1.295	1.289	1.283	1.277	1.270	1.264	1.258	1.251	1.245	746
748	1.340	1.334	1.328	1.323	1.317	1.311	1.305	1.299	1.292	1.286	1.280	1.274	1.267	1.261	1.255	1.248	748
750	1.344	1.338	1.332	1.326	1.320	1.314	1.308	1.302	1.296	1.290	1.284	1.277	1.271	1.264	1.258	1.252	750
752	1.347	1.342	1.336	1.330	1.324	1.318	1.312	1.306	1.300	1.293	1.287	1.281	1.274	1.268	1.261	1.255	752
754	1.351	1.345	1.339	1.333	1.327	1.321	1.315	1.309	1.303	1.297	1.291	1.284	1.278	1.272	1.265	1.258	754
756	1.355	1.349	1.343	1.337	1.331	1.325	1.319	1.313	1.307	1.300	1.294	1.288	1.282	1.275	1.269	1.262	756
758	1.358	1.352	1.346	1.341	1.334	1.329	1.322	1.316	1.310	1.304	1.298	1.291	1.285	1.279	1.272	1.265	758
760	1.362	1.355	1.350	1.344	1.338	1.332	1.326	1.320	1.314	1.307	1.301	1.295	1.288	1.282	1.276	1.269	760
762	1.366	1.360	1.354	1.348	1.342	1.336	1.330	1.323	1.317	1.311	1.304	1.298	1.292	1.285	1.279	1.272	762
764	1.369	1.363	1.357	1.351	1.345	1.339	1.333	1.327	1.321	1.314	1.308	1.302	1.295	1.289	1.282	1.276	764
766	1.374	1.367	1.361	1.355	1.349	1.343	1.337	1.331	1.324	1.318	1.312	1.305	1.299	1.292	1.286	1.279	766
768	1.377	1.371	1.365	1.359	1.353	1.346	1.340	1.334	1.328	1.322	1.315	1.309	1.302	1.296	1.289	1.283	768
770	1.380	1.374	1.368	1.362	1.356	1.350	1.344	1.338	1.331	1.325	1.319	1.312	1.306	1.299	1.293	1.286	770

II. *Bleach*.—50 cc. of bleaching powder solution are treated in an "azotometer" with hydrogen peroxide. The gas volume is then to be multiplied by the number given

by the accompanying table. The reaction is represented by the equation—

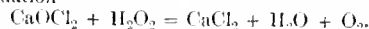


TABLE II.—WEIGHT OF 1 CC. OF CHLORINE IN GRAMS, AT TEMPERATURES VARYING FROM 10° C. to 25° C., AND PRESSURES VARYING FROM 700 to 770 MM.

Calculated from  $\frac{(p - w) 3 \cdot 16696}{760 (1 + 0 \cdot 00366t)}$   
(Barometric reading to be reduced as indicated in Table on p. 551.)

Baro- metric Height.	10°	11°	12°	13°	14°	15°	16°	17°	18°	19°	20°	21°	22°	23°	24°	25°	Baro- metric Height.
mm.	mg.	mg.	mg.	mg.	mg.	mg.	mg.	mg.	mg.	mg.	mg.	mg.	mg.	mg.	mg.	mg.	mm.
700	2'777	2'765	2'752	2'740	2'728	2'715	2'702	2'690	2'677	2'664	2'651	2'637	2'624	2'610	2'597	2'583	700
702	2'785	2'773	2'761	2'748	2'736	2'723	2'710	2'698	2'684	2'671	2'658	2'645	2'632	2'618	2'604	2'590	702
704	2'793	2'781	2'769	2'756	2'744	2'731	2'718	2'705	2'692	2'679	2'666	2'653	2'639	2'626	2'612	2'598	704
706	2'801	2'789	2'777	2'764	2'752	2'739	2'726	2'713	2'700	2'687	2'674	2'660	2'647	2'633	2'619	2'606	706
708	2'809	2'797	2'785	2'772	2'760	2'747	2'734	2'721	2'708	2'695	2'682	2'668	2'655	2'641	2'627	2'613	708
710	2'817	2'805	2'793	2'780	2'767	2'755	2'742	2'729	2'716	2'702	2'689	2'676	2'662	2'649	2'635	2'621	710
712	2'826	2'813	2'801	2'788	2'775	2'763	2'750	2'737	2'724	2'710	2'697	2'684	2'670	2'656	2'643	2'629	712
714	2'834	2'821	2'808	2'796	2'783	2'770	2'758	2'745	2'732	2'718	2'705	2'691	2'678	2'664	2'650	2'636	714
716	2'842	2'829	2'816	2'804	2'791	2'778	2'765	2'753	2'739	2'726	2'713	2'699	2'686	2'672	2'658	2'644	716
718	2'850	2'837	2'824	2'812	2'799	2'786	2'773	2'760	2'747	2'734	2'720	2'707	2'693	2'679	2'665	2'651	718
720	2'858	2'845	2'832	2'820	2'807	2'794	2'781	2'768	2'755	2'741	2'728	2'715	2'701	2'687	2'673	2'659	720
722	2'866	2'853	2'840	2'828	2'815	2'802	2'789	2'776	2'763	2'749	2'736	2'723	2'709	2'695	2'681	2'667	722
724	2'874	2'861	2'848	2'836	2'823	2'810	2'797	2'784	2'771	2'757	2'744	2'730	2'716	2'703	2'689	2'674	724
726	2'882	2'869	2'856	2'844	2'831	2'818	2'805	2'792	2'779	2'765	2'751	2'738	2'724	2'710	2'696	2'682	726
728	2'890	2'877	2'864	2'852	2'839	2'826	2'813	2'799	2'786	2'773	2'759	2'746	2'732	2'718	2'704	2'690	728
730	2'898	2'885	2'872	2'860	2'847	2'834	2'821	2'807	2'794	2'780	2'767	2'754	2'740	2'726	2'712	2'697	730
732	2'906	2'893	2'880	2'868	2'854	2'841	2'828	2'815	2'802	2'788	2'775	2'761	2'747	2'733	2'719	2'705	732
734	2'914	2'901	2'888	2'876	2'862	2'849	2'836	2'823	2'809	2'796	2'782	2'769	2'755	2'741	2'727	2'712	734
736	2'922	2'909	2'896	2'883	2'870	2'857	2'844	2'831	2'817	2'804	2'790	2'777	2'763	2'749	2'734	2'720	736
738	2'930	2'917	2'904	2'891	2'878	2'865	2'852	2'839	2'825	2'812	2'798	2'784	2'770	2'756	2'742	2'728	738
740	2'938	2'925	2'912	2'899	2'886	2'873	2'860	2'847	2'833	2'819	2'806	2'792	2'778	2'764	2'750	2'735	740
742	2'946	2'933	2'920	2'907	2'894	2'881	2'868	2'855	2'841	2'827	2'814	2'800	2'786	2'772	2'758	2'743	742
744	2'954	2'941	2'928	2'915	2'902	2'889	2'876	2'862	2'849	2'835	2'821	2'808	2'794	2'780	2'765	2'751	744
746	2'962	2'949	2'936	2'923	2'910	2'897	2'884	2'870	2'857	2'843	2'829	2'815	2'801	2'787	2'773	2'758	746
748	2'970	2'957	2'944	2'931	2'918	2'905	2'891	2'878	2'864	2'851	2'837	2'823	2'809	2'795	2'780	2'766	748
750	2'978	2'965	2'952	2'939	2'926	2'913	2'899	2'886	2'872	2'858	2'845	2'831	2'817	2'802	2'788	2'774	750
752	2'986	2'973	2'960	2'947	2'934	2'921	2'907	2'894	2'880	2'866	2'852	2'838	2'824	2'810	2'796	2'781	752
754	2'994	2'981	2'968	2'955	2'942	2'929	2'915	2'901	2'888	2'874	2'860	2'846	2'832	2'818	2'804	2'789	754
756	3'002	2'989	2'976	2'963	2'950	2'936	2'923	2'909	2'896	2'882	2'868	2'854	2'840	2'825	2'811	2'796	756
758	3'010	2'997	2'984	2'971	2'957	2'944	2'931	2'917	2'904	2'890	2'876	2'862	2'848	2'833	2'819	2'804	758
760	3'018	3'005	2'992	2'979	2'965	2'952	2'939	2'925	2'911	2'897	2'884	2'869	2'855	2'841	2'827	2'812	760
762	3'026	3'013	3'000	2'987	2'973	2'960	2'947	2'933	2'919	2'905	2'891	2'877	2'863	2'849	2'834	2'819	762
764	3'035	3'021	3'008	2'995	2'981	2'968	2'954	2'941	2'927	2'913	2'899	2'886	2'871	2'856	2'842	2'827	764
766	3'043	3'029	3'016	3'003	2'989	2'976	2'962	2'949	2'935	2'921	2'907	2'893	2'878	2'864	2'849	2'835	766
768	3'051	3'037	3'024	3'011	2'997	2'984	2'970	2'957	2'943	2'929	2'914	2'900	2'886	2'872	2'857	2'842	768
770	3'059	3'045	3'033	3'019	3'005	2'991	2'978	2'965	2'951	2'937	2'922	2'908	2'894	2'879	2'865	2'850	770

*Rapid Method for the Estimation of Phosphorus in Iron and Steel.* G. L. Norris. J. Frank. Inst. 1890, 99, 72—74.

This is a modification of Drown's method (Am. Inst. Min. Eng. June 1889). 5 grms. of pig iron or steel are treated in a 12-oz. beaker with nitric acid of sp. gr. 1·135, using 90 cc.

for steels and 120 cc. for pig irons. The beaker is heated until solution is complete and the acid boils, 20 cc. of a solution of potassium permanganate (containing 8 grms. of the salt per litre) are then added, and the boiling continued for a few minutes. A precipitate of manganese peroxide should be formed. The latter is then dissolved by the addition of

a crystal of tartaric acid, and 10 cc. of nitric acid sp. gr. 1.40 are added. The solution should then measure about 90 cc. In the case of a pig iron the solution is diluted to 100 cc. before the addition of the nitric acid, filtered, and the nitric acid added to 80 cc. of the filtrate. The liquid is transferred to a 500 cc. flask, 80 cc. of a solution of ammonium molybdate added, and the whole violently shaken for five minutes. The precipitate is then reduced with zinc and titrated with permanganate of potassium in the usual way. The molybdate solution is prepared by dissolving 100 grms. of molybdic acid in 400 cc. of strong ammonia, and adding the solution to 1,200 cc. of nitric acid sp. gr. 1.20.

For "high phosphorus pig-iron" only 1.25 grms. are weighed out and dissolved in 60 cc. of the nitric acid, and the solution treated as before. The whole analysis can be made in 35 minutes. Tables of results accompany the paper.—H. K. T.

#### *The Volumetric Determination of Zinc and Copper.*

E. Donath and G. Hattensaur. Chem. Zeit. 14, 323—325.

THE authors find that the use of standard sodium sulphide solution prepared by saturating caustic soda solution with sulphuretted hydrogen and then adding an equal volume of caustic soda, is to be preferred to the use of crystallised sodium sulphide for titrating zinc. The method of precipitating zinc with an excess of potassium ferrocyanide and estimating the excess of the latter by permanganate was abandoned as the precipitate did not settle readily and the clear liquid could not be decanted. The authors recommend the precipitation of zinc in an ammoniacal tartaric acid solution with potassium ferrocyanide. Ferric oxide is not precipitated in such a solution. The solution should not be too strongly alkaline, and the addition of ferrocyanide should take place in the hot solution. The reaction is complete when a drop of the solution acidified with acetic acid gives a blue colouration supposing iron to be previously present in the solution. 1 mol. of ferrocyanide is equivalent to 2 atoms of zinc, and therefore 33.5 grms. of ferrocyanide in 1 litre correspond to 10 grms. of zinc, or 1 cc. is equivalent to .010 gm. of zinc. The operation is carried out as follows: 3—4 grms. of the ore, &c. are dissolved in concentrated hydrochloric acid, with the addition of nitric acid, filtered, and diluted to a given volume. A measured portion is withdrawn, 20—25 cc. of a concentrated tartaric acid solution added, and then ammonia until alkaline. The solution is heated to 80°, and potassium ferrocyanide run in from a burette, the end of the reaction being determined as above.

Copper may be estimated in the same way, but the precipitation is greatly dependent upon the amount of ammonia present. The solution should be as nearly neutral as possible. The equivalent in copper of the ferrocyanide must be determined by direct experiment as no definite copper compound is precipitated. The end of the reaction is recognised in the same way as for zinc. This method enables the zinc and copper to be estimated in the same solution. The two are first titrated together, the copper is then precipitated with iron and the clear solution, after oxidation of the iron, again titrated for zinc.—J. B. C.

#### *The Estimation of Cadmium in the Products of Zinc Manufacture.* W. Minor. Chem. Zeit. 14, 348—349.

IN this paper the author gives a direct and an indirect method for the determination of cadmium. In the indirect method, the sample to be analysed is dissolved in hydrochloric acid, filtered from undissolved lead, and precipitated with sulphuretted hydrogen. The precipitate of cadmium sulphide, containing more or less zinc sulphide, is filtered off and weighed. It is then dissolved in hydrochloric acid, the solution is rendered strongly alkaline with caustic soda, and filtered from the precipitated cadmium hydrate. In the filtrate the zinc is determined by titration with sodium sulphide. The zinc found is calculated into sulphide and deducted from the weight of the cadmium sulphide precipitate. In the direct method the cadmium must be separated

in a state of purity and converted into neutral cadmium chloride. The solution of the cadmium salt is then titrated with standard sodium hydrate, using sodium sulphide paper or litmus paper as an indicator. The direct method is preferably used in the case of nearly pure cadmium compounds, whilst the indirect method is most suitable in the case of bodies containing much zinc. The test analyses given are quite satisfactory. (See also this Journal, 1890, 109.)—H. T. P.

#### *Quantitative Separation of Tin and Titanium, with Special Reference to the Analysis of Silicates.* A. Hilger and H. Haas. Ber. 23, 458—461.

FOR the separation of tin and titanium the mixed oxides are placed in a hard glass tube 15—20 cm. in length and heated in a current of hydrogen by a Bunsen burner for a quarter of an hour. The stannic oxide is thus reduced to metal, whilst the titanium dioxide remains unacted upon. The mixture is washed with water and a few drops of hydrochloric acid into a beaker, 30 cc. of 20 per cent. hydrochloric acid are added, and the whole boiled gently for half an hour, filtered, and washed. The tin contained in the filtrate is precipitated as sulphide, the precipitate washed with water containing ammonium acetate, again reduced in a current of hydrogen, and the resulting metal converted into the dioxide by means of nitric acid and weighed. The residue of titanic oxide on the filter, together with the filter paper, is ignited and fused with 10 times its own weight of potassium carbonate. The fused mass is next treated with about 200 cc. of water, and concentrated sulphuric acid is added drop by drop until the acid potassium titanate is dissolved, the solution then being neutralised with sodium carbonate; finally 2 grms. of concentrated sulphuric acid are added, the whole made up to 400 cc. and boiled for six hours. Titanic acid is precipitated, and is washed, dried, and weighed as the dioxide. The accuracy of the method is confirmed by a series of analyses given. To apply it to silicates the following method of procedure has been adopted as the result of many trials:—5—10 grms. of the mineral are decomposed by strong hydrofluoric acid, and the resulting solution, after neutralisation with sodium or potassium hydrate, treated with 2 cc. of sulphuric acid (conc.) and boiled for six hours after dilution to 400 cc. The hydrated oxides of tin and titanium are completely precipitated and can be separated as above described. Should any iron remain with the titanic oxide it can be readily removed by repeating the reduction in hydrogen and treatment with hydrochloric acid.

—C. A. K.

#### *Tests for Phenacetin.* S. Lüttke. Pharm. C. H. N. F. 1890, 65.

THE ortho-compound of phenacetin, diamidophenols, and diamidophenetols may occur as impurities in phenacetin. To determine the presence of the ortho-compound, 15 grms. are boiled with 25 grms. of dilute hydrochloric acid. Amidophenetol hydrochloride is formed, which may be separated with caustic soda, and its boiling-point (242.5°) determined. The hydrochloride of amidophenetol gives, with ferric chloride, a blood-red colouration. To detect the presence of diamido-compounds, 0.5 gm. of bleaching powder is ground with hydrochloric acid to a thin paste, and a few centigrams. of powdered phenacetin added. A red colouration indicates the presence of diamido-compounds.

—J. B. C.

#### *Tests for Quinine.* E. Hirschsohn. Pharm. Zeit. Russl. 1890, 1.

IN order to test the purity of quinine sulphate, 0.2 gm. of that salt is shaken with 5 cc. of a mixture of 30 vols. of petroleum ether (sp. gr. 0.680) and 70 vols. of chloroform, and filtered. To the clear filtrate 3 vols. of petroleum ether

are added. With pure quinine sulphate the liquid remains clear, whereas in the presence of any other alkaloid an opalescence or precipitate is formed.—J. B. C.

*Method of distinguishing Exalgin from Antifebrin and Phenacetin.* E. Hirschsohn. Pharm. Zeit. Russl. 1890, 17.

METHYLACETANILIDE, or exalgin, may be readily distinguished from antifebrin and phenacetin by its greater solubility in chloroform.

One grm. of exalgin dissolves completely in 2 cc. of chloroform in the cold. Antifebrin and phenacetin remain undissolved.

The two latter may be distinguished by their behaviour towards bromine water. Antifebrin yields crystalline acetparabromanilide. In the case of phenacetin, no separation is effected. The use of chloroform may also be used to detect the presence of 10 per cent. of phenacetin or 20 per cent. of antifebrin in a sample of exalgin.—J. B. C.

*The Use of Mercuric Nitrate containing Nitrous Acid as a Reagent for Phenols.* L. C. Plugge. Arch. Pharm. 1890, 9.

THE above reagent gives with a dilute solution of phenol a violet-red colour. This reaction is not limited to phenol, but occurs also with other hydroxy-derivatives of benzene. The position of the hydroxyl group with respect to other side chains appears to have no influence.—J. B. C.

*New Reaction for Nitrogen in Organic Compounds.* E. Donath. Chem. Zeit. 14, 157—158.

IT is known that organic substances containing nitrogen suffer decomposition more or less severely when heated with concentrated sulphuric acid. If, as in the case of Kjeldahl's method, a powerful oxidising agent is present at the same time, combustion of the carbon and part of the hydrogen ensues, whilst the nitrogen atoms present, so to speak, in a nascent state, combine with an equivalent number of hydrogen atoms to form ammonia. By reversing this reaction and effecting the decomposition by means of an active base in the presence of a powerful oxidising agent, it may be inferred that the nitrogen would combine with the oxygen to form an acid. Upon this assumption the author has made an extensive series of determinations with a large number of substances representing the most important groups of organic nitrogenous compounds. In each case nitrous acid was the product of the reaction, a result which leaves but little doubt as to the general applicability of this new reaction.—D. B.

*Pyridine Bases and Wood Fibre.* A. Ihl. Chem. Zeit. 14, 304.

THE author has examined the action of various pyridine bases, occurring in animal oil, on wood. Wood, moistened with an alcoholic solution of pyrrol ( $C_4H_5N$ ), and then with hydrochloric acid, assumes an intense carmine-red colour, which gradually deepens in tint. Paper containing wood cellulose gives the same reaction. Pyridine,  $C_5H_5N$ , under the same circumstances colours wood a pale pink. Picoline,  $C_5H_7(CH_3)N$ , gives, after some time, a slight yellow colour. Lutidine,  $C_6H_9(CH_3)_2N$ , has the same effect as picoline. Collidine could not be obtained, but it is present in commercial "pyridine bases," and is a colourless fluid. Wood, treated with an alcoholic solution of this fluid, followed by hydrochloric acid, becomes after some time of an orange-yellow colour. Collidine itself, therefore, can only produce a more or less yellow tint. The red colour which animal oil and tobacco juice impart to wood can thus be only ascribed

to the presence of pyrrol. During the dry distillation of albuminoids and kindred bodies pyrrol is always produced, and may be readily detected by its action on wood.

—H. T. P.

*The Colour Reactions of Carbon Compounds. Pyrrol Colours producible on the Fibre.* A. Ihl. Chem. Zeit. 14, 348.

THE author has observed the behaviour of various allyl compounds with phloroglucinol and pyrrol.

Dilute allyl alcohol boiled with phloroglucinol and a large excess of hydrochloric acid, assumes after some time a yellowish-red colour. Strong sulphuric acid acts on strong and weak solutions of allyl alcohol in the same way that it acts on saccharose. Allylsulphide, diluted with alcohol, yields, on warming with phloroglucinol and an excess of hydrochloric acid, a red colouration. Allyl mustard-oil, similarly treated, gives already in the cold a pale red colouration, which deepens on warming. Allylsulphide treated with an alcoholic solution of pyrrol, and strong hydrochloric acid, develops a cochineal-red colour, whilst allyl mustard-oil under similar circumstances assumes a paler red tint. Bitter almond-oil, dissolved in alcohol and mixed with an alcoholic pyrrol solution and hydrochloric acid, deposits a white precipitate, which rapidly turns grey, pale red, and finally bright carmine-red. The precipitate is a condensation product of pyrrol and benzaldehyde. Cotton cloth, dipped in a weak alcoholic solution of bitter almond-oil, and then treated with alcoholic pyrrol solution, ferric chloride, and hydrochloric acid, and gently warmed, is coloured red. White tissues similarly treated with oil of cinnamon, pyrrol, ferric chloride, and hydrochloric acid, are dyed a deep black.—H. T. P.

*A New Reaction for Tannin.* C. Böttger. Ann. 256, 341—344.

TANNIN reacts with phenylhydrazine, though not forming so elegant a test as gallic acid does. The substances are boiled together in aqueous solution, in the proportion by weight of 2:1, or better 1:1, or are heated together at 100° directly, with exclusion of water, in the proportion of 1:2, with subsequent treatment with water and heating to boiling. In either case solution is effected. Carbon dioxide escapes, and there are formed substances of the nature of colouring matters, which are either insoluble or with difficulty soluble in water, and evidently the result of a secondary reaction. Some of these substances are easily soluble, the rest insoluble in ether. The latter are also not completely soluble in caustic soda solution; the alcoholic-etheral solution of the former is quickly decolourised by zinc dust and a few drops of hydrochloric acid.

The principal product of the reaction described, is itself a mixture of several bodies. It is soluble in ether and in hot water, and possesses the property, on cautious treatment with caustic soda, of yielding a splendid green-blue solution, gradually turning yellow.

To obtain this reaction it is only necessary to boil the solution of the tannin for a little time with phenylhydrazine. Neither gallic acid nor pyrogallol give this reaction.

The author next describes a method for isolating the substance which gives the blue colour.

Gallic acid-phenylhydrazide, dissolves in caustic soda solution, giving a reddish-yellow, orange, or golden colouration.

*The Estimation of Tartaric Acid in the Crude Products of Tartaric Acid Manufacture.* P. Telbisz. Chem. Zeit. 14, 347.

TÓTH's recent work on the estimation of tartaric acid in raw materials (this Journal, 1890, 420—421) by the Goldenberg-Geromont, Goldenberg-Geromont modified, and Lorenz methods; but especially the conclusion derivable from Tóth's results, that the largely-used and modified Golden-

berg-Geromont method is unsatisfactory, induced the author to estimate the tartaric acid in a number of samples of crude calcium tartrate and wine-lees by the above methods. The following results were obtained :—

## CALCIUM TARTRATES.

No. of Sample.	Tartar according to the Goldenberg-Geromont Method.	Differences.	Tartar by the Modified Goldenberg-Geromont Method.	Differences.	Tartar by the Lorenz Method.	Differences.
	Per Cent.		Per Cent.		Per Cent.	
1	59'00 } 59'40 } 58'80 }	0'60	59'10 } 59'30 } 59'30 }	0'20	60'80 } 60'70 }	0'10
2	53'20 } 53'40 }	0'20	52'17 } 52'31 }	0'14	56'03 } 56'22 }	0'19
3	63'60 } 63'80 }	0'20	62'45 } 62'62 }	0'17	64'87 } 64'87 }	0'06
4	56'60 } 57'00 }	0'10	54'84 } 55'26 }	0'52	56'91 } 56'56 }	0'35
5	59'60 } 59'20 }	0'20	56'39 } 56'22 }	0'17	60'55 } 60'89 }	0'34

## DRY WINE LEES.

No. of Sample.	Tartar by the Goldenberg-Geromont Method.	Differences.	Tartar by the Modified Goldenberg-Geromont Method.	Differences.	Tartar by the Lorenz Method.	Differences.
	Per Cent.		Per Cent.		Per Cent.	
1	25'00 } 23'40 }	0'40	22'54 } 22'36 } 22'54 }	0'18	25'95 } 25'46 }	0'52
2	30'10 } 30'40 }	0'30	28'45 } 28'22 }	0'23	30'44 } 29'95 }	0'49

According to Wolfmann, the results obtained by the second of these methods agree best with the yield of tartar obtained on the large scale. The author is, therefore, of opinion that the modified Goldenberg-Geromont method is, for the present, at least, the most accurate. The results also show that differences of 7—10 per cent. between the results of different analyses of the same sample, as were found by Tóth, should never occur.—H. T. P.

*Volumetric Estimation of Tannin in Wines.* L. Roos, Cusson, and Giraud. *J. Pharm. Chim.* 1890, **21**, 59.

THE reagent used is a 10 per cent. tartaric acid solution to which ammonia is added until faintly alkaline. Neutral lead acetate is now added until the precipitate no longer redissolves, when the solution is filtered.

This solution precipitates tannin completely from its solutions. To 25 cc. of a tannin solution, which contains 5 grms. to the litre, 4—5 drops of ammonia, and for a preliminary estimation 2 cc. of the lead solution are added at a time. A drop of the solution is placed after each addition on paper, and a drop of sodium sulphide solution added. When a brown colouration is produced the end of the reaction is attained. In the case of wines 25 cc. are employed, made faintly alkaline with ammonia, and the operation conducted as above.—J. B. C.

*Quantitative Estimation of Codeine and Morphine.* E. Claassen. *Pharm. Rund.* 1890, **41**. (See this Journal 1890, 423.)

IF the free alkaloid alone is present without ammonium salts or substances, which decompose ammonium chloride, the codeine is heated in a flask with ammonium chloride and the ammonia, which is set free, collected in standard hydrochloric acid solution and titrated. 100 parts of  $\text{Nl}_3$  correspond to 1758'8 parts of anhydrous codeine. Or the following method may be adopted:—The liquid is acidified with dilute sulphuric acid and evaporated with excess of magnesia to dryness. The residue is extracted with alcohol, the latter distilled off and the residue treated as above with ammonium chloride. The residual liquid in the flask is acidified with hydrochloric acid and morphine, precipitated by the slightest possible excess of ammonia and weighed on a filter paper. As the codeine remains in the solution after extracting the morphine, the solution may be evaporated with magnesia and the codeine extracted with ether. When the ether has been driven off, morphine sulphate is added, and then from the amount of precipitated morphine that of the codeine may be calculated.—J. B. C.

*Estimation of Nitrogen in Organic Substances by use of Alkaline Potassium Permanganate.* R. L. Wagner. *Chem. Zeit.* **14**, 269.

THE communication by E. Donath (page 555) has led the author to communicate the following earlier research:—In the winter session 1885—86, in the laboratory of the



Leipzig Agricultural Institute, he estimated nitrogen in organic substances by the use of an alkaline solution of potassium permanganate. A note of previous publications of others on the subject is given. The substance and the alkaline solution of potassium permanganate were enclosed in a sealed tube 20—30 cm. long. There being a large evolution of oxygen with very oxidisable bodies, one end of the tube terminated in a short capillary tube. The tubes were heated in a sloping oven, the temperature being maintained at  $150^{\circ}$ — $170^{\circ}$  for 2—2½ hours. The substances were previously intimately mixed by heating on a water-bath and shaking vigorously. At the end of the operation the glass tube was opened, and the contents transferred to a porcelain basin. The contents were then reduced by heating with the requisite quantity of manganese sulphate solution and sodium carbonate. The filtrate from the manganese precipitate was washed with warm dilute soda solution, and concentrated in a 200—250 cc. flask over a small flame. The potassium nitrate was then estimated by the method of Eder (Zeits. Anal. Chem. 1877, 16).

In these experiments, 0·65 to 0·1 gm. of substance was mixed with 25—30 times the amount of potassium permanganate, and 5 cc. of solution containing 25 per cent. of pure potassium hydrate.

When too little time is allowed for the oxidation, ammonia and nitrite remain, and loss is shown on estimation of the nitrate.

Many bodies in which the nitrogen is combined with carbon or hydrogen being insoluble in solutions of salts and alkalis, could only be incompletely oxidised. This process is of practical value only for pure nitro-derivatives and nitric ethers. Those which are not volatile and are soluble in alkaline solution can be treated with the alkaline permanganate in a porcelain basin.

It is also a good plan to reduce the excess of permanganate with alcohol, and then to convert the nitrogen as nitrate or nitrite into ammonia. In order to do this the whole filtrate, about 100—150 cc., are placed in a 600—750 cc. round flask, 2 grms. of dissolved ferrous sulphate and about 1—2 grms. of zinc dust are added and agitated, and then neutralised with hydrochloric acid. The nitrogen acids are, on gentle warming, converted into ammonia in a few minutes, and this can be estimated by means of decinormal acid in the usual way.

The following are some of the author's results:—

	Calculated.	Found.
1. Calculated from nitric acid found—	Per Cent.	Per Cent.
Ammonium sulphate $(\text{NH}_4)_2\text{SO}_4$ .....	21·20	20·80
Asparagin $\text{C}_4\text{H}_8\text{N}_2\text{O}_3 + \text{H}_2\text{O}$ .....	18·66	18·51
Leucine $\text{C}_6\text{H}_{10}(\text{NH}_2)\text{COOH}$ .....	10·71	10·60
Caffeine $\text{C}_8\text{H}_{10}\text{N}_4\text{O}_2 + \text{H}_2\text{O}$ .....	26·44	26·31
Potassium ferrocyanide $\text{K}_4\text{FeC}_6\text{N}_6 + 3 \text{H}_2\text{O}$ .....	19·93	19·83
Quinine $\text{C}_{20}\text{H}_{24}\text{N}_2\text{O}_5$ , dried at $110^{\circ}$ .....	8·67	8·75
Brucine cryst. $\text{C}_{23}\text{H}_{26}\text{N}_2\text{O}_4 + 4 \text{H}_2\text{O}$ .....	6·01	6·12
Trinitrophenol $\text{C}_6\text{H}_3(\text{NO}_2)_3\text{OH}$ .....	18·34	18·30
Nitrobenzene $\text{C}_6\text{H}_5\text{NO}_2$ .....	11·38	11·42
White of egg .....	15·65	14·42
Uric acid $\text{C}_5\text{H}_4\text{N}_4\text{O}_3$ .....	33·40	30·20
Aniline hydrochloride $\text{C}_6\text{H}_5\text{NH}_2\text{Cl}$ .....	10·81	7·60
2. From ammonia found—		
<i>o</i> -Nitrophenol $\text{C}_6\text{H}_4(\text{NO}_2)\text{OH}$ .....	10·07	10·01
<i>p</i> -Dinitroresol $\text{C}_6\text{H}_3(\text{NO}_2)_2\text{CH}_3\text{OH}$ .....	14·14	14·10
<i>o</i> -Nitrobenzoic acid $\text{C}_6\text{H}_4(\text{NO}_2)\text{COOH}$ ...	8·38	8·27
Collodion .....	..	10·62
Gun-cotton .....	..	13·11

These last were estimated in the autumn of 1887. The oxidation was effected in a porcelain basin on the water-bath.

The author also estimated the sulphur contained in carbon bisulphide and in thiophen.

0·05 to 0·1 gm. of each liquid was enclosed in a very thin glass bulb which was broken by a strong shake when enclosed and sealed in the glass tube.

	Calculated.	Found.
Carbon disulphide $\text{CS}_2$ .....	Per Cent. 81·2	Per Cent. Sulphur. 81·08
Thiophen $\text{C}_4\text{H}_4\text{S}$ .....	38·09	38·16

—D. A. S.

*A New Method of Estimating Alcohol by Means of Chromic Acid.* R. Bourcart. Bull. Ind. Soc. Mulhouse, 1889, 558—562.

THE method of determining alcohol, previously described by the author, in which an oxidising mixture of potassium permanganate and strong sulphuric acid was used, is liable to give erroneous results, due partly to the difficulty of controlling the oxidation and partly to the spontaneous decomposition of the mixture when heated above  $50^{\circ}$ , or when kept any length of time before dilution with water. These drawbacks are not met with when a mixture of dilute solutions of potassium bichromate and sulphuric acid is employed. Such a mixture can be boiled for a long time without decomposition.

The determination of the alcohol is made by heating a small quantity of the sample with an excess of the oxidising mixture in a sealed tube, or more conveniently, to save the trouble of sealing and opening, in a well-corked tube, weighted or tied down, the tube being first shaken to mix the contents, and then heated on the water-bath for two or three hours. After cooling, the contents of the tube are collected in a flask, the tube rinsed out with water, and the excess of potassium bichromate determined with potassium iodide, sodium thiosulphate, and starch-paste. Experiments were made under slightly varying conditions, with 10 cc. of a dilute solution of alcohol, obtained by diluting with 200 volumes of water, 1 volume of alcohol of sp. gr. 0·7997 at  $15^{\circ}5'$ , containing 98·8 per cent. by volume according to Traill's tables. Very regular results were obtained, the mean assigning to the alcohol a percentage by volume of 97·3086. In most of the trial experiments there were used for 10 cc. of the dilute alcohol, 50 cc. of a 0·5 solution of potassium bichromate, and 10 cc. of 25 per cent. sulphuric acid; after heating, 10 cc. of a 10 per cent. solution of potassium iodide were added, then the liquid was titrated with sodium thiosulphate till its dirty-yellow colour changed to a green-yellow, when the starch-paste was added, and the titration was continued till the purple colour had disappeared, leaving the liquid a pale blue, due to the presence of chrome alum.

Estimating aldehyde in a similar way and calculating from the basis of three molecules of aldehyde being oxidised to acetic acid by one molecule of potassium bichromate, there were found respectively in two experiments, 1·2603 and 1·2549 grms., the amount taken being 1·2424 gm. of paraldehyde.

It was further ascertained that the oxidising mixture had no influence on acetic acid, the full amount of potassium bichromate used being found unchanged after prolonged heating in the tube.—E. B.

*Method for Detecting and Estimating Petroleum in Oil of Turpentine.* W. M. Burton. Amer. Chem. J. 12, 102—104.

REFERRING to Armstrong's method for the detection of petroleum in turpentine oil, which depends on the ready and nearly complete polymerisation of oil of turpentine by

sulphuric acid, and the comparative indifference to such treatment of petroleum, the author finds that this process does not merely take up much time and labour, but the yield of petroleum is far from being quantitative, leaving the operator in the dark as to the extent of the adulteration.

The method proposed by the author is based on the fact that petroleum is only slightly affected by fuming nitric acid in the cold, whilst oil of turpentine is readily oxidised to various acids of the fatty and aromatic series which are soluble in hot water. The apparatus necessary for effecting this separation consists of a balloon flask of 750 cc. capacity, which is fitted with a two-holed cork stopper. Through one hole is inserted the tube of a glass-stoppered drop funnel having a capacity of 100 cc. The flask is also connected with an inverted condenser. About 300 cc. of fuming nitric acid of sp. gr. 1.4 are placed in the flask, and 100 cc. of the oil of turpentine under examination are measured into the drop-funnel. The flask is surrounded by cold water, and the turpentine is allowed to drop slowly into the nitric acid. As each drop strikes the acid, violent action takes place with evolution of red fumes. It is well to shake the flask occasionally during the operation. When the turpentine oil has all passed into the flask the apparatus is allowed to stand until all action is over. The contents are transferred to a large separating funnel and treated with successive portions of hot water. In this way all the products resulting from the action of the acid on the terpenes are removed, whilst any petroleum paraffins remain insoluble in water and can be readily separated and measured.—D. B.

## New Books.

**DIE TIHERFARBEN MIT BESONDERER RÜCKSICHT AUF SCHÄDLICHKEIT UND GESETZGEBUNG HYGIENISCH UND FORENSISCH-CHEMISCH UNTERSUCHT, VON DR. TH. WEYL.** Mit einer Vorrede von PROF. DR. ECG. SELL. Berlin: Verlag von August Hirschwald. Unter den Linden, 68, N.W. London: H. Grevel and Co., 33, King Street, Covent Garden.

The whole work includes two 8vo parts, bound in pamphlet form, price 2s. 8d. each, and each complete in itself; Part I. containing its own Alphabetical Index, whilst Part II. contains a General Index. Part I. commences with Dr. Sell's introductory remarks, and these are followed by the Preface, Table of Contents, and 77 pages of Subject-matter. In Chapter I. the Coal Tar Colours are treated of, both chemically and technologically. Chapter II. deals with the Poisonous Tar Colours. Chapter III. with the laws against the Employment of Poisonous Colours. Chapter IV. Problems for Investigation. Chapter V. Method of Conducting the Investigations as to Poisonous Properties and Power.

**SPECIAL SECTION.**—Chap. I. Nitroso Colours. Chap. II. Nitro Colours.

**PART II.**—This is really a continuation of the Special Section concluding Part I., and the whole pamphlet, which comprises pages 81 to 138, and concludes with a General Alphabetical Index, is devoted to the consideration of the Subject-matter of Chapter III., which deals with the Azo-Colours. In this chapter, *first*, a chemical review is made of this class of colouring matters, their constitution, properties, and modes of employment—the textile arts, and *second*, a systematic report of the experiments tried with the representative members of the principal series of Azo-Colours. The conclusions drawn from the investigations are recorded on pages 136—137.

**YEAR BOOK OF THE SCIENTIFIC AND LEARNED SOCIETIES OF GREAT BRITAIN AND IRELAND.** Comprising Lists of the Papers read during 1889 before Societies engaged in Fourteen Departments of Research, with the names of their Authors. Compiled from Official Sources. Seventh Annual Issue. London: Chas. Griffin and Co., Exeter Street, Strand. 1890.

OCTAVO volume, bound in red cloth, and containing Preface, Synopsis of Contents, Subject-matter covering 220 pages, and an Alphabetical Index. A reference to the pages of the Year Book show that the subjects represented by the various Societies are as follows:—1. Science Generally. 2. Astronomy, Mathematics, and Physics. 3. Chemistry and Photography. 4. Geography, Geology, and Mineralogy. 5. Biology. 6. Economic Science. 7. Mechanical Science. 8. Naval and Military Science. 9. Agriculture. 10. Law. 11. Literature and History. 12. Psychology. 13. Archaeology. 14. Medicine.

**GASANALYTISCHE METHODEN.** VON DR. WALTHER HEMPEL, Professor an der Technischen Hochschule zu Dresden. Zweite Auflage. 1890. Braunschweig: Friedrich Vieweg und Sohn. London: H. Grevel and Co., 33, King Street, Covent Garden.

THIS, the second edition of Hempel's Methods of Gas Analysis, is an 8vo volume of considerably greater dimensions than the first edition. It is bound in paper cover, and the price is 11s. The work, which is illustrated with 101 excellent engravings, commences with a short preface by the author, in which the latter, whilst disclaiming any intention of going into detail on the subject of Methods of Gas Analysis, states it as his desire to make his book valuable as a laboratory guide. He has, however, made an exception in favour of Pettersson's apparatus, because the operation of this instrument involves the application of a completely new principle with regard to the measurement of gases. The text covers 364 pages, and the work terminates with an Alphabetical Index and List of Corrigenda.

The Subject-matter of the text is divided into three sections. PART I. General Methods; PART II. Special Methods; and PART III. Applications of Gas Analysis. In PART III. the subject of Chimney Gases is first dealt with, and thereafter Illuminating Gas, Water Gas, Generator Gas, and Coke-oven Gases. Then follows Photometry, with the amyl-acetate lamp of Hefner-Alteneck recommended as an instrument for furnishing the unit of light. It is claimed as its great advantage that when once regulated and lighted it continues to burn for a long time unaltered in intensity. Estimation of Specific Gravity (Bunsen and Schilling). Lux's Gas Balance. Examples given and worked out both of Illuminating and Water Gas. Estimation of Tar in Crude Gases (Tieftrunk), of Sulphuretted Hydrogen in Gas (Bunte), of Total Sulphur (Drehschmidt), and Carbonic Acid (Rüdorff).

Pages 240—246 are devoted to the methods for determining the gases employed in the Sulphuric Acid Chamber; pp. 246—285 the Gaseous Constituents of Atmospheric Air; and page 312 the Estimation of Fluorine as Fluor-silicic. The latter part of the work is devoted to important methods and apparatus for the Analysis of Nitre and Nitric Ether, Nitroglycerol, Gun-cotton, &c., and finally a method for the Estimation of the Calorific Value of Fuels.

## Trade Report.

### TARIFF CHANGES AND CUSTOMS REGULATIONS.

(From the Board of Trade Journal.)

#### ITALY.

##### Classification of Articles in Customs Tariff.

*Note.*—Lire =  $9\frac{9}{16}$ d. Quintal = 220·4 lb. avoirdupois.  
Hectogramme = 3·52 oz.

The following decisions affecting the classification of articles in the Italian Customs tariff have recently been given by the Italian Customs authorities :—

Rye-straw paste prepared with nitrates and hydrates (an explosive).—Category 51 B. Duty, 4 lire per quintal.

India-rubber, not prepared.—Category 334 A. Duty free.  
Oil of cherry laurel.—Category 61. Duty, 10 lire per quintal.

#### GREECE.

##### Modifications in Customs Duties on Imports from the United Kingdom.

*Note.*—Drachme = 100 Lepta =  $9\frac{9}{16}$ d. Oke = 2·84 lb. avoirdupois.

The following is a statement of some of the modifications (in accordance with the agreement of the 28th March 1890) in the Greek Customs tariff in favour of goods, the produce or manufacture of the United Kingdom, imported into Greece :—

*Vegetable Dyes.*—Class 59 (b).—Indigo.—Duty of 3 drachmes per oke, reduced to 10 leptas.

*Chemical Products.*—Class 103 (c).—Acids, pure, *i.e.*, sulphuric acid (vitriol), hydrochloric acid (spirits of salts), nitric acid (aqua fortis), phenic acid, and other similar acids, as well as all other chemical products not specially mentioned.—Duty of 20 leptas per oke, reduced to 10 leptas.

Class 103 (d).—Impure acids (so-called acids of commerce).—Duty of 10 leptas per oke, abolished.

#### UNITED STATES.

##### Recent Customs Decisions.

The following decisions affecting the classification of articles in the Customs tariff and the application of the Customs Law of the United States have recently been given by the United States Customs authorities :—

A solid expressed or rendered oil, obtained from some unknown vegetable source, probably the palm-nut, rapeseed, or other like substance, assimilating to cocoa butter in appearance, and the uses to which it may be applied, is dutiable at the rate of 25 per cent. *ad valorem*.

Essential oils of mustard and wintergreen are dutiable at the rate of 25 per cent. *ad valorem*.

#### CANADA.

##### Modifications in the Customs Tariff.

The following is a list of changes which have recently been made in the Customs tariff of Canada. The rearrangement of classification and the duties on the articles

enumerated below are to be substituted for those hitherto in force :—

Classification of Articles.	Rates of Duty.
	Dols. Cts.
Acid, acetic and pyroligneous, not otherwise specified, and vinegar, for each gallon of any strength not exceeding a strength of proof. . . . .	Per gall. 0·15
For each degree of strength in excess of the strength of proof an additional duty of 1 cent. The strength of proof shall be held to be equal to 6 per cent. of absolute acid, and in all cases the strength shall be determined in such manner as may be established by the Governor in Council.	
Acid, acetic and pyroligneous, of any strength, when imported by dyers, calico printers, or manufacturers of acetates or colours, for exclusive use in dyeing or printing, or for the manufacture of such acetates or colours in their own factories, under such regulations as may be established by the Governor in Council. . . . .	0·25 and 20 % <i>ad val.</i>
Acid phosphate . . . . .	Per lb. 0·03
Ferro-manganese, ferro-silicon, spiegel, steel bloom ends, and crop ends of steel rails, for the manufacture of iron or steel . . . . .	Per ton 2·00
Lead, nitrate and acetate of, not ground . . . . .	5 % <i>ad val.</i>
Dry white and red lead, orange mineral and zinc white, or carbonate of zinc. . . . .	5 % <i>ad val.</i>
Colours, dry, not otherwise specified. . . . .	20 % <i>ad val.</i>
Paints and colours, pulped or ground in oil or other liquids, not otherwise specified. . . . .	30 % <i>ad val.</i>
Paints, ground or mixed in, or with, either japan, varnish, lacquers, liquid driers, collodion, oil finish, or oil varnish; rough stuff, fillers, and all liquid, prepared or ready-mixed paints, not otherwise specified, the weight of the package to be included in the weight for duty . . . . .	Per lb. 0·05 and 25 % <i>ad val.</i>
Oxides, ochres and ochrey earths, fire proof, umbers, and siennas, ground or unground, washed or unwashed, calcined or raw. . . . .	30 % <i>ad val.</i>
Paints and colours, ground in spirits, and all spirit varnishes and lacquers. . . . .	Per gall. 1·00
Paraffin wax, stearic acid, and stearin of all kinds . . . . .	Per lb. 0·03
Photographic dry plates . . . . .	Per sq. ft. 0·09
Albuminised paper chemically prepared for photographers' use. . . . .	25 % <i>ad val.</i>
Plumbago. . . . .	25 % <i>ad val.</i>
" all manufactures of, not otherwise specified . . . . .	30 % <i>ad val.</i>
Red and yellow prussiate of potash. . . . .	10 % <i>ad val.</i>
Castille soap, mottled or white, and white soap . . . . .	Per lb. 0·02
Soap powders, pumice, silver and mineral soaps, sapollo, and other similar articles, the weight of the package to be included in the weight for duty . . . . .	" 0·03
Spirituous or alcoholic liquors distilled from any material or containing or compounded from, or with distilled spirits of any kind and any mixture thereof with water—for every gallon thereof of any strength not exceeding the strength of proof. When of a greater strength than the strength of proof, at the same rate on the quantity there would be if reduced to the strength of proof, as follows, <i>viz.</i> :—	
(a.) Ethyl alcohol or the substance commonly known as alcohol, hydrated oxide of ethyl, or spirits of wine; gin of all kinds not otherwise specified; rum, whisky, and all spirituuous or alcoholic liquors not otherwise provided for. . . . .	Per gall. 1·75
(b.) Amyl alcohol or fusel oil, or any substance known as potato spirit or potato oil . . . . .	" 2·00

Classification of Articles.	Rates of Duty.
<b>Spirituans and alcoholic liquors, &amp;c.—<i>cont.</i></b>	<b>Dols. Cts.</b>
(c.) Methyl alcohol, wood alcohol, wood naphtha, pyroxylic spirit, or any substance known as wood spirit or methylated spirit; absinthe, arrack, or palm spirit, brandy, including artificial brandy and imitations of brandy; cordials and liqueurs of all kinds not otherwise specified; ginger wine, mescal, pulque, rum, shrub, schiedam, and other schnapps; tafia, angostura, and similar alcoholic bitters or beverages.....	Per gall. 2'00
(d.) Spirits and strong waters of any kind mixed with any ingredient or ingredients and being, or known, or designated, as anodynes, elixirs, essences, extracts, lotions, tinctures, or medicines not otherwise specified.....	2'00 and 30 % <i>ad val.</i>
(e.) Alcoholic perfumes and perfumed spirits, bay rum, cologne and lavender waters, hair, tooth, and skin washes and other toilet preparations containing spirits of any kind, when in bottles or flasks weighing not more than 4 oz. each	50 % <i>ad val.</i>
Do., do., in bottles, flasks, or other packages weighing more than 4 oz. each.	Per gall. 2'00 and 40 % <i>ad val.</i>
(f.) Nitrous ether, sweet spirits of nitre, and aromatic spirits of ammonia.....	Per gall. 2'00 and 30 % <i>ad val.</i>
(h.) In all cases where the strength of any of the foregoing articles cannot be correctly ascertained by the direct application of the hydrometer, it shall be ascertained by the distillation of a sample, or in such other manner as the Minister of Customs may direct.	
Starch, including farina, corn starch, or flour, and all preparations having the qualities of starch, not sweetened or flavoured.....	Per lb. 0'02
Do., sweetened or flavoured.....	" 0'04
Saccharin or any product containing over one-half of 1 per cent. thereof.....	" 10'00
Illuminating oils composed wholly or in part of the products of petroleum, coal, shale, or lignite, costing more than 30 cents per gallon	25 % <i>ad val.</i>
Sulphuric ether.....	Per lb. 0'05
Yeast cakes, compressed yeast and baking powders in packages of 1 lb. and over or in bulk.....	" 0'06

The following articles are free of duty:—

Alkanet root, crude, crushed or ground.	Extracts of logwood, fustic, and oak bark.
Aloes, ground or unground.	Foot-grease, being the refuse of cotton seed after the oil has been pressed out, but not when treated with alkalis.
Alum, in bulk only, ground or unground.	Gas coke (the product of gas works), when used in Canadian manufactures only.
Aluminium, or aluminium and alumina and chloride of aluminium or chloralum, sulphate of alumina, and alum cake.	Grease, rough, the refuse of animal fat, for the manufacture of soap only.
Aniline dyes and coal-tar dyes, in bulk or packages of not less than one pound weight, including alizarin and artificial alizarin.	Gums, viz.:—Amber, Arabic, Australian, copal, damar, kauri, mastic, sandarac, senegal, shellac; and white shellac, in gum or flake, for manufacturing purposes; and gum tragacanth, gum gedda, and gum barberry.
Aniline salts and arseniate of aniline.	Indigo auxiliary or zinc dust.
Antimony, not ground, pulverised or otherwise manufactured.	Kryolite or cryolite mineral.
Ashes, pot and pearl, in packages of not less than 25 lb. weight.	Litharge not ground.
Asphalt or asphaltum and bone pitch, crude only.	Mineralogical specimens.
Azeal or argols, crude only.	Oils, viz.:—Cocoanut and palm, in their natural state.
Beans, viz.:—Tonquin, vanilla, and nux vomica, crude only.	Otto or attar of roses and oil of roses.
Bismuth, metallic, in its natural state.	Pelts, raw.
Borax, ground or unground, in bulk only.	Pipe clay, unmanufactured.
Celluloid or xylonite in sheets, and in lumps, blocks, or balls in the rough.	Platinum wire; and retorts, pans, condensers, tubing and pipe made of platinum, when imported by manufacturers of sulphuric acid for use in their works in the manufacture or concentration of sulphuric acid.
Colours, metallic, viz.:—Oxides of cobalt, zinc, and tin, not otherwise specified.	

Rags of cotton, linen, jute, hemp, and woollen, paper waste or clippings, and waste of any kind except mineral waste.  
Resin or rosin in packages of not less than 100 lb.  
Roots, medicinal, viz.:—Aconite, calumba, ipecacuanha, sarsaparilla, squills, turaxacum, rhubarb, and valerian.  
Rubber, crude.  
Soda, sulphate of, crude, known as salt cake, for manufacturing purposes only.  
Soda ash, caustic soda in drums; silicate of soda in crystals only; bichromate of soda, nitrate of soda or cubic nitre, salsoda; sulphide of sodium, arseniate, binarseniate, chloride and stannate of soda for manufacturing purposes only.  
Sulphate of iron (copperas) and sulphate of copper (blue vitriol).  
Terra japonica or gambier.  
Ultramarine blue, dry or in pulp.  
Whiting or whitening, gilders' whiting, and Paris white.  
Camwood and sumac for dyeing or tanning purposes when not further manufactured than crushed or ground.  
Blood albumen, tannic acid, tartar emetic, and grey tartar, when imported by the manufacturers of cotton and woollen goods for use in their factories only.  
Chloride of potash in crystals, when imported for manufacturing purposes only.

#### INCREASE OF THE DUTY ON ALCOHOLIC MEDICINES, &c. IN INDIA.

By order of the Governor-General of British India, the following increase, taking effect from March 21st last, has been made in the duty on spirits (including alcoholic, perfumed, and medicinal preparations) imported into the country:—*A.* Spirit in a proportion to 20 per cent. and upwards, from five rupees to six rupees per imperial gallon or six quart bottles, of the strength of London proof. The duty to be increased or reduced in proportion as the strength of the spirit exceeds or is less than London proof. *B.* Perfumed spirit, in wood or in bottles, per imperial gallon or six quart bottles, from seven rupees eight annas to eight rupees. As the reason for the advance in the duty, it is stated that in some provinces the rates of duty on country spirit have been raised so high that, if any further increase were made, such spirit would be unfairly handicapped in comparison with foreign alcohol. It is the policy of the Government to impose as high a duty on country spirit as it will bear, and also to discourage the import of cheap and deleterious European spirit, which will be less able, it is thought, to bear the increased duty than the better grades of alcohol. The proposed increase of duty is estimated to add nine lakhs of rupees yearly to the revenue.—*Chemist and Druggist.*

#### EXTRACTS FROM DIPLOMATIC AND CONSULAR REPORTS.

##### TURKEY IN ASIA.

##### Mines and Minerals in Trebizond.

Like all other parts of Asia Minor, the vilayet of Trebizond is very rich in minerals, and, as in all other parts of the Turkish Empire, these resources are almost entirely neglected. In the mountain ranges that traverse the southern part of the province there are some 120 known mineral deposits. The more important of these are distributed as follows:—In the sandjak of Trebizond, 21 mines of argentiferous lead, 34 of copper, 3 of lead and copper, 2 of manganese, 10 of iron, and 2 of coal; in the sandjak of Samsoon, 1 of argentiferous lead and 1 of manganese; in the sandjak of Gernushhané, 37 of argentiferous lead, 6 of copper.

None of these deposits are worked to any advantage. The lack of enterprise, difficulties of transportation, and restrictions imposed by the Government, prevent the development of these resources which might, under proper management, add so much to the wealth of the country. There is an abundance of wood, as well as the two undeveloped coal deposits referred to, for working the ores, so that the failure to develop these resources seems the more inexcusable.—*United States Consular Reports.*

## GUATEMALA.

*Matches.*

Under date of October 15th, 1889, Consul-General Hosmer reports that, although of comparatively insignificant consideration, the manufacture and supply of phosphorus matches is an important branch of modern industry and of general use among all classes of society in the civilised world. Guatemala imports annually 115,000 gross of wooden safety matches of Swedish, Danish, and German manufacture. These sell in this market for from 1·37½ dols. to 1·50 dols. per gross in Guatemalan currency, that is, about 98 cents to 1·07 dols. in United States gold. Belgium also contributes to the supply, and a small quantity is imported from the United States. These latter are of a better quality, are of coloured woods, and sell at a higher price. There are, in addition to these wooden matches, 15,000 gross of wax imported, chiefly from Italy, into Guatemala. The duty paid on the importation of matches is 4½ cents per gross on the average weight of cases of 50 gross each.

*Mica.*

Consul-General Hosmer, under date of December 6th, 1889, reports that large beds of mica are to be found in the Republic of Guatemala at 15 leagues distant from Guatemala City, the capital. They are said to extend some two or three miles of continuous length, and are capable of being cut into elastic plates of extreme thinness, excellent for commercial purpose.

There being no occasion for the use of stoves in this climate to obtain artificial warmth, and the cooking being done either upon a primitive arrangement for burning charcoal or in ranges built of bricks and adobe, the employment of mica for home consumption is rendered unnecessary, and therefore the beds of mica have been as yet untouched.

Reports upon this mineral in North Carolina show that the article in question is worth 450 dols. per ton, and that its refuse is good as a fertiliser. Such being the fact, the small cost for the labour of obtaining it in this country should make the quarrying and cutting of mica a most profitable undertaking as a matter of export. Access to the mica beds referred to is by a good cart road for some six leagues and the rest of the distance by mule-path.

*Rubber.*

Under date of October 11th, 1889, Consul-General Hosmer reports that the export of rubber from the Republic of Guatemala to the United States during the year ended June 30th, 1889, amounted to the sum of 64,170·59 dols., and the average price was 50 cents per lb. This quantity is likely to be increased in the future, owing to the stringent law enacted, and being more strictly enforced by the Government for the protection of the rubber-tree from a bleeding process to which it had been subjected by careless natives, and the probability of planting on the part of intended colonists. Inasmuch, however, as from 15 to 20 years are required to obtain a profitable result from planting the rubber-tree, it would not hold out a favourable inducement to the agriculturist, unless planted in connexion with the banana-tree, the fruit of which could be disposed of on a paying basis until the rubber reached the point of profitable production.—*Ibid.*

## TRINIDAD.

*Asphaltum.*

Under date of November 10th, 1889, Consul Sawyer reports that the shipments of asphaltum from Trinidad—the greater part to the United States—from January 1st to June 30th, 1889, amounted to 32,460 tons, being a prodigious increase over any previous semi-annual shipments during the history of the colony.—*Ibid.*

## MEXICO.

*Cinnabar in Sonora.*

Under date of September 10th, 1889, Consul Willard, of Guaymas, reports that within the past six months a deposit of cinnabar (quicksilver ore) was discovered in his consular district (Sonora), about 150 miles from Guaymas, in the Ures district. The deposits are being worked, and are reported to be extensive—one yielding 30 per cent., while as high as 50 per cent. has been found. Twenty flasks of quicksilver have been extracted. The owners of the five claims or mines located are developing their grounds with the object of proving their value. This is the first cinnabar located and worked in Sonora.—*Ibid.*

## RUSSIA.

*Chemical Industry in Odessa.*

The manufacturing chemical industry in Odessa is a growing and prosperous one. It now embraces the manufacture of acids, sulphates, oils from exotic seeds, glycerin, and soaps. The principal customers for the Odessa soap are found in the immediate neighbourhood of Odessa, throughout Southern Russia, the Crimea, the Caucasus, and the Black Sea littoral. A little goes to Turkey, and twice yearly Russian steamers take out from 10,000 to 12,000 poods of washing soap, small quantities of toilet soap, engine grease, and caustic soda to Wladiwostok, in Siberia. Business, however, is much impeded by the continual variations in the price of the rouble, which affects specially all transactions with foreign countries.

*Soap-making.*

The raw material used here for soap-making is principally mutton tallow, but the price of tallow is steadily advancing, owing partly to the decadence of sheep-farming in Southern Russia and to the increased local outlet for tallow in the manufacture of butterine and stearin candles, and the soap-makers are therefore beginning to fall back upon cocoa-nut and palm oils.

The attempt has also been made to introduce Australian tallow. Oils are all purchased through London, and a Swiss firm also competes. Marseilles oil firms are also endeavouring to gain a footing, but their success is limited, as the Marseilles oils, though cheaper, are much inferior to Ceylon cocoa-nut oil in quality.

*Caustic Soda.*

The consumption of caustic soda amounts to about 9,000 drums (1,800 tons) yearly. All this is of British manufacture, and is exclusive of the enormous quantities going direct to the Russian petroleum fields. The caustic soda used here is from 70° to 74° in strength, recently 76 and 77 per cent.

*Soda Ash.*

Soda has also been introduced. Since 1887 a large number of soapworks have been erected here, who now manufacture soda crystals as a by-product, and are superseding the English crystals, which used to be taken here. Soda ash also was formerly bought in England, but this has all ceased now.—*Chemist and Druggist.*

## SPANISH POSSESSIONS.

*Drugs for Cuba.*

Trade returns are not compiled very quickly in Spanish America, and so it is that we are only now in possession of the commercial statistics for Cuba of 1887. In that year 70,852 kilos. of pharmaceutical products were imported into the island against only 10,525 kilos. in 1886; the imports of soap were 423,658 against 534,257 kilos.; and of chemicals 72,988 against 75,367.

*Mineral Waters.*

There is a large demand in Cuba for mineral waters, especially Vichy water.

*Perfumery.*

The consumption of fine perfumery is growing every year, and France (according to her Consul) in this department comes first, with the rest nowhere. Some French firms find it a good plan to send over boxes of assorted bottles of scents, which have a particularly rapid sale. The imports in 1887 were 12,325 kilos, against 4,783 kilos, in 1886. Until quite recently the British manufacturing perfumers did not compete on the Cuban markets, but now English samples are beginning to be shown in pharmacies and bazaars.—*Ibid.*

## TURKEY IN ASIA.

*Drugs for Smyrna.*

The principal pharmacies of Smyrna are owned by British subjects, and it is, therefore, but natural that the greater part of the medicines consumed should come from England. Certain specialties, such as antipyrin and quinine preparations, however, are brought from Höchst and Stuttgart, in Germany. So-called house medicines are very largely bought in Paris, because the manufacturers pay great attention to neat and tasteful packing, and the population here are now quite accustomed to these neat preparations. The use of raw drugs is very great in Smyrna, and this is explained to some extent by the habits of the natives, who in illness prefer to make up their own remedies from the raw material. The wholesale drug and spice business is in the hands of a few very well established firms, who purchase in London, Marseilles, Stuttgart, and Trieste. The principal drugs consumed in this place are camphor, saffron, manna, coriander, star-anise, balsams, alum, rhubarb, jalap, sarsa, castor oil, tamarinds, pimento, and nutmegs.

*Soap.*

The finer toilet soaps and perfumery are drawn from France and sometimes also from Southern Germany. A very cheap soap, in 1-kilo. bars, selling here at 10s. per cwt., is imported from England, and is exceedingly popular.

*Chemicals in Smyrna.*

Potash is the largest article of consumption among the heavy chemicals, about 3,000 2-cwt. casks being consumed annually. The principal use of the article is for drying raisins, for which purpose it is dissolved and mixed with a little oil. Formerly, the potash came *via* Trieste, and this variety was much liked, but the Austrian railways raised their tariff to such a rate that Cologne has quite usurped the potash trade. Caustic soda (40 to 45 per cent.), for soap-making, is bought exclusively in England, about 8,000 casks a year, and the small quantity of carbonate of soda used here is made by the Solvay and Leblanc works in Northern France, and exported *via* Antwerp.—*Ibid.*

## MISCELLANEOUS TRADE NOTICES.

## BRITISH TRADE IN SERBIA.

The Skupstschina has passed the final reading of a Bill for granting a monopoly of the petroleum and other oil resources of the kingdom to a syndicate of London financiers. The monopoly is for 15 years, and in order to encourage the concessionnaires the Government have granted them immunity from import and export duties for 10 years, and from internal taxes for eight years. In political circles here this concession is regarded as the most important departure in commercial policy which has been taken by the Government for a very long time. Hitherto Serbian trade has depended entirely on Austrian markets; but since the opening of the Salonica railway an opportunity has been afforded the Serbian merchants of exporting direct to their foreign clients. It is hoped that the new concession may be the means of opening relations with English markets.—*Daily Graphic.*

## PROSPECTS OF THE IRON ORE SUPPLY.

The importation of iron ore into the United States shows remarkable fluctuations, which are not to be accounted for entirely by variations in the demand in this country or the condition of its iron business, but must, to some extent, be due to fluctuations in cost of mining and in the demand for ore in other countries than our own. Here are the figures:—

## IMPORTS OF ORE DURING THE FISCAL YEARS, ENDING JUNE 30.

Imported from	1885.	1886.	1887.	1888.	1889.
France and African Mediterranean ports	25,360	107,366	215,760	163,496	78,137
Italy .....	16,213	20,982	114,123	98,343	66,812
England .....	11,890	15,033	72,546	56,090	29,216
Canada .....	17,860	7,906	18,430	13,380	9,284
Greece .....	..	7,700	44,173	25,707	13,181
Spain .....	213,906	128,586	522,719	416,138	190,460
Cuba .....	28,209	51,268	109,928	117,504	225,525
Total all countries ..	425,870	701,243	1,141,774	919,614	652,032

The decline in imports during the last three years in the face of a greatly increased production of pig iron indicates that our iron mines are more than keeping pace with our blast furnaces in increase of production. Cuba is now the largest source of our foreign supply, and Spain has taken a second place. The mines in Cuba were opened only a few years ago by an American company, and they might almost be considered part of our domestic supply, as is also that from the American-owned Canadian mines. Leaving out the importation from Cuba, the total of foreign ores imported is less than 4 per cent. of our domestic production. The increasing cost of ore in Spain and other European countries indicates the probable cessation altogether of our importations from these sources, and will leave us dependent upon our own country, Cuba, and Canada. Fortunately in these countries, our near neighbours, as well as at home, there is a constant increase in the development of iron mines, and no prospect of any scarcity in either source for a long time to come.—*Engineering and Mining Journal.*

## THE TRADE OF BRITISH INDIA.

*Cinchona Bark.*

The quantity of cinchona bark exported rose from 1,286,900 lb. in 1886–87 to 1,449,313 lb. in 1887–88, and to 3,074,090 lb. in the following year. This expansion was accompanied by a largely reduced value, the result of a fall in prices which has been so heavy that cinchona has been sold below the cost of production. Production in fact has outrun consumption for the time being. Nearly the whole of the export is from Madras, the bark produced in Bengal (at least in the Government plantations) being manufactured there for consumption in India. It is now made into sulphate of quinine at a cost which is not higher than that of the imported drug, low as that is.

*Quinine.*

The very low price to which quinine has fallen has greatly stimulated its import. The fall in price has enabled quinine to more than resume its place in India, and has, for the time, slightly restricted the use of alkaloids made in the country.

*Oils.*

The importation of mineral oils has progressed in the following manner during recent years:—1883–84, 13,883,838 galls.; 1884–85, 27,306,999 galls.; 1885–86, 21,311,942 galls.; 1886–87, 31,949,633 galls.; 1887–88, 31,421,559 galls.; 1888–89, 39,951,885 galls.

Thus, the quantity imported last year was almost three times as large as the imports of six years ago. The use of mineral oil as an illuminant is now quite common in India, and is, indeed, in urban tracts, the rule rather than the exception.

These large importations of mineral oil have affected the domestic economy of the natives in two ways: the oil has displaced vegetable oils, and the tinned iron cans in which the import is made have displaced the earthen jar and the brass pot to a large extent. An importation of 30 or 40 million gallons of oil means also an importation of from six to eight million cans of 5 gallons each. These millions of cans were for some considerable time cut up by tinplate workers for the manufacture of other articles, but now they are also commonly used to hold and convey grain and other stores, water and other liquids, and they are amongst the most common objects in an Indian bazaar. They are also used as building materials in the repairs of roofs.

Russian oil, the importation of which commenced only three years ago, and which is said to be on the whole not quite equal to American oil in quality, but is appreciably cheaper, is competing with remarkable activity with American oil, as will be seen from the following figures:—

	From United States.	From Russia.
	Gallons.	Gallons.
1883—84 .....	12,715,837	..
1884—85 .....	26,026,608	..
1885—86 .....	20,229,350	..
1886—87 .....	20,141,942	1,577,392
1887—88 .....	25,040,517	5,036,055
1888—89 .....	20,653,975	17,516,003

—Board of Trade Journal.

#### PRODUCTION OF BAKU DISTILLERIES IN 1889.

The *Moniteur Officiel du Commerce* for the 27th March, quoting from *La Russie Commerciale*, gives the following statistics of the production of naphtha in the distilleries of Baku during the year 1889:—

The distilleries have received during the year a quantity amounting to 186,220,470 poods.

The consignments have been:—To foreign countries, 41,848,295 poods of naphtha product, of which 32,758,231 poods were distilled petroleum; to the home markets, 114,079,935 poods of naphtha products, of which 23,361,881 poods were distilled petroleum and 75,219,854 poods residues of naphtha; to Transcaaspia, 9,612,943 poods, 1,587,257 poods of which were naphtha residues.—*Ibid.*

#### THE EXPORTATION OF SALT FROM THE MERSEY.

The exports of salt to foreign countries from Liverpool during April amounted to 59,537 tons, against 48,536 tons during April last year, an increase of 11,001 tons. The shipments during the first five or six months of last year were equal to the average of previous years, the quantity being assisted very much by the balances of contracts at cheap prices. As the total output for the first three months of this year exceeded the output of the same period of last year, the maintenance of the foreign exports at a good average must be very reassuring to those interested in the salt trade, and who believed our English production of the article would be taken as usual abroad at prices that must leave good profits to the producers.—*Chem. Trade Journal.*

#### NITRIFICATION IN CAVES.

Caves containing deposits of earth with from 4 to 30 per cent. of calcium nitrate, and 5 to 60 per cent. of calcium phosphate, says the *American Druggist*, are common in Venezuela, not only in the littoral mountain chains, but also on the flanks of the Cordillera of the Andes. In

these deposits are imbedded remains of mammalian bones, preserving their form, but so friable as to fall to powder when they are extracted. They consist solely of calcium phosphate; the gelatin has been nitrified and dissolved out, and the calcium carbonate of the bones has been used up in neutralising the nitric acid produced. The nitric ferment is found in abundance throughout the deposits in a very well developed form. Some of these deposits are 10 metres thick.—*Ibid.*

#### CHEMICALS EXPORTED FROM THE TYNE.

The quantity of chemicals exported from the Tyne during last month shows a slight decrease in the total tonnage, as compared with the corresponding month of last year. The figures are as follow:—

	1889.	1890.
	Tons.	Tons.
Alkali and soda ash .....	1,590	1,175
Bicarbonate of soda .....	8	10
Bleaching powder .....	2,035	3,278
Manure .....	815	579
Soda crystals .....	2,482	2,701
Sulphate of soda .....	6	87
Other chemicals .....	2,877	1,957
Total tons .....	9,813	9,487

#### SUMMARY OF THE MINERAL PRODUCTION OF CANADA OF 1889.

We are indebted to the courtesy of Mr. Alfred R. C. Selwyn, Director of the Geological and Natural History Survey of Canada, for the preliminary statistical table of the mineral production of Canada for 1889. This is not the final statement, the returns not being quite complete, and is, therefore, subject to revision.

Name of Product.	Quantity (a).	Value (b).
Antimony .....	Tons 55	Dollars. 1,100
Asbestos .....	" 6,014	424,350
*Bricks .....	Thousands 190,724	1,252,667
*Building stones .....	Cub. Yds. 310,582	899,105
Cement .....	Bbls. 90,471	19,790
Charcoal .....	Bush. 1,426,800	83,573
Coal .....	Tons 2,719,469	5,570,742
Coke (c) .....	" 54,539	155,043
Copper (d) .....	Lb. 6,810,952	885,424
Feldspar .....	Tons 510	5,100
Fertilisers .....	" 775	26,606
Fire clay .....	" 490	4,800
Flag stones .....	Sq. Ft. 11,000	1,400
*Glass .....	..	150,000
Gold (e) .....	Oz. 61,739	1,116,145

\* Some returns yet to be received.

(a.) Quantity marketed, except when otherwise specified. The tons are of 2,000 lb.

(b.) Market value; less charges of transport from point of production.

(c.) Oven coke; all the production of N. S.

(d.) Copper contents of Canadian ores at 13 cents per lb. at the mine.

(e.) Nova Scotia gold is calculated at 19.50 dols. per oz., and that from British Columbia at 17.00 dols.



SUMMARY OF MINERAL PRODUCTION OF CANADA—*cont.*

Name of Product.	Quantity.	Value.
Granite..... Tons	12,535	Dollars. 78,624
Graphite..... "	220	1,630
Grindstones..... "	3,385	39,163
*Gypsum ( <i>f</i> )..... "	206,201	193,658
*Iron..... "	58,783	2,210,062
Iron ore..... "	84,181	151,640
Lead..... Lb.	164,000	5,863
*Lime..... Bush.	2,728,678	265,208
Limestone, for flux..... Tons	22,122	21,909
Manganese ore ( <i>g</i> )..... "	1,417	31,814
Marble and serpentine..... "	980	980
Mineral paints..... "	794	15,282
Mineral water..... Galls.	404,600	37,390
*Miscellaneous clay products..... "	..	239,385
Petroleum ( <i>h</i> )..... Bbls.	704,690	672,978
*Phosphate..... Tons	30,540	312,182
Pig iron..... "	25,921	499,859
Platinum..... Oz.	1,000	4,500
Pyrites..... Tons	68,047	306,212
*Salt..... "	24,716	110,367
Sand and gravel (exports)..... "	451,195	69,506
Silver..... Oz.	378,675	343,848
Slate..... Tons	6,935	119,190
Soapstone..... "	195	1,020
Steel..... "	26,333	917,822
Sulphuric acid..... Lb.	10,998,713	148,482
*Tiles..... Thousands	10,286	136,871
Estimated value of mineral products not returned (principally nickel, iron, mica, and structural materials)..... "	..	1,933,752
Total..... "	..	19,500,000

(*f*) Production of New Brunswick and Ontario, plus Nova Scotia exports.

(*g*) New Brunswick production, plus Nova Scotia exports.

(*h*) These figures are calculated from the inspection returns at 100 galls crude for 38 galls refined oil, and are computed at 954 cents per barrel of 35 imp. galls. The barrel of refined oil inspected was assumed to be 42 imp. galls.

—*Engineering and Mining Journal.*

## THE REGULATION OF THE SALE OF FERTILISERS IN GERMANY.

Under the liberal Government which France has chosen for itself, it has been thought necessary to enact special laws regulating the sale of manures.

In antioctate Germany the State has not yet turned its attention to this question, and it is through the private action of the individuals interested—both buyers and sellers—that proposals have been made for the regulation of transactions in fertilising materials.

The antithesis is sufficiently striking to give interest to some details as to the resolutions adopted by a committee composed of merchants and agriculturists.

In the United States, in France, and in Belgium, says the *Deutsche Landwirthschaftliche Presse*, from which we borrow these remarks, abuses have given rise to legislation against the delinquents.

In Germany, where there is just as much to complain of, it is beginning to be thought necessary to protect those who are the victims, but it is wished to effect this without having recourse to State intervention. It would be grand, worthy of the German spirit, said M. Schultz, to attain the end desired by a common effort, originated by the private action of manufacturers, merchants, agriculturists, and representatives of science, associated together with the view of putting the trade in fertilising materials on a secure footing throughout Germany. To succeed in such a movement, and by means independent of the public authorities, this would be a good honour for the industry and trade in fertilising materials for German scientific men in particular, for the Society of Agriculture, and, moreover, adds he, for the German nation, and for the lofty and noble spirit which animates it.

The enthusiastic appeal of M. Schultz has not fallen unheeded upon his country. The section dealing with the subject at the Magdeburg meeting appointed a special committee upon it, consisting of nine delegates, three representing agriculture, three the manufacturing interest, and three the agricultural stations. Meeting at Berlin on the 15th October last, under the presidency of Dr. Maercker, the eminent professor at Halle, this committee has decided upon the following recommendations:—

It is necessary that all sacks containing commercial fertilisers offered for sale should bear upon them in printed characters the exact designation of the manufacturer, the name of the fertiliser, and the amount and nature of valuable substances contained in it. It is also desirable that the sacks should always be of uniform weight, which might be fixed at 50, 75, or 100 kilos.; the sacks should be completely filled.

As regards the name and composition of the fertiliser, the sub-committee has arrived at the following conclusions:—

In the case of *superphosphate*, without any qualification, the amount of soluble phosphoric acid should be indicated.

When the *superphosphate* contains phosphoric acid partly soluble in water and partly insoluble, the value of which must therefore be expressed, the quantity of each of these two forms of phosphoric acid ought to be stated on the sack.

Sacks of *precipitate* should bear a statement of the total phosphoric acid, and those of *finely powdered Thomas-slag*, both of phosphoric acid and fine dust.

Sacks of *bone meal*, *fish guano*, *meat dust*, *Peru guano*, *wool dust*, &c., ought to be labelled with their contents of phosphoric acid and of nitrogen.

*Ammoniacal superphosphate*, obtained from *superphosphate* and pure ammonium sulphate, should tell the amount of soluble phosphoric acid and of nitrogen.

In the case of mixtures of *nitrogenous substances with superphosphate*, i.e., *superphosphate*, associated with organic nitrogenous matter (except leather, dust, wool, &c.), the amount of total nitrogen, and approximately the proportion of organic matter to ammoniacal nitrogen should be stated.

*Nitro superphosphates*, mixtures of *superphosphates* with Chili nitrate, should be marked with the amounts of nitrogen and soluble phosphoric acid.

Mixtures of *nitrate of soda*, *sulphate of ammonia*, and *superphosphate* should be labelled with their percentage of phosphoric acid, soluble in water, nitrate, and ammoniacal nitrogen.

*Dried blood*, *bone* and other *nitrogenous manures* should have their names and percentage of nitrogen indicated. In all mixtures of potash salts, there should be stated the percentage of potash together with that of all other valuable substances present. It would be well to arrange with the firms concerned to give the percentage of potash in kainite and other potash salts instead of, as at present, the amount of sulphate of potash contained in them.

The sampling should take place immediately upon receipt of the goods, and should be done in the presence of an agent of the seller or some impartial and competent person.

The sub-committee of the 15th October has decided to put these measures into practice until the autumn of 1890. It has also expressed a desire to see a uniform method of examining manures adopted, and has given it to be understood that a conference of delegates from the experimental

stations, with representatives of the manufacturers, would not only contribute towards this result, but also clear up the different questions raised on this subject.

Such are the commercial regulations which the German Society of Agriculture has just adopted, with the hope of ensuring integrity in the manure trade and thus rendering it more active. Small dealers will be the first to profit by them; small farmers, so often exposed to fraud against which they are unable to defend themselves, will regain confidence, and an excellent effect will shortly be produced on the progress of agriculture.—*Chem. Trade Journal*.

#### THE PHOSPHATE BEDS OF FLORIDA.

A. R. Ledoux. *Engin. and Mining J.* **49**, 175—177.

The phosphate beds occur in the marls of the Eocene and Miocene, which extend along the Atlantic coast from New Jersey to Florida. Associated with the phosphate nodules, which vary in size from a pea to masses of occasionally 100 lb. are the bones of fishes, the teeth of great sharks, the fossil horse, elephant, &c., representing many geological ages.

The phosphate deposits in South Carolina have been worked for some years, and the companies owning these deposits are said to have mined 423,720 tons in 1889. Up to last fall the river nodules were all that were known of the Florida phosphate beds and attracted comparatively little attention. A white subsoil containing 80 per cent. of phosphate of lime was then discovered, and the extent of these beds has not yet been ascertained. They lie in lenticular beds upon the limestone, and are covered by sandy soil and clay subsoil, rarely cropping out at the surface. The phosphate is found at varying depths below the surface from 2 to 10 or 12 ft. down. When wet it is dark or light yellow, but usually a dazzling white when dry. Some of the deposits are over 16 ft. thick. In the neighbourhood of Dunellen it has been estimated that 3,000 acres are underlaid with the mineral, averaging 55—60 per cent. of phosphate of lime.—J. B. C.

#### A NEW GUM.

E. Sickenberger. *Chem. Zeit.* **14**, 350.

For some time past large quantities of a gum have been sent from Bushir, on the Persian Gulf, to the small ports on the west coast of the Red Sea, for the purpose of mixing with the Kordofan Hashabi-gum. From thence the mixture is sent to Assonan, packed in old Kordofan gum cases, and sold as the best Hashabi-gum. This new gum closely resembles the best Hashabi-gum in appearance, but it is insoluble in water, in which it only swells up. According to the author it is probably a variety of the "Persian gum" of the Indian market, derived from various species of *Prunus*, perhaps from *Prunus Bokharensis* Royle and *P. Puddum Roxburgh*. It is probable that if its sale in the local market (Cairo) be stopped, other channels for its dissemination as "gum arabic" will be sought for.—H. T. P.

#### THE MEAN COMMERCIAL VALUE OF GERMAN PATENTS.

*Chem. Zeit.* **14**, 303.

It is well known that holders of German patents are confronted at the beginning of each year by the question, whether the protection granted to them is still worth the patent fees again falling due. As shown by the Patent Office, this fact renders it possible to calculate for each year the average value placed upon German patents by their holders. If the total patent fees for any one year =  $P$  marks, and the number of patents in force at the end of that year =  $n$ , then the average fee paid for each patent =  $p = \frac{P}{n}$ . Now it is perfectly admissible to consider these annual patent fees paid by patent holders as the interest on a certain capital. Assuming this interest to be at the rate of 4 per cent. the average market value of a German patent would be  $w = 25 p = 25 \frac{P}{n}$  marks.

By means of this simple calculation the following results are obtained for the last 12 years of the existence of the German Patent Office:—

Year.	Patent Fees, P Marks.	Number of Patents in Force, n	Average Annual Fee, $p = \frac{P}{n}$ Marks.	Capital Value of a Patent $w = 25 p$ Marks.
1878	265,150	4,227	62.73	1,568
1879	410,165	6,807	60.26	1,507
1880	514,525	8,007	64.26	1,607
1881	660,940	8,619	76.68	1,917
1882	787,850	9,452	83.36	2,083
1883	928,570	10,535	88.14	2,204
1884	1,058,610	10,994	96.29	2,407
1885	1,157,210	11,046	104.76	2,619
1886	1,274,940	11,249	113.34	2,834
1887	1,375,950	11,512	119.52	2,988
1888	1,472,050	11,810	124.64	3,116
1889	1,637,840	12,732	128.64	3,216

—H. T. P.

#### COMMUNICATIONS ON SEVERAL IMPORTANT DRUGS AND CHEMICALS.

*Chem. Ind.* **13**, 29—31.

*Hypnotics*.—This class has become augmented by the addition of chloralamide and somnal, but these are still only in the experimental stage. It may be noted that favourable opinions at first entertained as to the therapeutic value of new products may soon become discredited and the substances themselves relegated to obscurity.

*Iodine*.—After remaining at a selling price of 9d. per oz. for two years, it came down as low as 4d. on the 12th of August, but on the 2nd of September the price rose again to 9d. These fluctuations arose from the operations of a syndicate, but the instability of price depends largely on the uncertainty as to the extent to which the residues in the South American nitre factories may be worked up, together with the very varying requirements of the public consumption.

*Copper*.—Large transactions have lately been effected and the price had fallen from 80l. to 36l. per ton after the failure of the Société des Metaux à Paris, but has since risen to 50l. Although consumption has increased in electrical, ship building, and machinery work, it is still insufficient to account for the present high prices in view of the fact that stocks have not suffered any reduction, in fact supplies greatly exceed deliveries.

*Olive Oil*.—It was feared that much harm had been done to the fruit by worms, but the aspect of the present harvest is reassuring. It appears that such olives as were attacked by worms fell to the ground in due course, leaving the untouched olives on the trees, which have yielded an oil of excellent quality. Continuously favourable weather has resulted in imparting to the oil an exceptionally fine mild flavour, devoid of the bitterness frequently complained of. The quantity of the South Italian harvest is believed to be not much less than that of last year, but the harvest in the South of France and the Riviera has been so small as to give reason for fear that the yield will be augmented by admixture with other oils.

*Opium*.—Although the opium harvest of last year turned out to be smaller than was expected, and unfavourable reports on the condition of the new plants have come to hand, the condition of the market has remained on the whole unaffected. It appears that the consumption of Turkish opium has diminished whilst the inquiry for Persian opium has increased. The importation of foreign opium

into China has decreased in consequence of the increase in the home-grown article. Chinese opium is inferior in quality to the Indian, but is preferred for smoking as the narcotic constituents are not so quickly extracted on burning. The spread of opium-smoking in China may be seen from the fact that the Chinese production amounts to 225,000 piculs, and the imports of Indian opium amount to 75,000 piculs, whilst the yearly consumption of an accustomed smoker amounts only to one-twentieth of a picul.

**Oxalic Acid.**—The combination between the German and English manufacturers which had been kept up for several years came to an end in 1889, and considerable reduction in the price of oxalic acid and salts of sorrel may be expected. Already offers have been made by English manufacturers for delivery in this year at 30 per cent. below the present prices.

**Phosphorus.**—In consequence of the increasing French and Russian competition, the quotations for English phosphorus have been lowered occasionally during the year, but German consumption has been considerably assisted by importation from England.

**Quicksilver.**—The increasing consumption of quicksilver by the newly-established gold mines has augmented the value of this metal considerably, but the market has not been subject to the disconcerting fluctuations notable in former years. A diminishing value may, however, be looked for, as the Spanish and Russian production will probably be increased.

**Shellac.**—Shipments from Calcutta have experienced a very considerable decrease in the last year, and the increase in price would have probably been greater if the stocks in London had not been so large. The price for prime orange shellac is now 110s. per cwt., and for seconds, 75s., whilst the same qualities obtained only 65s. and 40s. per cwt. respectively in the year before.

**Sulphonal.**—Competition has reduced the price of this article to something near the limit of profitable production. The new substances named Trional and Tetronal, which stand in close relation to sulphonal, have not yet been manufactured on the large scale.

**Vanilla.**—The harvest of vanilla pods has proved inferior both in quantity and quality. This year's yield from Bourbon, Mauritius, and the Seychelles amounts to 60,000 kilos., against 80,000 kilos. in 1888 and 158,000 kilos. in 1887. As the world's needs in this article is estimated at 10,000 kilos. per annum, an increased price must be expected.—G. H. B.

#### THE FATALITIES AT THE LEEDS FORGE.

The report of Dr. Stevenson, who, at the request of the Home Secretary, attended the inquest in November last on the bodies of two men who met their death by water-gas poisoning at the Leeds Forge, has been issued. He states that it is now nearly half a century since Leblanc first announced the poisonous character of carbonic oxide gas, which is the poisonous constituent of a great number of mixed and commercial gases, such as charcoal vapours or fumes, the vapour of burning coke, the combustible gases from blast-furnaces, producer gas, forge gas, gas from regenerator furnaces, gas leaking from stove pipes, and the so-called water-gas. After describing the manufacture of water-gas, Dr. Stevenson says that it consists approximately of 40 per cent. by volume of carbonic oxide and 50 per cent. of hydrogen, the remainder being diluent gases. It is practically odourless, hence leakages of the gas cannot be detected by the sense of smell. He goes on to state that in the eight years ending 1887 the fatal cases of water-gas poisoning in the four American cities of New York, Boston, Baltimore, and Chicago, were 295. The manufacture of water-gas had, it was stated, been prohibited in the States of New Jersey and Massachusetts and in Paris. The alleged odourisation at the Leeds Forge was ineffectual. "It must be borne in mind," says Dr. Stevenson, "that safety is conferred not by simply imparting a decided and unmistakable odour to the gas, but by rendering it so far odorous that it is readily detected by smell when mixed

with at least 5,000 volumes of air. Anything short of this confers only fancied safety, so poisonous is carbonic oxide gas." In dealing with the Leeds case he simply gives a detailed account of the accident and the sensations experienced by the medical men at the *post mortem* examination, and then the inquest.—*Chem. Trade Journal*.

#### NOVA SCOTIA MINES.

The report of Mr. Edwin Gilpin, jun., Inspector of Mines for Nova Scotia, for the calendar year 1889, is issued with a relative promptitude commendable in view of the tardiness which characterises many Government statistical reports. Those of the United States, it may be remarked in passing, while not so prompt as would be desirable, are nevertheless published with more punctuality than the similar reports of several European countries. From the Nova Scotia report we abstract the following principal statistics and remarks:—

#### Total Production.

The summary of the principal mineral products for 1888 and 1889 is:—

	1888.	1889.
Gold .....	oz. 22,407	26,155
Iron ore .....	Tons 41,611	45,907
Manganese ore .....	" 88	67
Coal raised .....	" 1,776,128	1,756,279
Coke made .....	" 29,808	35,505
Gypsum exported.....	" 125,800	147,344
Barytes .....	" 1,100	None
Grindstones, &c.....	Value \$17,225	\$18,000
Moulding sand exported .....	Tons 169	179
Antimony ore exported .....	" 508	55
Limestone.....	" 15,448	19,000
Copper ore .....	" ..	500

It is to be regretted that there is no uniform system of stating quantities and values. The tons of coal and coke are stated as tons of 2,240 lb., from which the inference would be that the other tons quoted are short tons of 2,000 lb. The more important substances, with the exception of coal, show an increased output in 1889 over that of the preceding year.

#### Coal.

The total sales in 1889 were 1,555,107 tons, a slight falling off from 1888. Of these the United States took 29,986, about the same as in 1888. The remainder was sold in Quebec, Nova Scotia, New Brunswick, Newfoundland, and Prince Edward Island. Sixteen accidents in Pictou county were reported, of which two were fatal. Some trouble was caused by gas, but the system of inspection appears to be good. In Cape Breton county there were 10 accidents, of which five were fatal; one explosion of gas is reported. Close attention is being given to improved safety devices, better cables, ventilation, and prevention of fires. Under provisions of an enactment of the previous year, a number of instructors for miners' schools were appointed.

#### Gold.

The number of mines reported was 33; the mills (50) crushed 39,160 tons, an average yield of 17 dwt., 22 gr. The average earnings per man are figured at 2·22 dols. per day, or 666 dols. per year of 300 working days, not at all a bad showing. From 1862 to and including 1889 the total yield of the province has been 482,316 oz. At 18 dols. per

oz., which appears to be the average for Nova Scotia gold, this represents a total value of 8,681,688 dols. for the 21 years.

#### *Other Mineral Products.*

The greater part of the iron ore output was from London-derry. New discoveries of iron ore are reported on the East River in Pietou and at Brookfield, Colchester. The Loch Lomond manganese ore is sold on a guarantee of 90 per cent. peroxide. The barytes mines were closed during 1889. Some prospecting for lead was done in Colchester County. The small copper ore product is ascribed to the break in price of the metal.—*Engineering and Mining Journal*.

#### EXHIBITION OF APPLIANCES IN BERLIN IN RELATION TO CHEMICAL INDUSTRY.

The Exhibition was promoted for the display of methods and appliances having for their object the minimising of risk to life or limb where the circumstances include the element of danger. The author has limited his article to a notice of those means which can be employed for the protection of workers in the chemical industries, but the number of these is too large to admit of more than a general reference here. The noticed are arranged under several heads.

#### *Transport of Corrosive Liquids.*

For drawing off the contents of carboys, a rubber cap fitting over the neck is provided with bent glass tubes and a force pump, the arrangement working like that of an ordinary wash-bottle. For preventing the spirting of liquid in emptying a carboy, a rubber nose-piece having a vent hole is fitted on the neck. Instead of the usual straw packing it is proposed to surround the carboy with infusorial earth which would absorb the corrosive liquid in case of fracture, the packing being easily regenerated after an accident by washing and drying. An automatic arrangement for controlling the admission of compressed air to a montejus is described.

#### *Mineral Water Manufacture.*

All the exhibits in this department involve the use of liquid carbonic acid which avoids many of the dangers incidental to the preparation of the gas itself. The safety arrangements comprise separate expansion vessels, pressure gauges, safety-valves, and automatic pressure-reducing valves. As a provision against possible fracture of the iron cylinder containing liquid carbonic acid, it is enclosed in a strong case of iron trellis work.

#### *Varnish and Pitch Manufacture.*

To minimise the chance of firing in case the liquid should boil over the sides of the vessel, the furnace is separated from the boiling house by fire-proof walls. Vessels for boiling linseed oil are movable on wheels along a line of rails, so as to afford easy removal when necessary. Foul smelling vapours are drawn by a draught through a coke fire.

#### *Soap Manufacture.*

To avoid the dangers incidental to shallow boiling vessels, the rendering copper is made deep and is surmounted by a hood.

#### *Lampblack Manufacture.*

An automatic apparatus for the deposition and collection of lampblack in a closed chamber provided with windows for observing the progress of the operation. A vertical revolving shaft carries plates cooled with water, on which the lampblack accumulates, the deposits being removed and delivered into receptacles by scrapers.

#### *Match Manufacture.*

Dipping machines for pointing the matches with igniting composition are made to work automatically and in a closed chamber, by which all danger to the workmen from phosphorus fumes is avoided.

#### *Glue Manufacture and the Grinding of Bones and Slag.*

The bleaching of glue with sulphurous acid and the subsequent drying of it are carried out in a closed chamber through which a current of air is maintained, carrying the sulphurous acid and moisture to a chimney. This method is applied to the grinding and treatment of bones, mineral phosphates, and Thomas slag, where injurious dust and acid vapours are given off. Roller mills are closed in by curtains or boxes, and the dust is conveyed by a draught of air to a chamber containing baffling arrangements to cause the deposition of the dust. The ground material is sifted as it comes from the mill, and the coarser particles are returned automatically to the mill, thus confining the grinding practically to one operation. Some of the mills consist of revolving drums of metal containing steel balls. Draughts of air are used to remove foul smells from raw bones, and the vapours evolved in the treatment of phosphates in all cases where the workmen would be inconvenienced by them.

#### *Disposal of Dust.*

The means noticed under this head are comprehended under two general methods, that of the well known "Cyclone" dust collector, and of chambers containing flannel or other kind of filtering cloth, from which the accumulated dust is periodically removed by shaking or beating.—*Oil, Paint, and Drug Reporter*.

#### JAPANESE SULPHUR MINES.

The export of sulphur from Hakodate shows a decrease from 7,096 tons, value 16,917*l.*, in 1887 to 3,609 tons, value 8,681*l.*, in 1888. This is attributable almost entirely to the fact that no vessels could be induced to call at the port to take the sulphur away, the price, delivered in Hakodate (2*l.* 3*s.* 6*d.* per ton), being sufficiently high to prevent its being shipped. The shipments last year went to San Francisco.

What prevents Japan from rivalling Sicily as a sulphur-producing country is the absence of facilities for increasing the output of sulphur and for shipping it. At present there is only one place where sulphur is worked on any scale, and that is Atosanobori, near Kushiro, on the south-east coast of the island on which Hakodate is situated.

The Atosanobori mine is part of an extinct volcano. The sulphur covers a considerable portion of the slopes of the hill and of the sides of the old crater. Supposing the ore to be only 4 or 5 feet thick on the surface, there must be at this place over 1,000,000 tons of high-class ore. Recent borings (by Japanese) gave the thickness of ore as 30 feet in some places, but it is probable that the richness of the ore decreases with the depth below the surface. It would be quite safe to count upon at least 5,000,000 tons of good ore. At present the ore yields over 50 per cent. of pure sulphur, a small portion of it being almost pure. The residue from the refining kettles would well repay a second treatment, as it appears to contain a considerable percentage of sulphur, as much as 15 per cent. probably, or about the average yield of the whole of the Sicilian sulphur ore.

The present output from Atosanobori does not exceed 10,000 tons of sulphur per annum (output about 9,000 tons in 1888). The cost of the sulphur, free on board at Kushiro, would be about 30*s.* 10*d.*—*Eng. and Min. Journal*.

#### THE METAL PRODUCTION OF RUSSIA.

A comparison has been made between the latest report of the Russian Mining Commission, edited by M. Konbeline,

and that published in 1878 by M. Skalkovsky. The report does not give any figures later than the end of 1887, but the comparison supplies important data for measuring the progress of the Russian metal industry. So far as the precious metals are concerned, the product of gold has fallen during these 10 years from 2,572 to 2,128 poods, while that of silver has increased from 699 to 939 poods, and that of platinum from 126 to 269 poods. The figures are different for the precious metals chemically refined, namely, 1,898 poods of gold and 1,043 poods of silver in 1887. Those for platinum are unknown, the melting of that metal being mainly carried on abroad. Notwithstanding the affinity that exists between silver and lead, and the increase in the produce of silver, less lead was extracted in 1887 than was the case 10 years before—60,428 poods instead of 85,281. The amount of gold extracted is less than it used to be. It appears that less and less auriferous soil is discovered in the region of Yakoutsk. On the other hand, the fact that more gold is found in the quartz mines than formerly (164 poods instead of 87) is of favourable augury for the future. It is not believed by Russian economists that there will be much progress in the gold industry until the great Siberian railway is finished, as this scheme takes away the labourers from all other work, and makes the price of machinery abnormally high. The only progress of the gold industry at the present moment is found in the province of the Amoor. Foreign competition has for a long time been injurious to the production of copper, but the raising of the Customs tariff has made an alteration by which the Caucasus has benefited. In 10 years the smelting of copper has risen from 214,865 to 304,607 poods. On the other hand, zinc has fallen from 283,398 to 221,250 poods, in consequence of the decline of the home demand. Very little tin is obtained in Russia (629 poods in 1887), and that only in Finland. Quicksilver, a rare metal, was not worked in Russia until 1886, but in 1887, 3,911 poods of it were obtained. During the 10 years the extraction of manganese rose from 11,790 in 1878 to 3,553,535 poods in 1887. In 1886, however, the figure was 1,000,000 poods higher. The sudden fall is due to the rivalry of Chili.—*Ibid.*

## IRON AND STEEL STATISTICS.

### STATISTICS OF CONVERTERS.

The numbers of converters operative and inoperative during 1889 were as under:—

District.	Number of Converters.					
	Operative.		Inoperative.		Total.	
	Acid.	Basic.	Acid.	Basic.	Acid.	Basic.
South Wales.....	20	..	6	..	26	..
Cleveland.....	6	10	2	..	8	10
Sheffield, &c. ....	14	..	10	..	24	..
Cumberland .....	10½	..	2½	..	13	..
Lancashire and Cheshire ...	10	..	10	..	20	..
Staffordshire, Scotland, &c. .	..	12½	..	3½	..	16
Totals .....	60½	22½	30½	3½	91	26

The average production per converter appears to have increased, as between 1888 and 1889, the average of the two years having been—

	Tons.
1888.....	23,003
1889.....	25156

### STATISTICS OF OPEN-HEARTH STEEL.

An attempt has been made to obtain for the present report, for the first time, the quantities of acid and basic open-hearth steel produced, with the following result:—

#### QUANTITIES OF ACID AND BASIC OPEN-HEARTH STEEL PRODUCED DURING THE YEAR 1889.

District.	Production of Open-Hearth Steel Ingots.		Total.
	Acid.	Basic.	
	Tons.	Tons.	Tons.
Scotland.....	437,605	2,460	440,065
North-East Coast .....	437,100	..	437,100
Wales (North and South)...	209,392	3,226	212,618
Sheffield, Leeds, &c.....	114,544	7,253	121,794
Lancashire and Cumberland	104,685	11,927	116,612
Staffordshire, Cheshire, &c...	54,138	16,842	70,980
Total .....	1,357,461	71,708	1,429,169

At the end of 1889 there were 274 acid and 17 basic open-hearth furnaces erected in Great Britain, making a total of 291, as compared with 288 furnaces at the end of the previous year. The number at the end of 1889 would have been larger but for the fact that in the interval one important works in South Wales has been reconstructed, and has now only 6 furnaces, as compared with 14 in 1888.—*Ironmonger.*

## BOARD OF TRADE RETURNS.

### SUMMARY OF IMPORTS.

	Month ended 30th April	
	1889.	1890.
	£	£
Metals.....	1,871,161	1,856,217
Chemicals and dyestuffs.....	879,505	786,767
Oils.....	452,004	435,581
Raw materials for non-textile industries.....	2,887,633	3,033,320
Total value of all imports ....	37,255,785	35,680,242

### SUMMARY OF EXPORTS.

	Month ended 30th April	
	1889.	1890.
	£	£
Metals (other than machinery) ....	3,272,716	3,785,367
Chemicals and medicines .....	632,737	750,574
Miscellaneous articles.....	2,884,277	2,881,462
Total value of all exports.....	13,634,715	20,344,367

## IMPORTS OF METALS FOR MONTH ENDED 30TH APRIL.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
			£	£
Copper:—				
Ore ..... Tons	10,441	9,889	74,847	71,997
Regulus ..... "	5,962	7,363	147,952	198,008
Unwrought .... "	3,716	2,788	152,857	137,216
Iron and steel:—				
Iron ore ..... "	366,801	512,869	383,707	451,081
Iron bolt, bar, &c. "	4,616	5,957	41,578	60,429
Steel, unwrought.. "	164	290	2,191	3,239
Lead, pig and sheet "	10,869	12,001	139,139	152,019
Pyrites ..... "	50,139	55,683	99,502	109,543
Quicksilver ..... Lb.	947,275	879,694	99,128	110,164
Tin ..... Cwt.	69,303	33,777	342,625	151,283
Zinc ..... Tons	1,190	2,519	72,132	55,895
Other articles ... Value £	..	..	315,500	361,313
Total value of metals	..	..	1,871,161	1,856,217

## IMPORTS OF CHEMICALS AND DYE STUFFS FOR MONTH ENDED 30TH APRIL.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
			£	£
Alkali ..... Cwt.	1,117	3,071	1,738	1,588
Bark (for tanners, &c.) ..... "	33,681	53,469	13,738	22,566
Brimstone ..... "	37,319	64,29	8,903	13,952
Chemicals ..... Value £	..	..	133,615	143,755
Cochineal ..... Cwt.	708	902	4,405	5,989
Cutch and gambier Tons	3,908	1,100	76,350	39,244
Dyes:—				
Aniline ..... Value £	..	..	22,679	23,857
Alizarine ..... "	..	..	23,911	17,486
Other ..... "	..	..	2,325	2,549
Indigo ..... Cwt.	6,480	7,221	109,761	107,976
Madder ..... "	1,085	420	1,308	555
Nitrate of soda.... "	371,980	278,239	182,231	126,422
Nitrate of potash . "	32,332	28,575	27,335	25,194
Valonia ..... Tons	5,590	4,184	79,093	85,506
Other articles... Value £	..	..	192,965	171,028
Total value of chemicals	..	..	879,505	786,767

## IMPORTS OF OILS FOR MONTH ENDED 30TH APRIL.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
			£	£
Cocoa-nut..... Cwt.	10,422	10,339	13,363	12,529
Olive ..... Tons	3,334	2,068	116,590	71,957
Palm ..... Cwt.	66,225	61,729	67,021	64,890
Petroleum ..... Gall.	3,936,329	6,018,981	121,088	110,146
Seed ..... Tons	1,203	1,307	32,393	34,890
Train, &c..... Tons	761	1,005	15,837	23,008
Turpentine ..... Cwt.	18,848	15,739	29,082	23,533
Other articles .. Value £	..	..	56,630	64,626
Total value of oils ...	..	..	452,004	435,581

## IMPORTS OF RAW MATERIALS FOR NON-TEXTILE INDUSTRIES FOR MONTH ENDED 30TH APRIL.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
			£	£
Bark, Peruvian .. Cwt.	15,869	11,349	50,711	31,212
Bristles..... Lb.	253,312	353,599	35,352	61,577
Caoutchouc..... Cwt.	15,264	20,517	145,595	251,494
Gum:—				
Arabic..... "	4,203	3,186	15,921	10,658
Lac, &c..... "	16,512	10,792	60,989	44,077
Gutta-percha .... "	4,133	8,204	46,185	100,588
Hides, raw:—				
Dry..... "	77,590	41,579	209,529	115,833
Wet..... "	46,509	45,640	35,583	103,336
Ivory ..... "	1,807	1,130	84,849	58,714
Manures:—				
Guano ..... Tons	2,792	3,843	21,611	22,166
Bones..... "	5,044	8,980	24,270	47,002
Paraffin..... Cwt.	19,666	38,597	24,492	47,771
Linon rags..... Tons	4,649	3,158	49,056	32,118
Esparto, &c. .... "	16,648	29,809	84,055	97,609
Pulp of wood .... "	9,333	11,340	51,460	59,868
Rosin..... Cwt.	183,124	151,852	36,160	30,070
Tallow and stearin "	119,631	134,448	153,639	161,442
Tar ..... Barrels	3,198	5,171	1,593	3,132
Wood:—				
Hewn ..... Loads	172,076	194,593	373,966	380,254
Sawn ..... "	195,981	205,292	500,677	521,382
Staves ..... "	8,708	10,932	41,005	41,189
Mahogany ..... Tons	5,845	6,958	51,450	62,55
Other articles... Value £	..	..	751,275	748,760
Total value .....	..	..	2,887,633	3,033,320

Besides the above, drugs to the value of 66,231*l.* were imported as against 69,987*l.* in 1889.

## EXPORTS OF METALS (OTHER THAN MACHINERY) FOR MONTH ENDED 30TH APRIL.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Brass..... Cwt.	6,314	8,559	£ 35,009	£ 40,933
Copper:—				
Unwrought..... "	33,954	65,098	85,948	174,058
Wrought..... "	12,531	19,780	43,887	64,762
Mixed metal.... "	16,095	14,831	44,771	43,079
Hardware..... Value £	..	..	272,729	222,709
Implements..... "	..	..	107,556	108,696
Iron and steel.... Tons	371,611	346,354	2,458,813	2,716,075
Lead..... "	2,829	3,868	43,455	51,631
Plated wares... Value £	..	..	36,962	35,592
Telegraph wires... "	..	..	13,032	187,733
Tin..... Cwt.	9,306	8,299	44,854	39,747
Zinc..... "	11,778	11,578	8,101	11,089
Other articles .. Value £	..	..	78,899	86,863
Total value .....	..	..	3,272,716	3,785,367

## EXPORTS OF DRUGS AND CHEMICALS FOR MONTH ENDED 30TH APRIL.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Alkali..... Cwt.	536,326	600,373	£ 135,076	£ 181,514
Bleaching materials ..	111,592	161,143	41,756	47,751
Chemical manures. Tons	19,102	20,279	133,304	147,813
Medicines..... Value £	..	..	77,572	77,938
Other articles ... "	..	..	245,049	292,558
Total value .....	..	..	632,737	750,574

## EXPORTS OF MISCELLANEOUS ARTICLES FOR MONTH ENDED 30TH APRIL.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Gunpowder..... Lb.	1,036,390	659,899	£ 32,412	£ 14,978
Military stores.. Value £	..	..	112,829	132,816
Candles..... Lb.	983,500	912,500	18,138	16,951
Caoutchouc..... Value £	..	..	93,374	98,452
Cement..... Tons	66,219	54,478	129,720	104,407
Products of coal Value £	..	..	84,463	130,197
Earthenware ... "	..	..	181,620	186,448
Stoneware..... "	..	..	16,606	19,896
Glass:—				
Plate..... Sq. Ft.	325,791	330,400	19,622	19,946
Flint..... Cwt.	12,822	8,252	23,211	17,646
Bottles..... "	86,359	77,914	41,914	36,468
Other kinds.... "	18,697	17,518	14,559	16,965

## EXPORTS OF MISCELLANEOUS ARTICLES FOR MONTH ENDED 30TH APRIL—cont.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Leather:—				
Unwrought.... Cwt.	11,401	12,225	£ 105,873	£ 109,867
Wrought..... Value £	..	..	30,273	21,042
Seed oil..... Tons	6,441	5,891	133,261	134,736
Floor cloth..... Sq. Yds.	1,575,400	1,464,000	71,242	66,457
Painters' materials..... Value £	..	..	127,156	128,389
Paper..... Cwt.	75,834	79,629	139,648	138,825
Rags..... Tons	5,944	4,413	50,322	34,520
Soap..... Cwt.	48,084	46,093	48,881	46,731
Total value .....	..	..	2,984,277	2,881,462

## QUICKSILVER.

	IMPORTS.			
	1890.	1889.	1888.	1887.
April .....	Bottles, 11,729	Bottles, 12,630	Bottles, 7,960	Bottles, 11,752
Jan.—April ....	35,148	35,020	44,002	34,977
	EXPORTS.			
	1890.	1889.	1888.	1887.
April .....	3,541	8,749	4,186	6,357
Jan.—April ....	20,216	25,475	10,353	20,378
Average Price: April .....	£ s. d. 9 10 6	£ s. d. 7 15 0	£ s. d. 7 9 0	£ s. d. 6 19 0

## Monthly Patent List.

\* The dates given are the dates of the Official Journals in which acceptances of the Complete Specifications are advertised. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the said dates.

## I.—GENERAL PLANT, APPARATUS, AND MACHINERY.

## APPLICATIONS.

5661. H. J. Rogers and J. Paramor. Improvements in revolving boilers used in paper making and other industries. April 15.

5847. C. Wood. See Class X.

5991. J. W. Claridge. Improvements in apparatus for feeding fuel to furnaces mechanically. April 21.

6032. M. W. Dewey. See Class XI.

6060. J. D. Churchill. Improvements in fluid-pressure reducing apparatus. April 22.



6088. E. Brook. Improvements in kilns. April 22.

6107. J. C. Mewburn.—From C. C. Peck, United States. Improvements in the evaporation of brine and other solutions or liquids, and in apparatus employed therein. Complete Specification. April 22.

6136. J. Klein. Apparatus for condensing or cooling gaseous or liquid substances. Complete Specification. April 22.

6221. J. W. Lovibond. Improvements in instruments for estimating, measuring, or comparing colour, texture, and appearance of transparent or opaque bodies, and for testing colour vision and light perceptive power. April 23.

6399. J. von Langer and L. Cooper.—From G. Pietzka, Austro-Hungary. Improvements in or connected with reverberatory furnaces. April 26.

6400. J. von Langer and L. Cooper.—From G. Pietzka, Austro-Hungary. Improvements in recuperators or apparatus for utilising the waste heat of furnaces. April 26.

6433. G. E. Davis and A. R. Davis. An improved furnace for conducting chemical operations. April 28.

6484. G. Fletcher. Improvements in evaporating pans. April 28.

6579. W. P. Thompson.—From I. D. Smead, United States. Improvements in and relating to heaters of desiccating apparatus. Complete Specification. April 29.

6864. J. J. Shedlock and J. J. Meldrum. See Class X.

6928. S. E. Martyn. Improvements in air compressors and the like. May 5.

6951. C. Kellner. Improvements in lining boilers or digesters used in the manufacture of paper pulp and for similar purposes. May 6.

7009. P. U. Askham and W. Wilson. Improvements in disintegrating apparatus, more especially intended for disintegrating phosphates and other similar substances. May 6.

7075. B. Hunt and W. M. Mackey. Improvements in filtering apparatus. May 7.

7296. C. D. Arhens. Improvements in apparatus for polarisation of light. May 10.

7696. F. N. Mackay. See Class XII.

#### COMPLETE SPECIFICATIONS ACCEPTED.\*

1889.

6861. J. Price. Apparatus for expelling liquid from and compressing semi-fluid or plastic substances. April 23.

7343. J. Foster. Vacuum distilling and evaporating apparatus. April 30.

8169. Solvay and Co. See Class VII.

8872. W. L. Wise.—From Solvay and Co. Apparatus for the continuous treatment of pulverulent matters by fluids. May 21.

8928. F. N. Mackay.—From J. Baxter and J. Muter. Filter presses and filtering. April 23.

9041. D. B. Morison. Apparatus for evaporating liquids. April 23.

9850. J. Brown and G. Johnston. Vacuum drying apparatus. April 23.

10,907. W. P. Thompson.—From N. Notkin and P. Marix. Distilling products by means of steam, separating them according to their points of vaporisation, and apparatus therefor. May 14.

10,945. J. Vicars, T. Vicars, and J. Vicars, jun. Apparatus for feeding fuel to furnaces. May 14.

11,350. R. C. Garton, C. H. Garton, and W. Lawrence. Apparatus for evaporating, heating, or cooling liquids. May 21.

11,402. E. Bazin. Hydro-centrifugal apparatus for washing, amalgamating, and separating metallic ores, and for washing and separating other bodies of different densities. May 21.

1890.

2139. O. Bellot. Means for regulating the generation of heat in ovens, furnaces, &c. April 23.

2618. H. J. Haddan.—From S. T. Wellman. Method and mechanism for charging furnaces. April 30.

3261. H. H. Lake.—From O. H. Jewell. Apparatus for filtering water. April 30.

4354. A. Eickhorn and R. Jaspar. Hydrometers. April 30.

5267. A. Chapman. Construction and working of double, triple, and multiple-effect evaporating apparatus. May 14.

## II.—FUEL, GAS, AND LIGHT.

### APPLICATIONS.

5637. G. C. Fowler, M. B. Fowler, and G. A. Fowler. Improvements in or connected with night and other similar lights. April 14.

5788. S. Beson. An improved fire-lighter, and method of manufacture of the same. April 16.

5838. C. M. Pielsticker. Improvements in the distillation of mineral oils. April 17.

5971. J. Dewar and B. Redwood. Improvements in the distillation of oils and other viscous liquids. April 19.

6217. S. Griffin. Improvements in apparatus for producing combustible gas for gas-motor engines or other purposes. April 23.

6235. J. Hey and P. Hey. Improvements in fire-lighters. April 24.

6256. T. D. Rock. The application of anthracite gas for metallurgical and other purposes. April 24.

6282. R. de Soldenhoff. Improvements in means employed in charging coke ovens. April 24.

6323. T. T. Vernon. Improvements in or appertaining to apparatus for consuming smoke and economising fuel. April 25.

6463. E. Neave. Method of and apparatus for treating atmospheric air to obtain oxygen therefrom. Complete Specification. April 28.

6465. C. M. Pielsticker. Improvements in the distillation of hydrocarbon and other oils. April 28.

6466. C. M. Pielsticker. Improvements in the distillation of hydrocarbon and other oils. April 28.

6553. A. Longsdon.—From F. Salomon, Germany. Improvements in obtaining oxygen gas from atmospheric air. Complete Specification. April 29.

6575. J. H. W. Stringfellow. Improvements in the manufacture of gas and apparatus therefor. April 29.

6584. J. J. Shedlock and T. Denny. Improvements in gas producers. Complete Specification. April 29.

6800. W. C. Andrews. Improvements in the purification of illuminating or heating gases. Complete Specification. May 2.

6804. W. C. Andrews. Improvements in apparatus for the purification of illuminating gas. Complete Specification. May 2.

6877. J. J. Hiertz and A. S. Garnett. Combustible coal brick. Complete Specification. May 3.

6900. W. Gordon and G. A. Newton. Improvements in fire-lighting and fuel. May 5.

6966. J. Horne. See Class XVIII.—B.

7040. J. J. Cooper. Improvements in "carburettors." Complete Specification. May 6.

7063. H. Fourness. Improvements in the method of and in apparatus for the manufacture of carburetted water-gas. May 7.

7144. T. Williams. The manufacture of smokeless fire-lighters. May 7.

7252. R. C. Lindsay. An improved process for the manufacture of illuminating gas, and apparatus therefor. May 9.

7277. G. M. Cruikshank.—From J. C. Reissig, Argentine Republic; J. Landin, Sweden; and P. F. Macallum, Scotland. Improvements in the manufacture of gas for illuminating and heating purposes, and application thereof. May 10.

7335. G. M. Cruikshank.—From J. C. Reissig, P. F. Macallum, and J. Landin. Improvements in incandescent lighting, by means of calcareous gases, and in apparatus therefor. May 12.

7569. T. Le Poidevin. Apparatus for producing air gas. May 15.

7705. H. Rudge. Improvements in or relating to candles. May 17.

7714. C. B. Newton. Apparatus for automatically delivering any proportionate quantity of oxygen, air, or other gaseous body or bodies into gas purifiers or other vessels, pipes, or conduits, irrespective of any pressure there may be in such gas purifiers or other vessels. May 17.

7739. J. von Langer and L. Cooper. Improvements in or connected with the manufacture of water-gas. May 17.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

8040. A. Myall.—From J. H. Lancaster. Artificial fuel. May 21.

8993. S. W. Wilkinson. Producing clean gaseous fuel for the evaporation of brine, and recovering the ammonia formed during production of said gaseous fuel. May 21.

9783. A. Nielson and W. Black. Distilling shale, coal, and other oil and tar-yielding minerals, and retorts therefor. April 23.

10,164. W. Crookes and F. I. Ricarde-Seaver. Treatment of water-gas. April 23.

10,277. J. Dewar and B. Redwood. Distillation of mineral oils and like products, and apparatus therefor. April 30.

10,340. S. Clarke. Night-lights. May 7.

10,392. H. J. Haddan.—From F. F. Amoroso. Burning liquid fuel in boiler furnaces, &c. April 30.

10,488. J. H. R. Dinsmore. Apparatus used in the manufacture of gas from coal, &c. May 14.

10,581. J. C. Johnson. Producer for making producer-gas, water-gas, and coke. May 7.

11,104. J. Carr. Manufacture of illuminating gas from coal. May 21.

11,125. F. Sanders. Retorts for producing gas. May 14.

11,164. F. T. Schmidt. Method and apparatus for consuming smoke. May 21.

11,654. J. Drabble. Apparatus for the prevention and consumption of smoke, and more perfect combustion of fuel. May 21.

17,817. E. de Pass.—From E. Demoguin and L. Bourdier. Improved fire-lighter and fuel. April 23.

1890.

2611. W. W. Popplewell.—From H. Couried. Artificial fuel. March 26.

3306. H. Ekeland. Furnace for continuous carbonising or coking. April 23.

3363. C. Heucken. Coke ovens. May 14.

5464. J. G. Hawkins and J. Barton. Vertical gas retorts and attachments. May 14.

#### IV.—COLOURING MATTERS AND DYES.

##### APPLICATIONS.

5721. J. Hall. Improvements in the manufacture or production of colouring matters. April 15.

5722. B. Willcox.—From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture of diquinolyline derivatives. April 15.

5723. B. Willcox.—From The Farbenfabriken vormals F. Bayer and Co., Germany. Improvements in the manufacture of phenacetine derivatives. April 15.

5736. J. Imray.—From La Société Anonyme des Matières Colorantes et Produits Chimiques de St. Denis, A. F. Poirrier, and D. A. Rosenstiehl, France. Manufacture of azo colouring matters derived from azoxyamines. April 15.

5737. J. Imray.—From La Société Anonyme des Matières Colorantes et Produits Chimiques de St. Denis, France. Manufacture of colouring matters derived from fluoresceine. April 15.

5777. O. Imray.—From The Farbwerke vormals Meister, Lucius and Brüning, Germany. Production of grey basic colouring matters by the action of hydrochlorate of nitrosodimethylaniline, or hydrochlorate of nitrosodiethylaniline upon 1·5 dioxynaphthalin, and upon 1·2 tetroxydinaphthyl. April 16.

5780. O. Imray.—From The Farbwerke vormals Meister, Lucius and Brüning, Germany. Production of black colouring matters from amidoflavopurpurin and amidanthrapurpurin. April 16.

6195. S. S. Bromhead.—From Die Chemische Fabrik Grunau, Laudshoff and Meyer, Germany. A process for the preparation of a blue colouring matter for cotton. April 23.

6274. O. Imray.—From The Farbwerke vormals Meister, Lucius and Brüning, Germany. Production of blue-grey colouring matters from indulines soluble in alcohol, resulting from the action of hydrochlorate of amido-azobenzol upon an aqueous solution of hydrochlorate of aniline or orthotoluidine. April 24.

6346. The British Alizarine Co., Limited, and D. C. Bendix. Manufacture of soluble alizarine compound for dyeing and printing. April 25.

6486. J. Imray.—From La Société Anonyme des Matières Colorantes et produits Chimiques de St. Denis, France. Manufacture of nitrated colouring matters. April 28.

6729. C. Dreyfus. Improvements in the manufacture and production of colouring matters. May 1.

6874. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik, Germany. Improvements in the manufacture and production of substantive colouring matters. May 3.

6875. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik, Germany. Improvements in the manufacture and production of sulpho-acids of a red basic naphthalene colouring matter. May 3.

6879. H. H. Lake.—From A. Leonhardt and Co., through Messrs. Wirth and Co., Germany. Improvements in the manufacture of colouring matters. May 3.

6931. C. D. Abel. Improvements in the manufacture of colouring matters. May 7.

6932. C. D. Abel. Improvements in the manufacture of colouring matters. May 7.

6946. S. Pitt.—From L. Cassella and Co., Germany. Production of new basic blue colouring matters. May 5.

7018. W. Cole. Improvements in the manufacture of indigo. May 6.

7421. R. Holliday. Improvements in the production and utilisation of a new hydroxy-quinone. May 13.

7515. W. Akam. A new or improved copying ink. May 14.

7575. A. G. Green and T. A. Lawson. A process for the production of new amido bases. May 15.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

8156. H. H. Lake.—From Leonardt and Co. Manufacture of amido-phenols. April 30.

8299. B. Willcox.—From The Farbenfabriken vormals F. Bayer and Co. Azo colouring matters for dyeing and printing. April 30.

8673. B. Willcox.—From The Farbenfabriken vormals F. Bayer and Co. Manufacture of colouring matter and of leuco-bases employed therein. May 7.

8750. B. Willcox.—From The Farbenfabriken vormals F. Bayer and Co. Basic colouring matter. May 7.

9427. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik. Colouring matter of the oxyketone group, for dyeing and printing with the aid of mordants. April 23.

10,934. The Clayton Aniline Company, Limited, and C. Dreyfus. Preparation of new sulphonic acids for manufacture of colouring matters. May 14.

11,613. O. Imray.—From The Society of Chemical Industry, Basle. New red and blue colouring matters. April 30.

11,848. O. Imray.—From The Society of Chemical Industry, Basle. Blue colouring matter. April 30.

15,175. O. Imray.—From The Farbwerke vormals Meister, Lucius and Brüning. Production of amido-oxy-naphthalin-disulphonic acid, and amido-dioxy-naphthalin-monosulphonic acid, and the manufacture of their diazo compounds and of azo colouring matters therefrom. May 14.

15,176. O. Imray.—From The Farbwerke vormals Meister, Lucius and Brüning. Production of amido-naphthol-monosulphonic acids, and manufacture of their diazo compounds and azo colouring matters therefrom. May 14.

1890.

3095. H. H. Leigh.—From R. G. Williams. Manufacturing of colouring matters. April 23.

#### V.—TEXTILES, COTTON, WOOL, SILK, Etc.

##### APPLICATIONS.

6127. A. H. Norman. Improvements in the treatment of ramie. April 22.

6155. G. H. Croker. An improved process for the removal of fibre or lint from and cleaning cotton seed, and means or apparatus employed therein. April 23.

7389. J. Longmore and W. L. Watson. Improvements in or relating to the decortication of rhea, jute and other fibrous vegetable stems. Complete Specification. May 12.

7390. J. Longmore and W. L. Watson. Improvements in or relating to the decortication of rhea, jute, and other fibrous vegetable stems. Complete Specification. May 12.

7423. W. Peters. Improvements in the method of, and in means and appliances for, tarring, colouring, and otherwise preparing thread, hemp, jute, or other twine for sewing, and in feeding or supplying the same. May 13.

7568. K. T. Sutherland and G. Esdaile. An improved method of treating animal fibres so that they will flock and spin more easily. May 15.

#### COMPLETE SPECIFICATION ACCEPTED.

1889.

14,751. I. Smith. Treatment of woollen, worsted, back-worsted, unions, and other woven fabrics. May 14.

#### VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

##### APPLICATIONS.

6202. S. Stepanow. See Class XI.

6213. H. J. Haddan.—From L. Appelmans and L. Tibbaut, Belgium. Method for colouring and marbling paper on the paper machine. April 23.

6315. R. Harrison. Washing, scouring, and bleaching cotton, linen, wool, or other fibrous materials. April 25.

6506. W. H. Booth. Improved process and means to be employed in connexion with and relating to the operation of dyeing velvets, plushes, and like materials; applicable also in the carrying out of proximate processes relating to the treatment of fibrous substances or materials, either in a manufactured, partly manufactured, or pulpy state. April 28.

6612. R. T. Webb. Expediting and improving the process of bleaching linen, cotton and other yarns in the hank. April 30.

7198. W. H. Spencer. An improved process of bleaching textile fibres. May 8.

7202. P. Cavaillès. An improved process for dyeing wool, and apparatus for use in the same. May 8.

7522. W. Elbers. Process for obtaining designs or patterns in red colour on textile fabrics dyed with indigo. May 14.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

8107. V. Schevelin and P. Mindovsky. Cleansing and bleaching fibrous substances, and fixing colouring matters thereon by means of acid and alkaline residues of naphtha manufacture, or analogous bodies. April 23.

9836. W. E. Heys.—From C. Vandermeirssche. Method and apparatus for dyeing, &c. yarns and textiles. April 23.

10,993. B. Willcox.—From The Farbenfabriken vormals F. Bayer and Co. Manufacture of tannin compounds, especially for printing purposes. May 14.

1890.

1702. G. H. Craven. Dyeing machines. May 14.

4683. A. J. Boulton.—From A. and M. Koblenzer. Dyeing yarns. April 30

## VII.—ACIDS, ALKALIS, AND SALTS.

## APPLICATIONS.

6046. J. Pointon. Improvements in apparatus for the manufacture of alkaline silicates and aluminates, applicable also in part as a gas-producer for use with pig-iron or other blast furnaces. April 22.

6047. J. Pointon. An improved method of manufacturing alkaline aluminates. April 22.

6048. J. Pointon. An improved method of manufacturing alkaline silicates. April 22.

6333. F. M. Lyte. Improvements in the production of magnesic oxychloride. April 25.

6416. G. E. Davis and A. R. Davis. Improvements in the preparation of chlorine. April 28.

6549. J. Plummer, jun. Process to obtain hydrochloric acid and ammonia. April 29.

6698. G. E. Davis and A. R. Davis. Improvements in the manufacture of chlorine. May 1.

6758. J. McEwen. A new or improved continuous method of generating carbonic acid gas from a soluble carbonate in solution and an acid in solution, and in apparatus connected therewith. May 2.

6831. G. E. Davis and A. R. Davis. Improvements relating to the preparation of chlorine. May 3.

7058. J. Y. Johnson.—From J. Marx and Dr. Weller, Germany. Improvements in and apparatus for or connected with the obtaining, liquefying, and storage of chlorine. May 6.

7247. H. Baudouin and P. F. Escarpié. A process and apparatus for the manufacture of ammonia. May 9.

7527. J. Vivian and G. Bell. Improvements in apparatus for use in the manufacture of bicarbonate of soda by the ammonia-soda process. May 14.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

8169. Solvay and Co. Distillation and condensation of ammoniacal and other liquids, and apparatus therefor. April 30.

8177. E. Hermite, E. J. Paterson, and C. F. Cooper. Bleaching and disinfecting liquor. May 21.

8779. J. J. Alsberge. Manufacture of chlorine in combination with the ammonia or the Leblanc process. April 23.

8862. W. Thorp. Method and apparatus for condensing and utilising sulphuric and sulphurous acid fumes. April 30.

8993. S. W. Wilkinson. See Class II.

9225. T. Turner. Treatment of waste pickle from galvanising works. April 23.

9680. A. G. Greenway. Galvanising iron, and utilising the waste acids therefrom. April 23.

10,142. A. S. Ramage. Preparing iron oxides from ferrous solutions. April 30.

10,554. T. Parker and A. E. Robinson. Utilisation of sulphate of iron galvanising pickle. May 7.

11,351. R. C. Garton, C. H. Garton, and W. Lawrence. Crystallisation of salts or crystallisable acids. May 21.

13,003. A. W. Ellis. Treatment and distillation of ammoniacal liquor of gas and other works, manufacture of sulphate of ammonia therefrom, apparatus therefor, and prevention of nuisance therefrom. May 14.

1890.

4652. J. C. Mewburn.—From C. C. Peck. Purifying brine. May 7.

## VIII.—GLASS, POTTERY, AND EARTHENWARE.

## APPLICATIONS.

5815. S. R. Audnitz. Substitute for glass. April 17.

5830. W. H. Sharman and R. E. Oldham. The making of earthenware, granite, or china by a new machine press, such as oval, square, octagon, round dishes, bakers, plates, hand basins, &c. April 17.

5970. H. M. Ashley. Improvements in the manufacture of ceramic ware and apparatus for that purpose. April 19.

6058. G. Scott. A new or improved method or manufacturing slates from glass and other materials, and in the apparatus therefor. Complete Specification. April 22.

6196. W. R. May. Improvements in the manufacture of glazed bricks and tiles. April 23.

6231. A. Fielding. Improvements in ovens or kilns used for firing or hardening articles of pottery. Complete Specification. April 24.

6722. J. Kirkby. Drying kiln (for burning bricks or earthenware of like nature). May 1.

6979. J. H. Lewis. An improved asbestos composition shadow for glass and other furnaces. May 6.

7099. B. F. McTear. Improvements in or appertaining to kilns for burning bricks, terra-cotta, or the like. May 7.

7145. D. Rylands. Improvements in machinery for the manufacture of bottles and other glass ware, and in the processes of working the same. May 8.

7184. C. H. Edwards. Improvements in the manufacture of gas retorts, drain pipes, and other earthenware or clay ware. May 8.

7327. F. O. Thompson. Improvements in pots for melting glass. May 10.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

7245. W. H. Hollingshead and H. Griffiths. Decorative pottery ware. May 7.

7761. O. P. Erard and B. J. Round. Depositing metal on glass, porcelain, &c. April 23.

8760. A. Drummond. Apparatus for manufacture of corrugated, ribbed, or other glass having patterns on surface. April 23.

10,088. The Brosely Tileries Company, Limited, and J. Crump. Earthenware quarries or tiles. April 23.

10,887. D. Chapman. Casting or forming articles of pottery, china, and earthenware. May 14.

11,064. E. E. Cousins.—From B. E. Olsen. Fusible compounds suitable for drain pipes, flushing chambers, and the lining of conduits. May 14.

11,690. M. Poumarède. Manufacture of flower vases, pots, &c. May 14.

1890.

2583. J. H. Bailey. Glazed or enamelled railway signal arms. March 26.

3229. E. Leuk and H. Aynsley. Means for supporting biscuit or glost ware in kilns. April 23.

4021. A. J. Boulton.—From P. Sievert. Production of solution of alkaline silicate or water-glass, and apparatus therefor. April 23.

# IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

## APPLICATIONS.

5719. W. R. Taylor. Improvements in and means for the manufacture of cement. April 15.

5827. A. B. Rigby. An improved form and combination of concrete blocks. April 17.

6555. G. Brewer.—From J. C. Firth, New Zealand. A new or improved insulating and fire-proofing material or composition. April 29.

6711. F. C. Goodall. An improved marine cement. May 1.

6855. G. Higham. An improved composition for use in and in connexion with the manufacture of Portland cement. Complete Specification. May 3.

6952. C. Kellner. Improvements in coating iron, steel, or other metals or materials with Portland or other cement. May 6.

7107. J. P. Bayly.—From P. Arnold, United States. Improvements in paving blocks. May 7.

7641. A. M. Thomson. Improvements in paving, said improvements being also applicable to tiles and slabs. May 16.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

8428. M. May. Artificial polished or ground stone. May 21.

8795. A. N. Ford. Materials for roofing and other purposes. March 26.

9666. E. Numan. Base or foundation for the reception of plaster, &c. April 23.

11,128. G. Batchelor. Arrangements for drying slurry by the waste heat from cement kilns. April 23.

1890.

1097. H. H. Lake.—From J. F. Gesner. Manufacture of an artificial solid material, and apparatus therefor. May 21.

3025. G. West. Plastering material. May 21.

# X.—METALLURGY, MINING, Etc.

## APPLICATIONS.

5608. J. Letcher. Improvements in and connected with furnaces for recovering tin from tin-slag. Complete Specification. April 14.

5638. T. Teague. A process for extraction of tin from the slag or debris of tin smelting. April 14.

5641. H. Howard, of the Firm of Lloyd and Lloyd. Improvements in the manufacture of iron and steel tubes. April 14.

5659. G. W. Marble and M. W. Simons. Improvements in forging metals and in machinery therefor. Complete Specification. April 15.

5847. C. Wood. Improvements in the manufacture of fibrous material from slag or other vitreous substance, and in the apparatus used in such manufacture. April 17.

5894. A. J. Boulton.—From J. W. Bookwalter, Germany. Improvements in converters. Complete Specification. April 18.

5937. W. C. Loe. Improved method or process for treating metal-bearing bodies, and the recovery of metals therefrom. April 18.

6046. J. Pointon. See Class VII.

6216. C. T. J. Vautin. An improved hydrometallurgical method of and means for the extraction of gold or platinum from their ores. April 23.

6256. T. D. Rock. See Class II.

6316. S. Fox. Improvements in the method or process of manufacturing steel in connexion with the open-hearth system. April 25.

6462. H. Napier and T. Carr. Improvements in the production of metals and metallic alloys from their ores and oxides. April 28.

6524. W. Ryland. An improved process for securing the better adhesion of metals when electrically deposited or coated. April 29.

6529. J. H. Bramwell. An improved process to be employed for hardening steel. Complete Specification. April 29.

6572. A. McLean. Improvements in the preparation of metal for various purposes. April 29.

6645. J. H. Lancaster and M. R. Conley. Improvements in the manufacture of iron or steel. April 30.

6670. S. O. Cowper-Coles. Improvements in the deposition of alloys. April 30.

6694. B. Edwards, L. Davies, and W. R. Williams. An air receiver applied especially to the casting of ingots, shafts, cannons, and all manner of castings cast perpendicularly. May 1.

6821. Sir H. H. Vivian, Bart. Improvements in the manufacture of copper. May 2.

6864. J. J. Shadlock and J. J. Meldrum. Improvements in apparatus for the production of gases for metallurgical and other purposes. Complete Specification. May 3.

6936. G. Bamberg. Extraction of gold from ore. May 5.

6960. J. Neill. Improvements in the manufacture of steel plated and gated ingots, and in the moulds for same. May 6.

6980. A. C. Chance. Making sleeves, nozzles, and stoppers used in the manufacture of steel. May 6.

7055. A. Sinclair. Improvements in the treatment of ores, matte, spieess, and other products containing copper, together with nickel or cobalt. May 6.

7070. J. Heaton and G. H. Holden. Improvements in the manufacture of steel and iron, and in apparatus to be employed in such manufacture. May 7.

7129. J. D. Danton. A process and apparatus for reduction of iron and other oxide ores, and for fusion and purification of the produced metals. May 7.

7253. W. Hodge. Improvements in converting iron into steel, or the steelifying of wrought iron objects in a manufactured or partly manufactured state. May 9.

7305. J. Allan. A new or improved composition of metals. May 10.

7461. H. H. Lake.—From The Russel and Erwin Manufacturing Company, United States. Improvements relating to the annealing of metals. Complete Specification. May 13.

7464. W. J. Smuts.—From J. Cronoun, W. Hicks, and J. Laitz, Transvaal. An improved amalgamator, suitable for use in the separation of gold from quartz. May 13.

7647. C. Knight and W. F. Sharp. A new or improved solution for use in the preparation of copper utensils for re-tinning. May 16.

7733. H. H. Lake.—From G. W. Cummins, United States. An improved process and apparatus for preventing the access of air to iron and steel during annealing. Complete Specification. May 17.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

843. J. H. Selwyn. Hydrometallurgical processes. April 23.

9987. The Alkaline Reduction Syndicate, Ltd. Treatment of zinc ores. May 21.

10,154. J. C. Bull. Manufacture of copper alloys. May 21.

10,274. H. Cave. Process and apparatus for amalgamation of gold and silver contained in their ores. May 21.

10,298. J. H. Pollok. Wet method of extracting gold, and apparatus therefor. April 30.

10,394. J. Harper. Treating gold and silver ores by wet method. April 30.

10,835. T. Hydes. Treating blast-furnace and other slags, and apparatus therefor. May 14.

10,836. T. Hydes. Treating blast-furnace and other slags, and apparatus used therein. May 14.

11,344. C. F. Jozs. Manufacture of ornamental plates of metal. May 21.

11,402. E. Bazin. See Class I.

20,223. E. Taussig. Improvements in casting. April 23.

1890.

70. I. Bang and A. Rudin. Separating tin from other metals and recovering tin from tin-plate scrap, &c. April 30.

128. I. Bang and A. Rudin. Production of tin plates, and apparatus therefor. April 23.

1754. G. Höper. Method and apparatus for manufacturing dense-metal castings, and sheet metal and wire therefrom. April 23.

2981. M. F. Coomes and A. W. Hyde. Manufacture of steel. April 2.

3440. J. L. Sebenius. Removing gases and impurities from metals or alloys. May 14.

3483. L. L. Burdon. Seamless compound ingots or bars and wire. April 23.

4386. J. von Ehrenwerth. Producing iron and steel from ores and other materials. May 14.

4690. H. J. Haddan.—From G. Conkling. Concentrating magnetic iron ore. April 30.

5608. J. Letcher. Improvements in and connected with furnaces for recovering tin from tin-slag. May 21.

## XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

## APPLICATIONS.

5810. F. L. Rawson.—From Messrs. Hartmann and Braun, Germany. An improved galvanic battery. April 17.

5920. P. Schoop. Improvements in accumulators or secondary batteries. April 18.

5938. K. E. Boettcher. A new or improved secondary element. April 18.

6032. M. W. Dewey. Improvements in electric heating apparatus. Complete Specification. April 21.

6177. G. Barker.—From H. Woodward, Canada. Improvements in secondary batteries. April 23.

6202. S. Stepanow. An electrolytic process for the preparation of a liquid for bleaching fabrics, paper pulp, and other matters. April 23.

6223. M. Ancizar.—From R. Z. Bayon, South America. Improvements in galvanic batteries. April 23.

6236. A. J. Jarman. An improved storage or tertiary electric battery. Complete Specification. April 24.

6266. J. Marx. Improvements in means for use in electrolysis. April 24.

6476. H. T. Eagar and R. P. Milburn. Improvements in electrical batteries. April 28.

6795. H. Edmunds. Improvements in secondary or storage batteries. May 2.

6924. H. T. Eagar and R. P. Milburn. Improvements in electrical batteries. May 5.

7132. H. H. Lake.—From Lacombe et Cie., France. Improvements in the manufacture of carbon electrodes. May 7.

7185. W. P. Thompson.—From C. L. Coffin, United States. Improvements in welding metals electrically and apparatus therefor. Complete Specification. May 8.

7186. W. P. Thompson.—From C. L. Coffin, United States. Improvements in welding metals electrically, and apparatus therefor. Complete Specification. May 8.

7383. J. N. Levens. An improved battery element. Complete Specification. May 12.

7399. R. Kennedy. Improvements in heating by magnetism and electricity. May 13.

7444. A. Million and P. Bony. Improvements in and relating to galvanic batteries. Complete Specification. May 13.

7513. W. L. Wise.—From P. Schoon, Switzerland. Improvements in the manufacture of electrodes for secondary batteries. May 14.

7518. A. Walker and T. J. D. Rawlins. Improvement in means for preventing polarisation in primary batteries. May 14.

7581. W. A. Thoms. Improvements in the electro deposition of heavy metals and their alloys. May 15.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

9183. M. Sussmann. Exciting liquid for electric batteries. May 7.

10,061. G. J. Atkins. Apparatus for separating gold and other metals from their ores by the aid of electricity. April 30.

10,695. H. H. Lake.—From C. A. Hussey and E. H. Brown. Electric batteries. May 14.

10,696. H. H. Lake.—From C. A. Hussey and E. H. Brown. Electric batteries. May 14.

11,049. J. J. Wood. Dynamo-electric machines. May 7.

11,070. J. Y. Johnson.—From A. L. Riker. Elements or plates for secondary batteries. May 14.

13,795. P. von Knobloch. Turning artificial and natural light into electrical current. April 23.

18,481. C. Payen. Electric batteries. April 30.

19,129. C. R. Bonne.—From H. Meinecke, jun. Electric dry batteries. May 7.

1890.

350. L. Epstein. Electrodes for primary and secondary batteries. May 14.

2819. C. Lütke. Electrolytic solution for galvanic batteries. March 26.

3008. W. B. Hollingshead. Secondary or storage batteries. April 9.

3013. C. J. Hartmann. Storage batteries. April 9.

3439. S. C. C. Currie. Storage batteries. April 30.  
 3455. H. H. Lake.—From T. Ewing. Secondary batteries. April 23.  
 3717. L. Weigert. Electrolytic solution for galvanic batteries. April 30.  
 4657. H. H. Lake.—From E. Thomson. Soldering or uniting metals by electricity, and apparatus therefor. April 30.

## XII.—FATS, OILS, AND SOAP MANUFACTURE.

### APPLICATIONS.

5620. J. Bowden and S. Hern. Improvements in the manufacture of soap for washing purposes. April 14.  
 5833. G. Tall. Refining cotton and other oils. April 17.  
 6268. A. W. Mellwaine. Improvements in the treatment of rape and other seed preparatively to the extraction of oil therefrom, and in apparatus applicable therefor. April 24.  
 6706. C. W. Kimmins and T. Craig. Improvements in the separation of fatty or greasy matters from the wash waters of wool-washing or scouring establishments. May 1.  
 6834. J. Taylor. A new or improved process and apparatus for the recovery of glycerin and salt from soap-makers' lyes. May 3.  
 7019. C. A. Serre. An improved manufacture of soap. May 6.  
 7117. H. Scott. Improvements in the manufacture of soap. May 7.  
 7142. H. Stern. Improved method of decolorising mineral, animal, and vegetable oils, fats, and the like, and the employment of silicic acid for the purpose. May 7.  
 7696. F. N. Mackay. Improvements in appliances for cooling oil and like liquids. May 17.  
 7706. J. A. Clough. Improvements in or appertaining to the manufacture of soap. May 17.

### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

8567. J. Taylor. Soap powder. April 30.  
 8740. W. P. Thompson.—From A. Schumacker and C. Tschiffeli. Extracting oil, &c. from seeds or the like. April 23.  
 9201. J. Snowdon. Toilet and cleansing soaps. May 21.  
 18,345. H. A. A. Dombrain and O. Trumper. Apparatus for extracting fatty and other matter by means of volatile solvents. April 30.

## XIII.—PAINTS, PIGMENTS, VARNISHES, AND RESINS.

### APPLICATIONS.

6805. W. P. Thompson.—From A. F. von Pereira, Germany. Improvements in or appertaining to paints, colours, varnishes, and distempers, and in the method of applying them to canvas or other like surfaces. May 2.  
 6850. E. Parry and H. Foskett. A white pigment to be used in the manufacture of paints and colours. May 3.  
 7260. M. N. D'Andria. Improvements in the treatment of salts of iron and oxide of iron to obtain pigments, and in the utilisation of products therefrom. May 10.

### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

- 10,428. I. M. Lamb and D. Boyd. Lacquers and varnishes. April 30.  
 18,286. T. Pridham. Preservative and waterproof coating for metals, timber, masonry, &c. April 23.  
 20,289. J. Y. Johnson.—From N. C. Mitchell. Treating waste rubber goods to recover rubber therefrom, production of new product thereby, and apparatus therefor. May 14.

1890.

1684. A. J. Boulton.—From N. A. Bibikof. Improvements in paints. May 7.  
 2308. H. Beck.—From E. Schaal. Production of resin acid esters, and manufacture of lacquers and varnishes therefrom. April 23.

## XIV.—TANNING, LEATHER, GLUE, AND SIZE.

### APPLICATIONS.

5966. W. R. Pullman and H. Smith. Improvements in machinery or apparatus for finishing green, or manufactured, or partly manufactured hides or skins. April 19.  
 7509. F. R. Maggs. The preparation of skins, kips, and hides by an improvement in the process known as "un-hairing" or pulling. May 14.

### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

9776. B. Nicholson and T. Pulmer. Means for tanning hides, skins, &c. May 21.  
 10,900. W. G. Richardson. Liquid waterproof glue. May 14.

## XV.—AGRICULTURE AND MANURES.

### APPLICATION.

5614. J. M. Fletcher. See Class XVIII.—A.

### COMPLETE SPECIFICATION ACCEPTED.

1889.

9087. J. W. Lodge. Partially destroying and mixing night soil and other refuse, and converting same into manure. May 7.



## XVI.—SUGARS, STARCHES, GUMS, ETC.

## APPLICATIONS.

7737. A. Niemöller. The production of a new glutinous material similar to gum arabic and other by-products from the seed kernels of the fruit of the carob tree (*Ceratonia*). May 17.

## COMPLETE SPECIFICATIONS ACCEPTED.

1890.

1247. A. Fairgrieve. Manufacture of raw or refined sugar, and apparatus therefor. April 23.

3589. C. Steffen. Process for manufacture of sugar. May 14.

4181. S. Spitzer. Method and apparatus for manufacture of starch, and treatment of the residues obtained therein. May 21.

## XVII.—BREWING, WINES, SPIRITS, ETC.

## APPLICATIONS.

5629. R. Haddan.—From J. B. Cabean, Belgium. Improved apparatus for brewing and storing beer in casks. April 14.

5673. J. Salamon. An improved fermenting process for beer, wine, and other fermentable liquids. April 15.

6075. F. M. Maynard. The aëration of yeast for exciting alcoholic fermentations or "yeast aëerator." April 22.

6220. H. H. Lake.—From A. Joergensen and A. Bergh, Sweden. Improvements in apparatus for the production of yeast and similar substances. April 23.

6337. W. Paterson. A new or improved process for preventing and arresting the development of acids during the process of malting barley and the storage of the same or other cereals such as rye, oats, or wheat, and in the infusion whilst macerating or infusing malted barley for the manufacture of concentrated extract of malt, or for all brewing purposes, or for the manufacture of malt vinegar. Complete Specification. April 25.

6480. G. Schneider. Method for the production and storing of beer without contact with the atmosphere. Complete Specification. April 28.

6587. W. T. Whiteman.—From F. Haecck, Belgium. A new or improved process and apparatus for the distillation and rectification of alcohols. Complete Specification. April 29.

7017. W. Cole. Improvements in the manufacture of beer and like fermented liquors. May 6.

7098. A. J. Bonlt.—From La Société Generale de Maltose, Belgium. Improvements in or relating to the manufacture of maltose, or to the fermentation of amylaceous matter. May 7.

7195. J. Campbell. A new or improved process for the recovery and utilisation of yeast. May 8.

7268. W. Dawson. Improvements relating to distillers' safes, said improvements being also applicable for check sampling the spent wash at outlets of "patent stills." Complete Specification. May 10.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

7909. A. Perry. Preparation of rice and other grain or cereals for brewing, &c., and apparatus therefor. May 21.

9276. A. Gough, sen. Apparatus for making and drying malt. May 7.

## XVIII.—CHEMISTRY OF FOODS, SANITARY CHEMISTRY, AND DISINFECTANTS.

## APPLICATIONS.

*A.—Chemistry of Foods.*

5614. J. M. Fletcher. Improvements in apparatus for the compression and preservation of vegetable products. April 14.

6102. W. S. Simpson. Improved means and appliances for preserving flesh food. April 22.

6421. A. G. Cunningham. An improved bread for dyspeptics. Complete Specification. April 28.

6883. W. J. Clarke. An improved process for preserving milk and cream. May 3.

6901. C. R. C. Tichborne and W. McDonnell. Aërated milk. May 5.

7193. A. S. Krueger and J. B. Black. Improvements in and relating to alimentary substances. May 8.

*B.—Sanitary Chemistry.*

6245. C. G. Moor. Improvements in the treatment of sewage. April 24.

6252. J. Longshaw. Improvements in apparatus for filtering or purifying water, sewage, or other liquids. April 24.

6397. F. B. Hill. Improvements relating to the treatment of sewage and the ventilation of drains and sewers, and to apparatus therefor. April 26.

6660. M. M. Brophy. Improvements in furnaces chiefly designed for destroying organic substances. April 30.

6966. J. Horne. Improved method and apparatus for hermetically carbonising vegetable and other products, and also for drying waste vegetable and other products for fuel and other purposes. May 6.

7165. F. N. Mackay. Improvements in apparatus for drying sludge or like matter. May 8.

7419. W. Walkington. An improved city and town refuse destructor. May 13.

7500. L. Hesse. Improvements in destructors for town and other refuse. Complete Specification. May 14.

7673. E. L. Mayer. Improvements in or connected with the treatment of sewage. May 16.

*C.—Disinfectants.*

5677. W. Radam. A fumigating composition for preserving and purifying purposes. April 15.

## COMPLETE SPECIFICATIONS ACCEPTED.

*A.—Chemistry of Foods.*

1889.

8945. J. L. Johnston. Preparation and combination of animal substances with vegetable substances for use in food. May 7.

10,352. G. A. Brinck and A. W. Rhenström. Food preparations. May 7.

*B.—Sanitary Chemistry.*

1889.

9763. G. Brownen and J. Gamgee. Bleaching, purifying, disinfecting, and preserving substances, and apparatus therefor. May 21.

9777. B. Nicholson and T. Palmer. Treatment of sewage, &c., for purification thereof, and obtainment of products therefrom. 21 May.

*C.—Disinfectants.*

1889.

8177. E. Hermite, E. J. Paterson, and C. F. Cooper. See Class VII.

16,369. J. Orchardson. Means and apparatus for disinfecting, destroying vermin, &c. May 7.

1890.

5677. W. Radam. A fumigating composition for preserving and purifying purposes. May 21.

## XIX.—PAPER, PASTEBOARD, Etc.

## APPLICATIONS.

6348. H. H. Lake.—From J. H. Carpenter and C. E. O'Hara, United States. Improvements in the manufacture of coloured paper. Complete Specification. April 25.

6458. P. Whiteside. A new or improved absorbent writing paper. April 28.

6644. J. Johnston and G. Johnston. Improvements in and relating to the treatment of paper-making fibre materials. April 30.

6993. C. Kellner. Improved apparatus for separating or disintegrating fibres in the manufacture of paper pulp. Complete Specification. May 6.

## COMPLETE SPECIFICATION ACCEPTED.

1889.

10,911. J. R. Thame and H. Sanguinetti. Manufacture of hollow ware from paper or like pulp, and apparatus therefor. May 14.

## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES, AND EXTRACTS.

## APPLICATIONS.

5940. H. H. Lake.—From La Société Anonyme des Parfums Naturels de Cannes, France. An improved method of and apparatus for extracting and purifying perfumes, oils, and other like substances. Complete Specification. April 18.

6620. F. Valentiner. A new manufacture of hydrocarbons and of derivatives which are applicable as perfumes or odoriferous bodies. April 20.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

11,099. T. Parker and A. E. Robinson. Manufacture of carbon bisulphide. May 14.

1890.

1988. F. L. Johnstone. Apparatus for filtering and derisining perfumes, tinctures, essences, &c. April 23.

5367. H. H. Lake.—From W. V. McKenzie and H. J. Braker. Refining apparatus for camphor, &c. May 21.

## XXI.—PHOTOGRAPHIC PROCESSES AND MATERIALS.

## APPLICATIONS.

5742. E. Fairweather. Developing photographic plates or other substances carrying a sensitive film. April 16.

6028. P. Rudolph. An improvement in photographic objectives. Complete Specification. April 21.

6029. E. Abbe and P. Rudolph. Improvements in photographic objectives. Complete Specification. April 21.

6066. B. Jumeaux. Improvements in or relating to developers for use in photography. April 22.

7453. A. G. Green, C. F. Cross, and E. J. Bevan. A process for photographing by means of organic compounds. May 13.

## COMPLETE SPECIFICATION ACCEPTED.

1889.

7747. W. F. Green. Rendering cardboard, paper, &c., impervious to the action of photographic chemicals, and suitable for photographic purposes. May 7.

## XXII.—EXPLOSIVES, MATCHES, Etc.

## APPLICATIONS.

5640. C. Watson. Improvements in electric safety fuses. April 14.

5948. G. Moffit. A mechanical time fuse for shells. April 19.

6614. W. Hurst. Improvements in the manufacture of wax matches, vestas, and tapers, and other similar goods. April 30.

6715. K. K. Malstrom. Improvement in percussion fuses. May 1.

6867. C. B. Levita. An improved percussion fuse. May 3.

7301. C. O. Lundholm and G. H. Hosie. Improvements in the manufacture of explosives. May 10.

7596. E. Turpin. Improvements in, and in apparatus connected with, the preparation and application of explosive substances for charging shells, torpedoes, mine cartridges, and the like. Complete Specification. May 15.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

1115. H. M. Chapman. Manufacture or treatment of explosives. May 14.

8718. Sir F. A. Abel and J. Dewar. Manufacture of explosives. April 30.

9361. A. V. Newton.—From A. Nobel. Preparation of explosives. May 7.

9433. W. B. McGavin.—From E. W. McGavin. Explosive compounds. April 30.

10,312. C. O. Lundholm. Manufacture of dynamite. May 14.

10,376. C. O. Lundholm and J. Sayers. Manufacture of explosives. May 14.

10,437. T. Parker and A. E. Robinson. Manufacture of phosphorus. May 7.

12,249. J. C. Butterfield and T. C. Batchelor. Explosive cartridges. May 21.

1890.

4243. W. Schuckher. Grained smokeless explosive. May 7.

# THE JOURNAL

OF THE

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## NOTICES.

In accordance with the provisions of Rule 18 of the Bye-laws, notice is hereby given that those Members of Council whose names are placed in italics in the annexed list will retire from their respective offices at the forthcoming Annual General Meeting.

Mr. E. Rider Cook has been nominated to the offices of President and Treasurer; and Sir Lowthian Bell, Bart., F.R.S., has been nominated Vice-President under Rule 11.

Mr. David Howard, Mr. B. E. R. Newlands, and Mr. J. C. Stevenson, M.P., have been nominated Vice-Presidents under Rule 8; and Mr. R. R. Tatlock and Mr. T. W. Stuart have been nominated Ordinary Members of Council under Rule 17, in the place of Mr. B. E. R. Newlands and Mr. J. C. Stevenson, M.P., nominated Vice-Presidents.

The Foreign Secretary has been nominated for re-election.

Mr. Alfred H. Allen, Mr. John R. Ashwell, Mr. H. Grimshaw, Professor R. Meldola, F.R.S., Mr. E. K. Muspratt, Mr. T. L. Patterson, and Mr. Boverton Redwood have been nominated, under Rule 18, to fill four vacancies among the Ordinary Members of Council.

## ANNUAL GENERAL MEETING.

Notice is hereby given that the next Annual General Meeting will be held in Nottingham on Wednesday, Thursday, and Friday, the 9th, 10th, 11th July next.

The following is a synopsis of the arrangements:—

## Wednesday, July 9th.

- |  |   |                     |
|--|---|---------------------|
| 1. Annual Meeting, University College  | - | 11 a.m.             |
| 2. Midland Railway Works, Derby; train leaves Nottingham   | - | 1.10 p.m.           |
| Or, Messrs. Morley's Hosiery Factory, Messrs. Turney's Leather Works, Messrs. Blackburn's Hosiery Machine Works, and Messrs. Lindley, Wright, and Cox's Lace and Hosiery Finishing Works | } | 2.30 p.m. to 4 p.m. |
| 3. Conversazione at Nottingham Castle  | - | 8 p.m.              |

## Thursday, July 10th.

- |  |   |                       |
|--|---|-----------------------|
| 1. The Burton Breweries; train leaves Nottingham   | - | 9.50 a.m.             |
| 2. The Butterley Iron Company; train leaves Nottingham   | - | 1.25 p.m.             |
| Or, The Birks Company's Embroidery Works, Messrs. Manlove's Engineering Works, The Nottingham Company's Hosiery Factory, and Messrs. Dobson's Lace Factory   | } | 10 a.m. to 11.30 a.m. |
| And Messrs. Lambert's Lace Dressing Rooms, The Midland Lace Company's Prilling Factory, Messrs. Doughty's Lace Factory, and Messrs. Morley's Hosiery Factory | } | 2.30 p.m. to 4 p.m.   |
| 3. The Dinner at the Exchange Hall   | - | 7.30 p.m.             |

## Friday, July 11th.

- |   |   |           |
|---|---|-----------|
| Excursion to Welbeck Abbey and Clumber; train leaves Nottingham | - | 8.20 a.m. |
|---|---|-----------|

Tickets of membership will be found with the Ballot Lists in this number of the Journal.

Members who require extra sets or back numbers of the Journal are requested to make application to the General Secretary only, to whom also changes of address should be communicated.

Notice is hereby given, for the information of members and advertisers, that the advertisement columns of this Journal have been contracted for by Messrs. EYRE and SPOTTISWOODE, the Society's printers and publishers, to whom all communications respecting them should be addressed.

The Secretary is instructed to negotiate for the purchase of copies of the Society's Journal for January 1883, and January, February, and April 1886. Members possessing odd copies of these numbers are particularly requested to communicate at once, stating price required, with Mr. Cresswell. The stock of all other numbers is at present sufficient for the Council's requirements.

## LIST OF MEMBERS ELECTED, 23rd JUNE, 1890.

- Baly, E. C. C., Bank of England, E.C., chemical student.  
 Banks, Jno. H., East 49th Street (corner 4th Avenue), New York, U.S.A., mining engineer.  
 Bradley, Edw. F., The Star Brush Co., Lim., Eden Grove, Holloway, N., engineer.  
 Bryce, Jno. Annan, Messrs. Wallace Bros., 8, Austin Friars, London, E.C., merchant.  
 Chorley, Jno. C., Lodge Lane, Bewsey, Warrington, chemical student.  
 Claypole, Dr. E. W., Buchtel College, Akron, Ohio, U.S.A., professor of natural science.  
 Crowder, S., Hillside, Auckland Road, Upper Norwood, S.E.  
 Ellershausen, Francis, Hebburn-on-Tyne, alkali manufacturer.  
 Foster, Jas., Baltic Chambers, Sunderland, consulting engineer, M.I.M.E.  
 Griffin, Jno. R., 22, Garrick Street, Covent Garden, W.C., chemical apparatus maker.  
 Halls, Claude W. G., 5, Carter Gate, Newark, Notts., chemical student.  
 Harmon, Lawrence E., c/o Edw. Elsworth, 71, Park Place, New York, U.S.A., analyst.  
 Heape, Chas., 19, George Street, Manchester, calico printer.  
 Hewlett, Jno. Cooke, 40—42, Charlotte Street, Great Eastern Street, E.C., manufacturing chemist.  
 Holmes, Geo. H., 9, Regent Street, New Basford, Nottingham, lace merchant.  
 Irving, Jos., Minas de Azualc6llar, Province de Sevilla, Spain, chemist.  
 Jarnain, Geo. Sales, Croft House, Marsh, Huddersfield, wool extractor.  
 Lord, Fred J., 150, Yorkshire Street, Rochdale, chemical student.  
 Mordle, F. Dare, Holloway House, Highbury Vale, Nottingham, starch manufacturer.  
 Nahusen, Dr. Rudolf, Hayle, Cornwall, technical manager, National Explosives Co., Lim.  
 North, E. Gordon N., Bella Vista, 14, Minas de Rio Tinto, Huelva, Spain, chemist.  
 Penn, Alf. Edw., 40, Westbury Street, Wandsworth Road, London, S.W., engineer.  
 Potter, E. C., 13, Circus Street, Greenwich, S.E., manure works chemist.  
 Selby, Wm., 113, Willoughby Street, Lenton, Nottingham, brewer.  
 Shaw, Herbert D., Castle Mount, Sandal, near Wakefield, analytical chemist.  
 Stillman, Dr. Thos. B., Stevens Institute of Technology, Hoboken, N.J., U.S.A., professor of analytical chemistry.  
 Sutherland, Alex., Ballynure-by-Ballyclare, co. Antrim, Ireland, mining engineer.  
 Taylor, Leo, Beechfield, Copeland Road, Walthamstow, E., analytical chemist.  
 Tompkins, H. K., 40, Torriano Avenue, Camden Road, N.W., chemist.  
 Twedy, Jas., 77, Western Road, Jarrow-on-Tyne, chemist and assayer.  
 Vult6, Hermann T., School of Mines, Columbia College, New York, U.S.A., assistant in analytical chemistry.  
 Wheeler, Wm. E., Cumberland House, Meynell Road, South Hackney, N.E., colour chemist.  
 Wilson, Alf., c/o Messrs. Jno. and E. Sturge, 18, Wheelleys Lane, Birmingham, chemical manufacturer.

## CHANGES OF ADDRESS.

Alexander, J. O., 1/o Demerara; 6, Sussex Terrace, Plymouth.

Bamber, H. K., Journals to 9, Victoria Street, Westminster, S.W.

Briggs, Wm., 1/o Arbroath; 4, Erskine Terrace, Dundee.

Carran, T. W., 1/o Shiel Road; 12, Rawlins Street, Liverpool.

Colby, W. H., 1/o Denbigh; Carregiven, Aberystwith.

Craw, Jno., 1/o Paisley; 15, Cadogan Street, Glasgow.

Eastlake, A. W., Journals to 17, Temperley Road, Balham, S.W.

Follows, F. W., 1/o Fairfield; Gorton, Manchester.

Gillman, Sr. Don Gustavo, 1/o Granada; Cantoria, Prov. de Almeria, Spain.

Hindle, J. H., 1/o Avenue Parade; 8, Cobham Street, Acerington.

Hunton, H., Journals to Greystone, Carlton, Ferryhill, co. Durham.

Laurie, A. P., 1/o Mile End Road; King's College, Cambridge.

McCulloch, Jas., 1/o Hebburn; Oakleigh, Rose Street, Garnet Hill, Glasgow.

Newton, H., 1, Glegg Street (not Slegg Street), Macclesfield.

Prinz, Dr. Otto, 1/o Schwarzenburg; Markt Redwitz, Bavaria.

Rademacher, H. A., 1/o Ohio; c/o Perkins, Goodwin, and Co., 66, Duane Street, New York, U.S.A.

Rideal, Dr. S., 1/o Forest Hill; Chemical Laboratory, St. George's Hospital, London, W.

Shearer, A., 1/o Boake and Co.; The Laboratory, 8, Hamfrith Road, Stratford, E.

Steedman, R. H., Journals to Sprengfield House, Dalnair, N.B.

Walsh, P. H., 1/o Cineionati; 13, Penny Street, Blackburn.

Williams, W. Collingwood, 1/o Grove Street; 44, Mulgrave Street, Liverpool.

Wright, J. C., 1/o Eglinton Street; Fern Bank, Irvine, N.B.

## CHANGES OF ADDRESS REQUIRED.

Kent, W. H., 1/o The Lodge, Buhmershe Road, Reading.

Manby, Eardley B., 1/o Halliwell, Bolton-le-Moors.

Walker, Jas. S. H., 1/o Morningside Road, Edinburgh.

## Deaths.

Lord Magheramorne, 17, Grosvenor Gardens, S.W.

A. E. Smith, Widnes, Lancashire.

## London Section.

## CHEMICAL SOCIETY'S ROOMS, BURLINGTON HOUSE.

Chairman: David Howard.

## Committee:

Sir P. A. Abel.

H. E. Armstrong.

W. Lant Carpenter.

W. Crowder.

W. J. Dibdin.

C. Graham.

S. Hall.

C. C. Hutchinson.

R. Messel.

B. E. R. Newlands.

B. Redwood.

T. Royle.

John Spiller.

Wm. Thorp.

C. R. Alder Wright.

Hon. Local Sec. and Treasurer: Thos. Tyrer, Battersea, S.W.

The names in Italics are those of members of Committee who retire at the end of the current Session.

The following have been elected to fill the vacancies and will take office in July next:—Chairman: T. Tyrer; Secretary: T. W. B. Mumford; Committee: J. Dewar, A. G. Green, C. W. Heaton, D. Howard, W. S. Squire, T. E. Thorpe.

## SESSION 1889-90.

1890.

July 9th, 10th, and 11th:—Annual General Meeting in Nottingham.

Meeting held on Monday, June 2nd, 1890.

MR. DAVID HOWARD IN THE CHAIR.

ON THE RESISTANCE OF OILS AND RESINS  
TO THE PASSAGE OF MOISTURE.

BY A. P. LAURIE, M.A.

THE experiments of Captain Abney and Professor Russell on water-colours have shown that many pigments fugitive under ordinary conditions are permanent in dry air, though exposed to sunlight. It is therefore of importance to know what mediums used for painting protect a pigment absolutely from moisture, and what allow moisture to pass through. This is of special importance in the painting of pictures when durability is required, but may also have some bearing on the commercial use of oils and resins to protect surfaces. My attention was the more directed to this subject, as I found receipts in 15th century MSS. for the preparation of such fugitive colours as Brazil wood lakes, which must, to account for the remarkable state of preservation of pictures by the old masters, have been carefully locked up from moisture. I determined therefore on some experiments to discover how far ordinary oils and varnishes could be held to protect a pigment from the action of moisture.

With a view to testing this, I ignited pure sulphate of copper, obtaining the white anhydrous sulphate. Using this as a pigment, I ground it with oil and painted it out on three glass slides. One I exposed to the air near an open window, one on the mantel-piece of a warm room, and one I placed in a desiccator. In 12 hours, the slide near an open window was green, the slide in the warm room was slightly green, and the slide in the desiccator was still white. On now exposing all three to the air near the window, they all turned completely green in another 12 hours. I had evidently here a delicate method of testing the permeability to moisture of such vehicles. I therefore experimented as follows:—The sulphate of copper having been ground with the vehicle to be tested, and painted out on a glass slide, it was next placed in a desiccator to dry.

When quite hard, it was then either placed under a bell-jar containing a dish of water or exposed to the air of the room. In most cases a duplicate slide was made and was kept in the desiccator for comparison. It would be tedious to recount all the experiments, and I merely give the results.

I first compared pale boiled oil, best copal oil varnish, amber dissolved in oil, amber dissolved in turpentine, common rosin in turpentine, and mastic in turpentine.

The amber in turpentine remained unchanged.

The other turpentine varnishes turned an opaque bluish-green.

The oil and oil varnishes turned a transparent pale green.

On next examining these slides under the microscope, a curious difference presented itself. In the case of the oil and oil varnishes, a uniform change had taken place, the copper sulphate being apparently completely hydrated, and here and there perfect crystals of sulphate of copper could be seen.

In the case, however, of the turpentine varnishes, the surface consisted of planes of unaltered sulphate, with cones of sulphate of copper crystals rising here and there on the surface, making the whole surface rough as seen under the microscope. In only one case out of three the amber in turpentine showed a similar appearance over part of the surface.

Apparently then, the resins when dissolved in a volatile oil, do of themselves protect the sulphate of copper from moisture, but tend to form holes and cracks through which moisture enters. Amber in turpentine, however, seems to be free from this tendency. On the other hand, linseed oil, whether mixed with resins or not, seems to be permeable to moisture over the whole surface with such completeness as not to cause the formation of rough projections of crystals.

It next seemed of importance to test how far the linseed oil when dry could be protected by coating with a resin dissolved in turpentine. I therefore prepared slides with linseed oil, and when dry, coated with (1) amber in turpentine, (2) copal in turpentine, (3) mastic in turpentine, (4) copal in oil. After drying and 12 hours' exposure to moisture, (1) and (2) were covered with green spots, but (3) and (4) unchanged. Under the microscope these spots were round in outline with sulphate of copper crystal cones filling them. In three or four days all the slides were green. Apparently, then, a resin in turpentine does not protect linseed oil, and the better the resin the more likely it is to be pulled into holes, doubtless owing either to capillary forces, or to different expansions of the two layers.

It seemed next of some importance to decide how far the preparation of the linseed oil would affect the result. I therefore obtained the best seed, had it picked over, cold pressed, refined over water in sunlight, and converted into boiled oil with borate of manganese. In some of this oil I dissolved amber, and in some copal. I also had a very fine specimen of commercial copal varnish, and two good samples of commercial boiled oil, Meguill, an artist's medium from a good maker, another pure linseed oil slightly differently prepared. These I tested in various ways, sometimes mixing a little varnish with the oil, sometimes using the varnish pure, sometimes allowing the slide to dry, then varnishing with the same medium and drying again before exposure.

None of these combinations resisted the action of moisture, nor did they differ greatly in the rapidity with which the change took place. On the whole the pure oil usually stood longer than the commercial oil, and the presence of a little varnish, especially amber varnish, seemed to preserve it a little longer still; but the results were uncertain, the method not lending itself well to comparative tests of this kind, as the thickness of the layer of oil of course affects the result.

One result of interest was, however, obtained, and that was that the power of linseed oil to resist moisture is immensely improved by keeping the slide for some time after the oil is dry, every week in the desiccator improving it in this respect.

As far, then, as the evidence goes, resins in volatile oils resist the passage of moisture in themselves, but tend to become full of holes and cracks, amber resisting best in this respect.

No vehicle containing linseed oil resists the passage of moisture unless possibly by long keeping. (This point is under examination.)

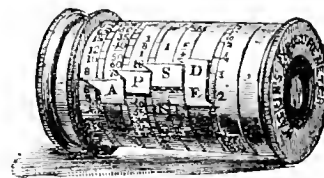
If such painters as Van d'Eyck had a medium which protected colours from moisture as apparently, judging from their pigments and pictures, they had, it remains to be discovered.

The method should prove useful in testing preparations meant to absolutely protect surfaces from moisture, with a view to finding how far they may be trusted to do so.

## A NEW EXPOSURE METER.

BY ALFRED WATKINS.

THIS instrument which I have invented for the calculation of photographic exposures is a combination of—1. an actinometer, for which I use a bromide paper sensitised with nitrite, according to Captain Abney's suggestion. II. a chain pendulum, beating half seconds. III. a set of calculating slide rules arranged so as to be worked in a mutually dependent series. The instrument deals with the several factors of photographic action in order, to each being appropriated a graduated brass ring, moving independently but in a common axis. These factors are



A the actinic force of the light falling upon the worst lighted part of the subject. P the sensitiveness of the plate used. S the capacity of the subject for reflecting the actinic rays. D the size of diaphragm, *i.e.*, its ratio to the focal length of the lens. These factors are reduced to numerical expression in a series of values representing their influence severally and independently upon the photographic action: the rings are graduated in terms of these values. The interdependence of these values is established on a basis which is necessarily empirical and to some extent arbitrary; this I have fully discussed in a paper read before the Hereford Photographic Society and published in the *British Journal of Photography* (April 18th and 25th, 1890). On this occasion I merely wish to call the attention of the Society to the instrument itself and to the photographs which I produce as evidence of the trustworthiness of its indications. The prints are from four negatives as follows:—

	Subject.	Duration of Exposure.
a.	Cloud picture.	$\frac{1}{2}$ second.
b.	Landscape.	4 seconds.
c.	Interior.	8 minutes.
d.	Interior (dark oak).	45 minutes.

the negatives being developed simultaneously in the same solution.

## DISCUSSION.

THE CHAIRMAN said that the question of correct exposure was one of vital importance to the practical photographer; and though the use of the little instrument now before them might render unnecessary much of the skill at present shown in the development of photographs, it might still, he thought, be well to substitute scientific certainty for that personal



skill which was at present shown by many artists in correcting the mischief done by inaccurate exposure. He understood that by the use of the apparatus now introduced to them photographers could rely on getting accurate exposures and thus greatly reduce the difficulties of development.

At the conclusion of the ordinary business of the meeting, Mr. W. THORR moved and Mr. S. HALL seconded the following resolution: "That the best thanks of the members of the London Section be and are hereby tendered to its Chairman, Mr. David Howard, for his close attendance in the chair and his courtesy in its occupation for the last six years."

The resolution was carried by acclamation.

Mr. HOWARD suitably replied.

Dr. L. T. THORNE then moved—"That the heartiest thanks of the members are due, and are hereby tendered, to Mr. Thomas Tyrer for his able conduct of the secretaryship of the London Section since the formation of the Society in 1881."

The resolution was seconded by Mr. DAVID HOWARD, who in doing so referred to the fact that Mr. Tyrer would succeed him in the chairmanship of the Section in the next session. This resolution also having been carried by acclamation,

Mr. TYRER replied.

#### DISCUSSION ON MESSRS. CROSS AND BEVAN'S PAPER ON THE CHEMISTRY OF HYPOCHLORITE BLEACHING. (This Journal, May 1890, 450.)

The CHAIRMAN said that modern science had no doubt made bleaching apparently effectual; but those who had had an opportunity of studying the wonderful collections of linen made by our grandmothers and great-grandmothers when they began housekeeping, were bound to confess that in these days one could not for love or money get new linen of similar quality. The fatal facility with which bleaching could be effected by bleaching powder had tempted the users thereof to ignore the fact that the process was not so simple as it appeared at first sight, and the thanks of the meeting were due to the authors for calling attention to the complexities of the process of bleaching. The authors had, he thought, shown an unnecessary amount of modesty in apologising for the incompleteness of their paper. It was not in the nature of things that such a paper could be perfect unless the authors had studied completely all the forms and data of bleaching. He agreed with them that it was far more useful to study the methods of investigation than every possible application of them. It was only by the minute study of the progress of the decompositions which occurred during the process of bleaching that each user of the agents employed could decide what was best suited for his own particular purpose.

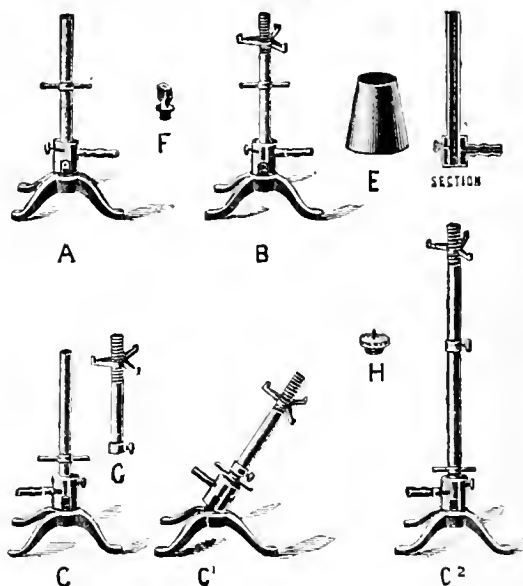
Dr. L. T. THORNE said that the paper abounded with points of great scientific and practical interest. He was unfortunately unable to follow the authors in some of their theoretical and experimental considerations, having been obliged up to the present time to confine his attention rather to the practical side of the question, and to leave to a later time the elucidation of several points of great interest which he had noticed in watching the ordinary operations of bleaching. There were two or three points, however, to which he would like to refer in connexion with Messrs. Cross and Bevan's work. In the first place, referring to their suggestion that the loss of chlorine was possibly due to the formation of chlorinated organic compounds of low atomic weight, it seemed to him highly probable that

such compounds were formed, especially in the earlier stages of the process. In all bleaching houses it was well known that chlorine compounds came off, and in watching carefully the bleaching of pulps such as were used for newspaper, he had himself noticed that when the bleaching had just begun, at the point when the change was most rapid, there was a very pungent odour, undoubtedly chlorinous in character, but equally undoubtedly due to something beyond chlorine or an oxide of chlorine. He was inclined to think that it was not even simple chloroform, but rather some other organic chlorine compound. Of course further complications arose from the fact that large quantities of either simple chlorine or its derivatives were liberated at the same time. That fact was clearly brought out in the course of his own experiments in passing air through pulp while undergoing the process of bleaching. The large quantities of nitrogen which passed away were found to be strongly impregnated with chlorine, and more bleaching powder was required when air was passed through than when no air was employed, though the oxygen of the air was used up, and so tended to reduce the quantity of chlorine required. Again, he thought that the presence and reactivity of the ketonic groups had much to do with bleaching. Experiments in which he had used oxygen in conjunction with bleaching powder, seemed clearly to show that such was the case. Oxygen alone did not cause any great amount of bleaching effect, but when used in conjunction with bleaching powder it showed a very marked effect. That, he thought, was undoubtedly due to its action on the aldehydic and ketonic groups existing in the compound celluloses or formed as intermediate products. The percentage of base in bleaches was another point of great importance. The way in which weak bleaching solutions were made from partially exhausted lime sludge was very questionable; in fact, the way in which the whole process of bleaching was often carried on was open to great improvement. There was, altogether, too much rule of thumb. Bleachers did not seem to consider that if they treated their partially extracted sludge with water there was a possibility that they might get a solution of a different character from that at first used. One point struck him as curious in connexion with the experiments dealing with the quantity of base not reverting to chloride, and that was the difference found in the magnesium hypochlorite made by double decomposition, as compared with that obtained by the electrolytic method. For whereas, as was shown on the board, from 13 to 16 per cent. of the former did not revert to chloride—i.e., 13 to 16 per cent. of chlorine acted in some way or other in a chlorinating manner—in the case of the latter there was no reversion whatever. That was a very curious difference, and he would like to know whether Messrs. Cross and Bevan had formed any opinion as to its cause. Chlorination was a most important point in the process of bleaching, and one that had been hitherto almost completely disregarded. He thought that the non-permanency of many bleached substances was largely due to that disregard of chlorination compounds, and that when bleaching was carried on under different conditions, the chlorination and the final result would be different also. The authors had shown that at a low temperature chlorination took place to a less extent than at a high temperature; and everyone who had experience of bleaching on a large scale well knew that when the process was carried on at a low temperature—at a sacrifice of some amount of time—the pulp obtained was more permanent in character, and had not that tendency to "go back" which was generally shown by pulps bleached at higher temperatures. We were now living in a time of high pressure, and the practical bleacher wanted to get as much pulp bleached in a day as possible. To that end, he pushed on his bleaching by means of high temperatures, in some cases working at 120° F. He got his bleaching done quickly, but there was a larger consumption of bleaching powder, and if he left his pulp for a short time—say 10 or 12 hours—before working it up, there would generally be a marked tendency to "go back." That effect was also due to the difference of action and chlorination of which he had already spoken; in fact, one of the most important points in bleaching was the avoidance, as far as possible, of that

strong chlorination of the cellulose compounds. Such a course was absolutely necessary in order to produce a good sound paper that would keep its colour, and at the same time not exercise that deleterious action on artistic colours which one heard so much about, and also to avoid a tendering effect on the paper itself. The actual isolation of the organic chloride was of great interest, as proving beyond doubt its presence in some of the bleached pulps. Altogether, the paper just read opened up a great many lines of thought, and would, he expected, be of great value, not only to scientists, but to practical bleachers; and if followed up should produce an improving effect on the bleaching of the future.

Mr. C. F. CROSS, in reply, reminded Dr. Thorne that he had expressly stated in reading the paper that he refrained from dealing with the question of bleaching efficiency; but Dr. Thorne having asked a question with respect to the difference in this respect of the magnesium hypochlorite prepared in the ordinary way and by the electrolytic method, he could state that nothing was more striking than the difference in the action of the two solutions in that particular ease. It was not only that the results shown by the numbers for proportion of residual base were obtained, but, if he might be allowed to use the expression, he would say that the electrolysed solution simply "went to pieces" on those particular pulps. It was exhausted with extreme rapidity, and to all appearance and up to a consumption of 3 percent. of chlorine produced no bleaching effect whatever. Upon well boiled pulps the reverse effect resulted; the electrolysed solution getting to work with much greater rapidity and effecting a considerable economy of chlorine. It would appear that chlorination of the pulp constituents was an advantage in one case and not in the other. This opened up a question which, as Dr. Thorne would see, could only be touched upon in the paper just read, namely, as to whether chlorination was not an essential factor in certain bleaching in *breaking down* the molecules. It was possible that chlorination might act in breaking down and resolving those complex ketonic bodies, and the resulting products become saponified in the after process, and so the effect of chlorination might not be evident from the study of the final products. Such points as these, however, would require special and very careful study. Mr. Bevan and he had aimed at but little more than indicating in their paper a general method of proceeding which, if followed up by other workers would, they hoped, in time result in a common understanding. If those interested in the subject would take the method in hand and make their own quantitative observations, it would not be long before they would have an important contribution, not only to practical bleaching but to theoretical chemistry. He knew that people in the habit of dealing with tons and gallons were apt to "shy" when talked to about molecular problems, and object to what they are pleased to call "long words." Investigators could not be always apologising for terms necessitated by the progress of science. If those who understood both the theoretical and practical sides of the question would agree (or even disagree) with them, and at least arrive at a common understanding as to the best way of thoroughly investigating this widely extended subject and analysing it down to its last terms, the object of their work would have been attained.

may be stated that in this new lamp the novelty is the simultaneous and corresponding regulation of the admission of gas and air to Bunsen burners. To this end the short vertical tube or jet used in ordinary Bunsen burners for the admission of gas into the outer tube (the lamp) is dispensed with, and the burner—or outer—tube is made to act as a regulating cock both for gas and air.



In order thus to serve as a regulating cock the extremity of the burner tube tapers to a conical shape, and is ground so as to fit the tube forming the base of the lamp and containing the air-holes so as to form in it a kind of plug cock, as a stopper does in the neck of a bottle. The gas enters the piece of metal tube, fixed at right angles to the burner tube, but at its extremity directly enters the burner by a horizontally disposed orifice, which on turning the burner tube is gradually closed. This arrangement obviates the objectionable "firing-back" of the flame when the supply of gas is reduced. Mr. Watson Smith's additions consist (1) in a telescope tube G, fixed around the burner tube of the ordinary lamp C, the effect being to make possible the raising or lowering of the position of the lamp-flame at will (*vide C''*); (2) in a hinge arrangement at the base, such that the lamp-tube may be inclined at any angle (as at C'), or placed in a perfectly horizontal position if desired; (3) in a cylindrical brass perforated shield placed around the base containing the air-holes, so that by revolving this around the base the air-holes are either gradually and at last entirely closed, or gradually and at last entirely opened, as the case may be, and with ease; and (4) in the addition of a gallery with chimney E, for protecting the flame. No. 2 addition is most especially useful in the process of soldering, or in the burning of filters over glazed paper in gravimetric analysis, &c. The telescopic arrangement (No. 1) is found very useful in fractional or ordinary distillations.

#### NEW AND IMPROVED FORM OF BUNSEN'S BURNER.

MR. J. R. GRIFFIN exhibited some new forms of Bunsen's lamp. The first, A, was the invention of G. Reimann, of Berlin, Eng. Pat. 20,744 of December 24, 1889, the others were modified forms of Reimann's lamp made by Mr. Griffin's firm (J. R. Griffin and Sons) at the suggestion of Mr. Watson Smith, and with the consent of Herr Reimann. Briefly it

## Glasgow and Scottish Section.

Chairman: R. R. Tatlock.

Vice-Chairman: Wm. Dittmar.

### Committee:

G. Beilby.  
Wm. Boyd.  
A. Crum Brown.  
J. Christie.  
W. S. Curphey.  
D. B. Dott.  
Wm. Foulis.  
J. Gibson.

R. Irvine.  
G. McRoberts.  
E. J. Mills.  
J. M. Milne.  
T. L. Patterson.  
E. Smith.  
E. C. C. Stanford.  
A. Whitelaw.

Hon. Treasurer: W. J. Chrystal.

Hon. Local Secretary:

Dr. G. G. Henderson, Chemical Laboratory, University of Glasgow.

The names in *Italics* are those of members of Committee who retire at the end of the current Session. The following have been selected to fill the vacancies, and will take office in July next:—  
Chairman: E. C. C. Stanford; Vice-Chairman: A. Crum Brown; Secretary: G. G. Henderson; Treasurer: W. J. Chrystal; Committee: W. J. A. Donald, C. A. Fawcitt, J. Falconer King, T. P. Miller, J. Pattison, and R. R. Tatlock.

### SESSION 1890.

Notices of Papers and Communications for the Meetings to be sent to the Local Secretary.

Meeting held in the Societies' Rooms, 207, Bath Street, Glasgow, on Tuesday, 3rd June 1890.

MR. J. PATTISON IN THE CHAIR.

## ON TRANSVAALITE—A NEW COBALT MINERAL.

BY THOS. B. MCGHIE AND JOHN CLARK, PH.D.

IN 1889 Mr. McGhie, who had occasion to be in the Transvaal, took the opportunity of paying a visit to a cobalt mine about 30 miles north of Middleburg, and brought away with him a number of specimens.

The ore of this mine, which consists essentially of arsenide of cobalt or smaltine, occurs in a vein of quartzite, and was worked for several years, when it was abandoned, owing, probably, to the cost of transit and the difficulty of separating the ore from its matrix.

A sample of this ore was found on analysis to have the following composition:—

	Per Cent.
Arsenide of cobalt .....	57.39
Arseniate of cobalt .....	8.27
Sulphide of cobalt .....	1.02
Sulphide of nickel .....	0.38
Sulphide of iron .....	None.
Siliceous matter .....	30.55
Combined water .....	1.46
Moisture .....	0.52
	<u>99.59</u>

From the above analysis it will be observed that the ore itself, apart from the matrix, is remarkably pure, but although the vein at some parts is several inches in thickness, the greater part of the ore is distributed throughout the matrix in such a manner that it can only be separated by washing appliances.

The specimens on the table show the appearance of the ore at different parts of the vein.

In the same rock, at a short distance from the vein of arsenide, there is another vein of a different kind of cobalt

ore, varying in width from about 2 inches to a thin streak, which, we understand, was followed for a short distance in search of gold, and then abandoned. This is the mineral which forms the subject of our communication. Where the vein is wide the ore is black in colour, with reddish-brown patches, and somewhat nodular in its external appearance, but where the vein is thin the ore is black, and has a slaty fracture. It dissolves in hydrochloric acid readily, with the evolution of a great deal of chlorine, and gives a solution which is bluish-green when concentrated, but pink when diluted.

The nodular portion, of which I show you a specimen, and of which the greater part of the sample was composed, was found on analysis to have the following composition:—

	Per Cent.
Sesquioxide of cobalt .....	65.80
Protoxide of cobalt .....	3.82
Oxide of nickel .....	0.15
Arsenic acid .....	5.79
Sesquioxide of iron .....	2.41
Alumina .....	2.68
Manganese dioxide .....	None.
Lime .....	0.40
Magnesia .....	0.30
Sulphuric acid .....	..
Silica .....	6.35
Water .....	<u>12.19</u>
	<u>99.89</u>

	Per Cent.
Cobalt .....	49.54
Specific gravity .....	3.846
Hardness .....	4.0
Streak .....	Brownish black.
	Per Ton.
Gold .....	1 oz. 12 dwt.

The percentage of sesquioxide of cobalt was determined from the chlorine evolved by heating with HCl, and found to be equivalent to 6.42 per cent. of oxygen, which has been assumed to be in the form of  $\text{Co}_2\text{O}_3$ , and the water was ascertained by igniting a weighed portion of the mineral and deducting the oxygen given off from the loss.

The results which we have obtained indicate that this mineral is essentially a hydrated sesquioxide of cobalt with 7.72 of arseniate of cobalt ( $3\text{CoO} \cdot \text{As}_2\text{O}_5$ ), and if the oxide of iron exists in the form of hydrated arseniate of iron, which is probably the case, there is just about enough arsenic acid remaining, after calculating the protoxide of cobalt to arseniate, to form  $2\text{Fe}_2\text{O}_3 \cdot \text{As}_2\text{O}_5$  (iron sinter).

3.82 of $\text{CoO}$ requires .....	3.90 $\text{As}_2\text{O}_5$
2.41 „ $\text{Fe}_2\text{O}_3$ „ .....	1.73 „
	<u>5.63</u> „

With the exception of wad, or earthy cobalt ore, which is regarded as consisting essentially of  $\text{CoO}$ ,  $\text{MnO}_2$ , the only known cobalt mineral which so far as we are aware resembles Transvaalite is Heubachite, a natural hydrated oxide of cobalt and nickel, described by F. Sandberger in 1877 (Journ. Chem. Soc., Vol. II., p. 855) as having the following composition:—

	Per Cent.
$\text{CoO}$ .....	65.50
$\text{NiO}$ .....	14.50
$\text{Fe}_2\text{O}_3$ .....	5.13
$\text{MnO}_2$ .....	1.50
$\text{H}_2\text{O}$ .....	<u>12.59</u>
	<u>99.22</u>
Specific gravity .....	3.44
Hardness .....	2.5

Heubachite is found in extremely thin soot-like deposits in clefts in barytes, and is said to evolve a good deal of chlorine with HCl. The cobalt is nevertheless represented by Sandberger as existing entirely in the form of protoxide of cobalt ( $\text{CoO}$ ), and, if the analysis is correct, this mineral

cannot contain more than a small quantity of sesquioxide of cobalt ( $\text{Co}_2\text{O}_3$ ), and it differs essentially from Transvaalite by containing manganese and a large proportion of oxide of nickel but no arsenic acid. It is also considerably lower in specific gravity and hardness. The occurrence of Heubachite is attributed to the action of water which contained the constituents of the mineral in the form of carbonates, and we think it is possible that Transvaalite has been produced in a similar manner. The occurrence of arsenide and arseniate of cobalt in the same rock veined in exactly the same way, combined with the presence of arseniate of cobalt in the Transvaalite, has led us to consider whether that mineral might not be a product of the decomposition of the arseniate of cobalt, and for this purpose we have analysed the rock itself, which in the neighbourhood of the vein is white, with here and there a brown film of oxide of cobalt, but at other places it contains purple patches of arseniate of cobalt.

#### ANALYSIS OF THE ROCK.

	Ignited Rock.
	Per Cent.
Silica.....	82.20
Alumina .....	11.40
Oxide of iron.....	Trace
Lime.....	..
Magnesia.....	..
Potash .....	0.60
Soda .....	0.04
Oxide of cobalt.....	2.80
	100.04
Loss on ignition .....	0.72

With the exception of the potash, there is nothing in the rock which would be likely to decompose the arseniate of cobalt, and the quantity is so small that we do not feel justified in concluding that the mineral has been formed from the arseniate by the action of the alkali of the rock and atmospheric influences; but at the same time there are so many circumstances in favour of this theory, that we think it is only proper to mention it as a possible method of formation.

#### DISCUSSION.

Dr. CLARK, in reply to questions, said he quite appreciated the view of those who looked at this mineral not from the scientific but from the commercial standpoint. He understood that the mine was a very long distance from the coast. The carriage some time ago was about 14*l.* per ton, but even 14*l.* should not interfere with its utilisation, as he supposed a mineral of this kind was worth more than twice that sum. The rock had apparently been worked for a short time, more with the view of getting gold than cobalt, and the Cobalt Company who opened up the mine evidently found that taking into account the carriage and difficulties of separation, &c. it would not pay, and the result was that a year ago the mine was standing idle. Even taking all this into consideration, however, he was satisfied that there was a sufficient quantity of the ore, with the present prices of cobalt and with proper machinery, to pay the cost of export to this country.

#### ON THE REVISION OF CONSTANTS EMPLOYED IN THE ANALYSIS OF FATS AND OILS.

BY R. T. THOMSON, F.I.C., AND H. BALLANTYNE.

THERE are certain chemical and physical constants, such as the iodine absorption and the specific gravity, which are of great importance in the analysis of oils and fats. In the present state of this branch of chemical analysis, the values assigned to these in books ought always to be revised

by chemists undertaking oil analyses. Our experience, at least, has been that while the specific gravities and alkali-neutralising powers of oils are as correct as the somewhat varied character of different specimens of the same oil will allow, the iodine absorptions of some oils are certainly understated.

But besides this, the ordinary arrangement of constants in books on oil analysis, as for instance in that useful collection of facts in Allen's "Commercial Organic Analysis," Vol. II., is not perhaps the most desirable. There is one table of specific gravities, another of potash-neutralisation equivalents, and a third in which the iodine absorptions are recorded. In such tables we have no means of knowing the relation between the different constants assigned to a given sample. Thus, we do not know whether an olive oil of low gravity has a higher iodine absorption than one of high gravity, or *vice versa*. Our second object, then, has been to give the specific gravity, potash-neutralisation equivalent, and iodine absorption of each specimen of oil tested. Of course it may be objected that other constants were not determined, such as Maumené's temperature test, but we have preserved the samples so that these and other values may be dealt with in a future paper.

Before proceeding further it may be noted that the results of our determinations are embodied in the accompanying table, in which the first point for consideration is the specific gravity of oils.

*Specific Gravity of Oils.*—The specific gravities which we have determined agree substantially with those already published, but allusion may here be made to the effect of the presence of free fatty acid in oils. It has been observed by Archbutt that olive oils of low density are high in free oleic acid. On this observation Allen has built the theory that 5 per cent. of free acid diminishes the specific gravity of the sample by 0.7. But this result is based merely on taking the highest density (917) observed by Archbutt as that possessed by pure olive oil, and assuming that variations in density are entirely due to free oleic acid. As a matter of fact such is not the case, as will be seen from the table, where an olive oil with 3.86 per cent. of free acid has a specific gravity of 914.8, while another with an almost identical density (914.7) contains 23.78 per cent., and yet another possessing a high specific gravity (916.8) contains as much as 5.19 per cent. of free acid. In the face of these results it is plain that no faith can be placed in the formula for altering the specific gravity of an oil according to the proportion of free acid contained in it. To make perfectly certain of this conclusion a sample of olive oil (sp. gr. 915.6) was treated with a solution of caustic potash in a separator, the soap solution run off, the oil washed with water and filtered through a dry filter. The density of the acid-free oil thus prepared was only 915.2, so that the free oleic acid (9.42 per cent.) appeared to have actually raised the specific gravity of the sample in the proportion of 0.2 for each 5 per cent. Free fatty acids prepared from the same oil had a specific gravity of 900.8 at 20.5° C. (compared with water at 15.5°), while the acid-free oil showed 911.8 at the same temperature. Assuming that the rate of expansion is the same for each, and that the fatty acids would be liquid at 15.5°, the specific gravity of the latter would stand at about 904. We have not had time to repeat these experiments with other oils, and confirmation is certainly desirable; but it would appear that we must not regard oils as mixtures of neutral oil with free fatty acid. This is consistent with theory, for it is generally conceded that high acidity is caused by the oil becoming rancid, or in other words, by the decomposition of the glyceryl oleate, etc. When this is taken into account it is not altogether surprising to find that the acid-free oil possesses a less density than the same oil after having become rancid.

*Potash-neutralising Power of Oils.*—There is nothing special to remark on this portion of the subject, except that our results are in unison with those of other observers.

*Iodine Absorption of Oils.*—Nübl's iodine absorption test seems to have almost entirely superseded the bromine absorption test, and it is in many cases the best datum on which the percentage of adulterant in any oil may be calculated, after the fact of adulteration has been proven

beyond doubt. It is therefore an imperative necessity to have this constant accurately determined. Hübl directed that absorption should be allowed to go on for two hours, but Archbutt has added to this two important suggestions: (1) that at least double the amount of iodine absorbed ought to be present; and (2) that the absorption should be allowed to go on for six hours. Our experience is that the first of these suggestions must be strictly adhered to, and that the second may be safely extended to at least eight hours. We have found that the absorption of iodine by lard oil is practically complete in about three hours, but that linseed and seal oils require fully eight hours. This will be apparent from the following table:—

RATE OF ABSORPTION OF IODINE PER CENT.

Time of Absorption.	Seal Oil.	Linseed Oil.
Two hours .....	136.6	175.5
Four hours .....	140.8	179.7
Six hours.....	145.1	184.1
Eight hours .....	145.8	187.7

When the time was extended to 18 hours no further absorption took place. In view of these facts we have found it a very good plan, to start the absorptions in the evening, and titrate the solutions on the following morning. A blank experiment should be made in every case with the chloroform and iodine solution employed.

As will be seen from the table appended, our results agree as a rule with those of Hübl, Moore, and Archbutt, given in Allen's "Commercial Organic Analysis," but in a few cases there are large variations. Thus, menhaden oil is given as 147.9, while our results show it to be as high as 160. Again, the highest recorded iodine absorption for raw linseed oil is stated at 160, while our results vary from 168 to 187.7 for different qualities of the oil. We have found two specimens of olive oil which show only 79 per cent. of absorption, this being 2.6 per cent. below the lowest recorded result, and one sample absorbing 86.4 per cent., which is 1.4 per cent. higher than any mentioned by Allen. It may also be noted here that iodine values calculated from Mills' bromine absorptions must not be accepted as correct for iodine, as seal oil, for example, is some 50 per cent. too low.

*Relation of the Constants to each other.*—If we examine the figures for lard given on the table, we find that the specific gravity is high when the iodine absorption is high, and *vice versa*. These lards were rendered by ourselves,

and can therefore be guaranteed genuine. We found that the whole of the lard contained in the portion of the pig operated upon had to be melted out before an average sample could be got. When only a portion was rendered, a soft lard was obtained, which had a high specific gravity and iodine absorption. The facts stated and observations made on other lards received for analysis go to show that these two constants have a definite relation to each other, and by interpolations we may arrive at the following relationship between the specific gravity and iodine absorption:—

TABLE OF RELATIONSHIP BETWEEN THE SPECIFIC GRAVITY AND IODINE ABSORPTION OF LARD.

Specific Gravity at 99° C. (Water at 15.5° C.= 1,000.)	Iodine Absorption.
	Per Cent.
859.8	52.1
859.9	53.4
860.0	54.7
860.1	56.0
860.2	57.3
860.3	58.6
860.4	59.9
860.5	61.3
860.6	62.5

This shows an increase of 1.3 per cent. for 0.1 increase in the specific gravity, and it will be observed that beef fat follows the same rule, as nearly as possible. Cotton-seed oil, the other adulterant of lard, would show only an increase of 0.6 per cent. of iodine absorption for an increase of 0.1 in specific gravity. These data may be utilised, if substantiated by other observers, in the analysis of lards, and especially of those samples containing cotton-seed oil which do not answer to the colour tests.

The only other oils of which series were tested were linseed, rape, seal, and olive oils, but the specific gravities and iodine equivalents of these do not appear to have any direct relationship. It is quite possible that a fuller examination of the relations between different constants may tend to throw considerable light on oil analysis, and this and other parts of the subject it is our intention to follow up.

TABLE OF CONSTANTS IN THE ANALYSIS OF OILS.

Nature of Oil or Fat.	Sp. Gr. at 15.5° C. (water at 15.5° = 1,000).	Sp. Gr. at 99° C. (water at 15.5° = 1,000).	Iodine Absorption.	KOH Neutralised.	Free Acid.
			Per Cent.	Per Cent.	Per Cent.
Olive (Gioja) .....	915.6	.. ..	79.0	19.07	9.42
Olive (Gioja) after removal of free acid .....	915.2	.. ..	79.0	19.07	None.
Olive .....	914.8	.. ..	83.2	18.93	3.86
Olive .....	914.7	.. ..	80.0	.. ..	23.78
Olive .....	916.8	.. ..	83.1	19.00	5.19
Olive .....	916.0	.. ..	81.6	.. ..	19.83
Olive (for dyeing) .....	915.4	.. ..	78.9	19.00	9.67
Olive .....	914.5	.. ..	86.4	18.90	11.28
Olive (for cooking) .....	915.1	.. ..	83.1	19.20	4.15
Olive (for cooking) .....	916.2	.. ..	81.2	19.21	Not done.
Lard (from omentum).....	.. ..	859.8	52.1	.. ..	.. ..
Lard (from leg) .....	.. ..	860.5	61.3	.. ..	.. ..

TABLE OF CONSTANTS IN THE ANALYSIS OF OILS—*continued*.

Nature of Oil or Fat.	Sp. Gr. at 15.5° C. (water at 15.5° = 1,000).	Sp. Gr. at 99° C. (water at 15.5° = 1,000).	Iodine Absorption.	KOH Neutralised.	Free Acid.
			Per Cent.	Per Cent.	Per Cent.
Lard (from ribs) .....	.. ..	860.6	62.5	.. ..	.. ..
Beef fat (from suet) .....	.. ..	857.2	34.0	.. ..	.. ..
Beef fat (oleomargarino) .....	.. ..	858.2	46.2	.. ..	.. ..
Fat from marrow of ox.....	.. ..	858.5	45.1	19.70	.. ..
Fat from bone of ox.....	.. ..	859.2	47.0	19.77	.. ..
Cotton seed.....	923.6	868.4	110.1	.. ..	.. ..
Cotton seed.....	922.5	.. ..	106.8	19.35	0.27
Linseed (Baltic).....	934.5	.. ..	187.7	19.28	.. ..
Linseed (East India) .....	931.5	.. ..	178.8	19.28	.. ..
Linseed (River Plate) .....	932.5	.. ..	175.5	19.07	.. ..
Linseed.....	932.5	.. ..	173.5	19.60	0.76
Linseed.....	931.2	.. ..	168.0	19.60	.. ..
Rape .....	916.8	.. ..	105.6	17.53	2.43
Rape .....	913.1	.. ..	110.7	17.38	.. ..
Rape .....	914.5	.. ..	104.1	17.06	2.53
Rape .....	915.0	.. ..	104.5	17.19	3.10
Rape .....	914.1	.. ..	100.5	17.39	.. ..
Castor (commercial).....	967.9	.. ..	83.6	18.02	2.16
Castor (commercial).....	965.3	.. ..	.. ..	17.86	.. ..
Castor (medicinal) .....	963.7	.. ..	.. ..	17.71	.. ..
Arachis (commercial) .....	920.9	.. ..	98.7	19.21	6.20
Arachis (French refined) .....	917.1	.. ..	98.4	18.93	0.62
Lard oil (prime).....	917.0	.. ..	76.2	.. ..	.. ..
Southern sperm .....	880.8	.. ..	81.3	18.25	.. ..
Arctic sperm (bottle-nose).....	879.9	.. ..	82.1	18.04	.. ..
Whale (crude Norwegian).....	920.8	.. ..	109.2	.. ..	.. ..
Whale (pale) .....	919.3	.. ..	110.1	.. ..	.. ..
Seal (Norwegian).....	925.8	.. ..	152.1	.. ..	.. ..
Seal (cold drawn, pale) .....	926.1	.. ..	145.8	19.28	.. ..
Seal (steamed, pale) .....	924.4	.. ..	142.2	18.93	.. ..
Seal (tinged) .....	925.7	.. ..	152.4	.. ..	.. ..
Seal (boiled).....	923.7	.. ..	142.8	.. ..	.. ..
Menhaden.....	931.1	.. ..	160.0	18.93	.. ..
Newfoundland cod .....	924.9	.. ..	160.0	.. ..	.. ..
Scotch cod .....	925.0	.. ..	158.7	.. ..	.. ..
Cod-liver (medicinal).....	926.5	.. ..	166.6	18.51	0.86
Mineral .....	873.6	.. ..	12.8	.. ..	.. ..
Mineral.....	886.0	.. ..	26.1	.. ..	.. ..
Rosin .....	986.0	.. ..	07.9	.. ..	.. ..

## DISCUSSION.

The CHAIRMAN said this was a paper which was difficult to lay hold of on hearing read, but he had no doubt that in print it would be very useful. Anything which gave information as to how to detect mixtures of oils was sure to prove valuable. He supposed that in regard to these constants one of the chief difficulties was to get pure samples of oil in order to form a basis. It seemed to be the practice for various reasons (not always to decrease the value) to mix oils.

Dr. CLARK asked if in giving the iodine absorption, the authors took samples of the whole fat of the pig or only of different parts, and whether they had done anything with the fat of the feet? There was considerable difference in the properties of the fat from different parts of the pig which affected the results very materially. When getting a sample of lard to examine, the great difficulty lay in ascertaining from what part of the pig the sample was taken. It might be the refuse from one part or it might be from the whole animal, so that the results must often of necessity

vary very much. As to the method adopted, however, it was an excellent one, and he was sure that the table given would be found very serviceable to public analysts, who had great difficulty in determining what was genuine and what was not.

Mr. BALLANTYNE, in reply to Dr. Clark, said that the first portion of lard rendered had a very high specific gravity and iodine absorption, and the portion rendered last had the lowest. They found that the lard from the omentum gave the lowest gravity and absorption, and that from the legs and ribs the highest, the latter of which were practically identical.

## THE PURIFICATION OF ARTIFICIAL SALICYLIC ACID.

BY G. G. HENDERSON, D.S.C., M.A., UNIVERSITY OF GLASGOW.

WITHIN the last few years the employment of salicylic acid, both as an antiseptic and for pharmaceutical purposes, has become very general, and the quantity of this substance manufactured for various purposes is now very considerable. There is a pretty large difference between the price of the "natural" acid obtained from oil of wintergreen or from salicin and that of the "artificial" acid synthetically prepared from phenol, and the natural result is that the artificial is used instead of the natural acid for many purposes. It has been observed, however, that the administration of the former (usually as a sodium salt) as a medicine is often followed by disturbances more or less severe, and sometimes of a grave nature, and it has recently been proved that these serious after-effects are due to the presence of impurities in the artificial acid and are not produced by use of the natural acid.

Professor Charteris of Glasgow University has carried out a series of comparative experiments on rabbits, using on one hand the natural acid and its sodium salt, and on the other hand a number of specimens of the artificial acid and its sodium salt which were obtained from various sources. The results of these experiments may be summarised as follows:—

Salicin, in 30-grain doses, natural salicylic acid, in 10-grain doses, and sodium salicylate (from the natural acid) in 32-grain doses, have no deleterious effect on rabbits weighing  $2\frac{1}{2}$  lb.

Artificial salicylic acid, in 10-grain doses, and sodium salicylate (from the artificial acid) in 18-grain doses, in ordinary use, cause the death of rabbits weighing  $2\frac{1}{2}$  lb. Even the very best specimens of the artificial acid (sold as "chemically pure"), in 15-grain doses, proved fatal to one rabbit, and in two others caused marked prostration and paralysis, but not death; while with less pure specimens a fatal result followed the administration of the acid in every case.

From these experiments it is evident that serious objections may be raised to the use of ordinary artificial salicylic acid for medical purposes, but the comparatively high price of the natural acid makes the production of a pure artificial acid very desirable. I therefore, at Dr. Charteris' request, made a number of experiments with the view of testing the possibility of so purifying commercial salicylic acid that it may be used with safety instead of the natural acid, and after trying various methods I find that the purification can be effected simply and economically by the process suggested by Williams some years ago (*Pharm. J.* 3rd S. 8, 785). A hot aqueous solution of the acid is neutralised with calcium carbonate and allowed to cool, when calcium salicylate, which is sparingly soluble in cold water, separates out in hard glistening crystals. When the best specimens of the commercial acid are used the crystals of the calcium salt are well formed and almost colourless, or only slightly tinged with brown, while inferior qualities of the acid yield a mass of indistinct brown crystals of the calcium salt. A further quantity of the salt, of course in a

more impure state, is obtained on concentration of the mother-liquor, and the greater part of the impurities is contained in the last mother-liquor, which is usually rather thick and of a dark reddish-brown colour. The calcium salt is recrystallised from hot water once, or oftener if necessary, and finally decomposed by hydrochloric acid, and the salicylic acid thus set free is washed with cold water and crystallised from dilute spirit. It is now obtained in large well-formed prismatic crystals, which are exactly similar in appearance to the crystals of the natural acid, and the melting point, solubility, and other properties of the two substances are exactly the same. The physiological action of the purified acid was investigated by Dr. Charteris with satisfactory results, for it was found that after administration of the acid in 15-grain doses there was "no paralysis, not even depression, after the last injection; rabbit able to run easily."

The impurities found in commercial salicylic acid are chiefly cresotic acid, which results from the use of impure phenol in the manufacture, parahydroxybenzoic acid, and hydroxyisophthalic acid. The presence of the two latter substances is due to the use of caustic soda containing potash or to undue raising or lowering of the temperature during the manufacture, as has been shown by Fischer in an interesting paper (*J. Pharm.* [5], 20, 258), in which methods for estimating the amount of the impurities in salicylic acid are also described. None of the specimens which I examined contained more than a very small quantity of impurity; this was separated from the last mother-liquor by addition of hydrochloric acid, conversion of the precipitated acids into calcium salts, and fractional crystallisation of the latter, and consisted principally of cresotic acid (m.p.  $151^{\circ}$ ). It was found that one grain of this impurity was sufficient to cause the death of a rabbit weighing 2 lbs.

It is troublesome to detect the presence of the impurities in the better specimens of commercial salicylic acid by direct tests, for the quantity of these impurities is usually too small to exercise an appreciable influence on the melting point or the solubility of the acid, but the crystalline form of the acid is a reliable indicator of its purity. As stated above, the pure acid crystallises from dilute spirit in large and distinct crystals, but the presence of even a very small quantity of impurity causes the acid to crystallise in a network of delicate closely interlacing needles.

It is therefore a comparatively simple matter to manufacture a pure salicylic acid—indeed I understand that this is now being done—and in view of the dangers attending the use of an impure acid it seems reasonable that some measures should be taken to ensure the purity of the acid used for pharmaceutical purposes or in the preparation of preservatives of different kinds.

## ERRATUM.

In the May number of this Journal, page 473, line 35 from bottom of column 1, instead of "This smokeless powder has been adopted by," read "This smokeless powder has been adopted for trial by."

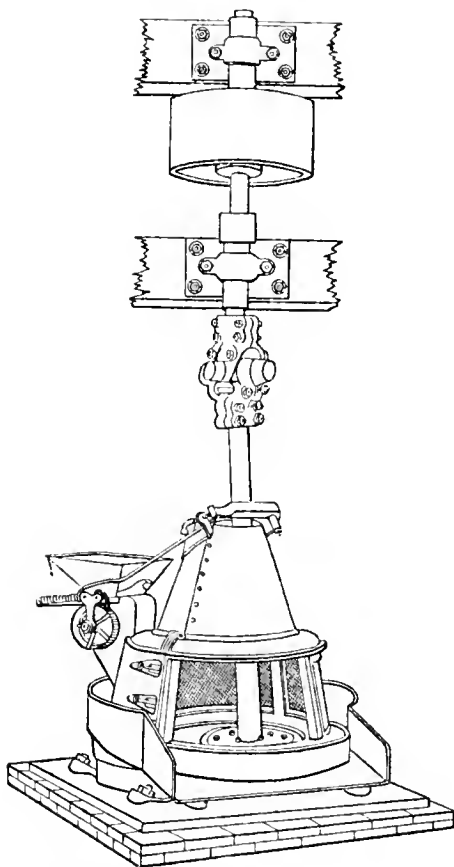


## Journal and Patent\* Literature.

### I.—GENERAL PLANT, APPARATUS, AND MACHINERY.

*The Griffin Roller Mill.* Eng. and Mining J. 49, 589.

THIS mill, designed for the pulverisation of wet or dry ores, phosphates, cements, carbon, and hard and refractory substances, receives its motive power from the vertical shaft which runs in two self-adjusting journal bearings bolted to the timbers in the mill building, or, independently of this, to a specially devised timber or iron frame. The step bearing which carries the weight of all the running parts is fastened to the lower journal box and may be raised or lowered as the accurate adjustment of the working parts of the apparatus requires. A smoothly running universal joint connects the perpendicular shaft at its lower end with the roller shaft; to the latter is secured the cylindrical crushing roll, which on the revolution of the shaft moves freely about in a ring or die (weighing 260 lb. and encased in a base or pan), and by centrifugal force presses against the inner surface of the ring.



\* Any of these specifications may be obtained by post, by remitting the cost price, *plus* postage, to Mr. H. Reader Lack, Comptroller of the Patent Office, Southampton Buildings, Chancery Lane, London, W.C. The amount of postage may be calculated as follows:—

If the price does not exceed 8d. ....	½d.
Above 8d., and not exceeding 1s. 6d. ....	1d.
" 1s. 6d., " " 2s. 4d. ....	1½d.
" 2s. 4d., " " 3s. 4d. ....	2d.

When the material to be pulverised, after crushing in a rock breaker to 1 in. cubes, is introduced into the pan through an automatic feeder, it is stirred up by the 12 lb. shoes or plows attached to the lower side of the roll, and then is thrown against the ring and crushed by the roll. When sufficiently fine the pulverised material passes through screens at the base. It is then carried off, in a special conveyor, through openings in the bottom of the pan outside of the screens. The screening arrangement operates so efficiently as to give passage to four tons an hour of phosphate rock, 90 per cent. of which would pass through a 60-mesh screen. A fan, attached to the shaft above the roll and cased in with it, forces air through the screens into the discharge and keeps the dust within the mill.

For wet grinding, a trough is substituted for the openings in the bottom, and the fan is omitted; otherwise the construction is the same.

The mill is said to grind from 1½ to 4 tons per hour, at 150 to 175 revolutions per minute, according to the character of the material, consuming about 20 horse-power, though an engine rated at 25 horse-power is preferable to one of nominal 20 horse-power. The even distribution of the material to be acted on, its quick discharge when finished, and the uniformity of the work as to fineness, are indicated by the fact that 37 tons 400 lb. of Charleston phosphate rock were pulverised in nine hours, and, when sampled and tested with standard screens, showed 91·5 per cent. able to pass through a 60-mesh and 84 per cent. through an 80-mesh.

The mill occupies 4½ sq. ft., weighs 6,000 lb., and requires but little attendance. The repair is so slight that, in a certain case 1,500 tons of Charleston rock were ground without causing a measurable wear. The wearing parts, *i.e.*, the ring, the tire of the roll (weighing 100 lb.), and the shoes, are easily removed and replaced. The heaviest piece is the pan (2,400 lb.), which could be sectionalised. Outside of this no part weighs over 300 lb. The journal bearings are several feet away from the mill, run cool, and consume about half a pint of oil for 10 hours. The vibration of the apparatus, when based on a good foundation, is light. (Compare this Journal, 1889, 196.)

### PATENTS.

*Improvements in Apparatus for Separating Liquids from Solid Matters Suspended therein.* H. J. Hadden, London. From Gaillet and Huët, Lille, France. Eng. Pat. 972, February 22, 1883. (Second Edition.) 8d.

THIS invention has for its object the separation of solid particles from the liquids in which they are suspended, and is particularly applicable for the clarification of water used for feeding boilers, or other purposes.

The apparatus consists chiefly of a vertical vessel containing a number of parallel partitions, inclined at an angle of about 40° and alternately riveted to the opposite sides of the vessel. These partitions are also inclined sideways at an angle of 45°, and form a series of compartments. The mixture of solid and liquid matter is caused to flow alternately upwards and downwards between the inclined plates, and sufficient space is allowed for the solid particles which glide over the inclined partitions to settle at the bottom of the channels formed by the plates, and from which they can be easily removed. In removing impurities from water by means of chemical precipitation, the chemicals to be dissolved are placed in a vessel near the separating apparatus, both vessels being supplied with water from the same tank through two outlets and two adjustable valves. The proportion between the liquid to be purified and the dissolving liquid can thus be regulated and kept constant.—E. S.

*Improved Method of and Means for Separating Solids from Liquids.* W. Hucks, London. Eng. Pat. 6391, April 15, 1889. 6d.

IN this patent, which consists of improvements on Eng. Pat. 2528 of 1889 (this Journal, 1889, 694), centrifugal force is used to separate the solid matter from the liquid.

A hollow circular chamber is formed in two parts, which can be connected together at their circumference by a water-tight joint, but can be opened or closed at will whilst in motion by a lever attached to the upper half. Inside is a hollow disc, having inlets and outlets, and is rotated by a shaft independently of the chamber, and is provided with perforated plates or wire gauze to intercept the solid matter. Other perforations or diaphragms are also used elsewhere. The liquid enters through a hollow spindle, rotating in a gland, at the bottom of the chamber, and escapes through an outlet. The centrifugal action caused by the rotation of the disc causes the solid matter to be driven towards the circumference, from which it can be removed by raising the top half of the chamber. There are nine claims.—E. S.

*Improvements in the Construction of Apparatus used for Expelling Liquid from, and Compressing Semi-fluid or Plastic Substances.* J. Price, London. Eng. Pat. 6861, April 24, 1889. 8d.

THE improvements relate to the construction of moulds employed in or in combination with presses for expelling liquids, or expressing moisture from solid or insoluble substances, when held in a fluid or plastic condition, where it is required to separate the liquid from the solid portion. The invention is intended to remove the difficulty experienced in removing the pressed material from the mould without affecting its shape or sharpness of outline. The head and bed-plate of the press are provided with loose plates, which form the top and bottom of the mould, and which may have any design on or attached to them. The sides of the mould are capable of a certain amount of vertical motion. The material having been compressed, the plunger is secured, and the sides of the mould are then slid upwards or downwards clear of the compressed material and the loose plates. These plates are then securely locked in position, and, the plunger being withdrawn, the plates and pressed material can be removed in one piece and placed aside until dry, when the plates may be removed.

Preferably, the mould is combined with an arrangement of perforated plates and filtering media described in Eng. Pat. 8491, 1888 (this Journal, 1889, 563).—E. S.

*Improvements in Vacuum Distilling and Evaporating Apparatus.* J. Foster, Sunderland. Eng. Pat. 7343, May 2, 1889. 1s. 1d.

THIS invention embraces the construction and arrangement of parts of apparatus for the distillation and evaporation in vacuo of sugar or other liquids, the process being carried out in two, three, or more vessels, on the principle of the "triple-effect" apparatus. The object is to secure greater convenience in the arrangement of the various parts, and to obtain increased heating surface with a higher degree of efficiency of evaporation or distillation than has hitherto been achieved. The evaporation vessels are of the tubular kind, disposed either vertically or horizontally, the tubes being fitted in cylindrical vessels with suitably constructed end chambers. The liquid is, by preference, passed through the tubes, the separation of the vapour taking place in the end chamber, whence the vapour is led around the tubes of the next vessel, whilst the liquid proceeds to pass through them. The specification fully describes and illustrates the various details. There are nine claims, the first referring to the various modes of combination; the third claim for the duplex and return end chambers; the sixth for the distribution of the liquid through the apparatus, and for its retention in any vessel without interfering with the rest; and the seventh claim for the introduction of *venæ contractæ* at the tube entrances, for the regulation of the speed of the passing liquid, seem to be the most important.—B.

*Improvements in the Method of and Means for Treating or Washing Crushed Ores, Coal, and similar Materials.* O. Bilharz, Freiberg, Germany. Eng. Pat. 7500, May 4, 1889. 1s. 1d.

See under X., page 626.

*An Improved Separating or Assorting Machine for Granular or Pulverulent Materials or Substances.* P. Janssen, Hamburg. Eng. Pat. 8119, May 15, 1889. 6d.

THE assorting machine consists of a closed sieve frame, in which one or more sieves, having a zigzag formation, are arranged, one below the other. The frame is suspended on four rods, so that it may move freely, motion being imparted to it by disc cranks moved by bevel wheels. The material to be sorted is brought through funnels. As the finer material passes through the sieves, the coarser parts are successively urged to the outlets by the motion given to the frame and by the particular zigzag formation of the sieves.

The arrangement of zigzag surface of the sieves has the advantage of allowing the direction of motion of the sieves or sieve frame to be varied as necessity requires, the matter being always urged forwards from the inlet to the outlet.

—E. S.

*Improvements in Evaporating or Condensing Milk and other Liquids.* W. P. Branson and H. W. Neild, London. Eng. Pat. 8882, May 28, 1889. 6d.

THE improvement consists in admitting a small quantity of air into the vacuum pan, below the liquid which is being evaporated or condensed, for the purpose of preventing an undue accumulation of the froth. The air pipe, at its outer (and upper) end, is fitted with a tap, by which the passage of air can be controlled. An inlet pipe of about half an inch in diameter is said to be sufficient for a vacuum pan 6 ft. in diameter.—E. S.

*Improvements in Filtering and in Filter Presses.* F. N. Mackay, London. From J. Baxter and J. Muter, Rangoon, Burmah. Eng. Pat. 8928, May 29, 1889. 6d.

THE object is to control the temperature of liquids during filtration in filter presses. The invention consists: "(1.) In causing a heating or refrigerating fluid to circulate on one side of filter-press plates or trays whilst filtration is being conducted on the other side of the said plates or trays." "(2.) "In making the trays with ways or passages there-through, and in connecting such ways or passages to appliances of any usual or suitable construction for causing heating or cooling liquids to circulate." "(3.) "In constructing filter presses in such a manner that the material to be filtered, and the heating or refrigerating fluid, may be contained between alternate plates or trays."—E. S.

*Improvements in Apparatus for Evaporating Liquids.* D. B. Morison, Hartlepool. Eng. Pat. 9041, May 31, 1889. 11d.

THESE improvements refer to the details of connexions used in apparatus in which water or other liquid is evaporated in closed vessels by means of coils of steam pipes, the rising vapour being conducted away for any desired purpose. The principal object is to facilitate the cleaning of the coils. There are three sheets of drawings and four claims.—B.

*Improvements in the Treatment of Sea Water for Marine Boilers.* W. Bramley, Middlesbrough. Eng. Pat. 9246, June 4, 1889. 4d.

As the scale formed in marine boilers supplied with sea water consists of calcium sulphate, the present invention seeks to remove the lime from sea water by precipitating with a mixture of sesquicarbonates of sodium and potassium, and leaving the magnesia in solution.—G. H. B.

*Improvements in Vacuum Drying Apparatus.* J. Brown and G. Johnston, Glasgow. Eng. Pat. 9850, June 15, 1889. 8d.

THE apparatus consists of an upright iron cylinder with flat ends, which are strengthened for resisting a partial vacuum maintained within, for facilitating the drying of the substances under treatment, such as granular, fibrous or powdery materials. The cylinder contains a number of superimposed circular hollow trays, heated by steam, which circulates through pipes from one tray to the other. An upright shaft passing through the centre openings of the annuli puts in rotation a number of arms with stirrers which sweep the upper surfaces of the trays and deliver the materials from one to the other through suitably placed openings. In order to facilitate the feeding in of the material at the top, and the withdrawal of the dried material below, without affecting the vacuum, a special hopper with valve has been designed, of which the accompanying illustrations give an idea. The figures represent a longitudinal and two cross sections.

Fig. 1.

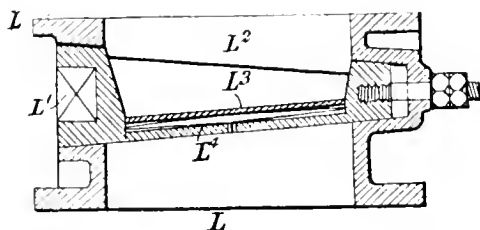


Fig. 2.

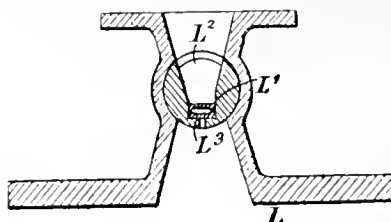
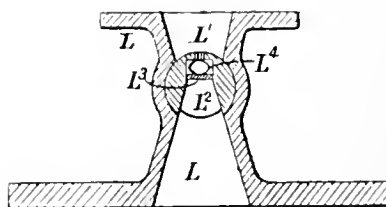


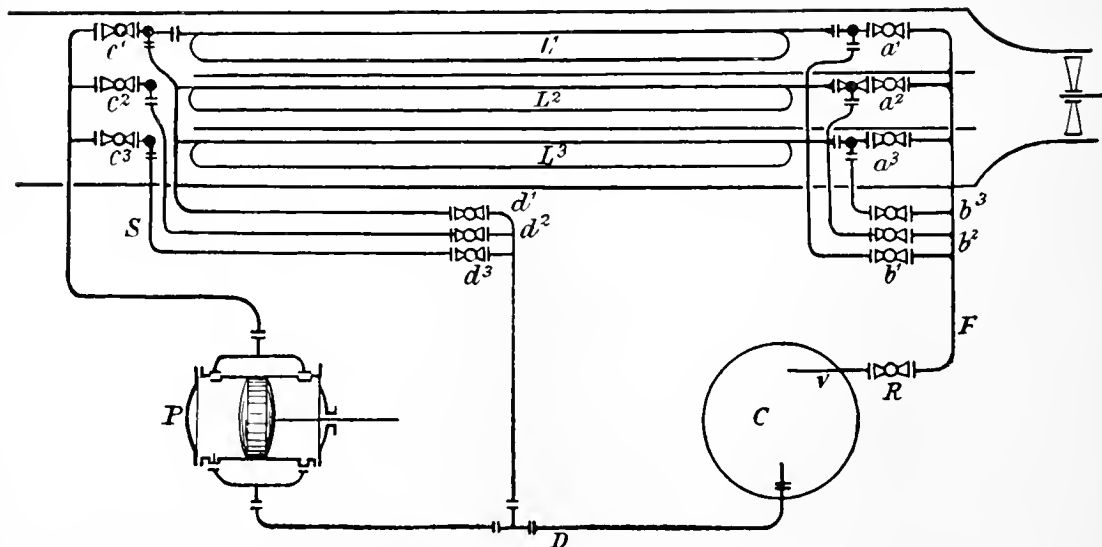
Fig. 3.



The valve consists of the conical plug  $L^1$ , bedded across the hopper casting  $L$ , similarly to the plug of an ordinary cock. The plug is, however, not pierced through, but is only provided on one side with a trough-like recess  $L^2$  which receives the material when turned to the top, and drops it when turned down, at the same time preserving a close joint against the vacuum. For the purpose of disengaging, from the sides of the recess, moist materials that would otherwise adhere, the loose plate  $L^3$  is introduced, the weight of which starts the movement. It may be further assisted by a spring or by a hollow tube  $L^4$  of thin flexible material and suitable shape, which being filled with air and hermetically sealed, expands when turned towards the vacuum in the cylinder, and drives the plate before it. There are two sheets of drawings and three claims.—B.

*Method of and Apparatus for Effecting the Thawing of the Refrigerating Pipes of Air Refrigerators.* C. Linde, Wiesbaden, Germany. Eng. Pat. 10,918, July 6, 1889. 6d.

THE proposed method applies to that class of refrigerating apparatus (represented by the accompanying diagram) in



which the air comes in contact with pipes cooled by the volatilisation of a liquid such as ammonia or carbonic acid contained within them. P represents the compressing pump,

C the condenser,  $L^1$ ,  $L^2$ , and  $L^3$ , are air refrigerators, simultaneously acting as vaporisers of the volatile liquid. The compressed vapours pass through pipe D into C, thence

the resulting liquid through V and valve R into the refrigerators through the cocks  $a^1$ ,  $a^2$ , and  $a^3$ , whilst cocks  $c^1$ ,  $c^2$ ,  $c^3$  are closed. After evaporation the position of the cocks is reversed and the vapours drawn back through pipe S to the pump. If now coil  $L^2$ , for instance, should require to be thawed, the cocks  $a^2$  and  $c^2$  are closed, and the auxiliary cocks and passages  $b^2$  and  $d^2$  opened, connecting the system  $L^2$  with the compression pipes from the pump and with the liquid pipe F. Through the latter the liquid condensed within  $L^2$  during the thawing process, will return. Upon resetting the cocks as at starting, refrigeration again commences. There are two claims.—B.

or (2), and preferably, as—



These formulæ note the fact that the hydrogen has been taken up (in all probability) as water.

The authors prefer the second series of formulæ as extending Brodie's suggestion that the graphites are distinct radicles.

The paper is followed by a thermo-chemical investigation of the compounds described (Compt. Rend. 110, 106—109).—P. J. H.

*Manufacture of Ammonium Chloride from By-Products in Gas Manufacture and Metallic Chlorides.* Dubosc and Heuzey. Bull. Soc. Ind. Rouen, 1889, 438—442.

See under VII., page 614.

## II.—FUEL, GAS, AND LIGHT.

*On the Different Forms of Graphite and their Corresponding Chemical Derivatives.* Berthelot and Petit. Compt. Rend. 110, 101—106.

BERTHELOT has previously put forward the view that the allotropic forms of carbon are really different polymers of the element (Ann. Chim. Phys. [4], 9, 475, and [4], 19, 396). They give rise by the processes of oxidation and hydrogenation to compounds of corresponding complexity. Thus the oxidation of the graphites in the wet way at a low temperature yields certain ternary compounds, the first of which was discovered by Brodie (graphitic acid). Berthelot has since shown that there are several graphites of distinct chemical nature, each one giving rise to (1) a "graphitic oxide;" (2) a "hydrographitic oxide;" (3) a "pyrographitic oxide;" and from these the original form of graphite can again be obtained (Ann. Chim. Phys. [4], 19, 405).

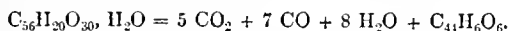
The authors employ the methods described in the memoir referred to, to prepare the graphitic and pyrographitic "oxides" of various species of graphite.

1. *Graphite from Cast Iron.*—The "oxide" corresponds to an empirical formula  $C_7H_2O_3$ , or its multiple  $C_{28}H_8O_{12}$ , which is used not as a rational formula, but for the sake of comparison with other compounds. The oxide is neutralised by 54 per cent. of BaO, hence its least formula is  $C_7H_2O_3$ . It deaggregates suddenly when heated to a certain temperature.

Heated in a sealed tube, the following reaction takes place— $2 C_{28}H_8O_{12} = 4 CO_2 + 6 CO + 5 H_2O + C_{48}H_6O_5$ .

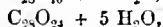
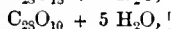
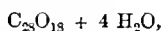
The formula of the pyrographitic oxide,  $C_{48}H_6O_5$ , thus obtained was determined by analysis and by neutralising with baryta (being saturated by 10 per cent. of BaO).

2. *Amorphous Graphite or Plumbago.*—The oxide corresponds either to a formula  $C_{36}H_{20}O_{30} + H_2O$ , or to  $C_{22}H_8O_{12}$ . On heating in a sealed tube the corresponding pyrographitic oxide was obtained as before—



3. *Electric Graphite.*—This substance, formed by the transformation of the carbons in the electric arc, could only be obtained in small quantity, and was not free from ordinary carbon. It yielded a "graphitic oxide" corresponding to a formula  $C_{28}H_{10}O_{19}$ , or else  $C_{16}H_6O_{11}$ . The authors had too little substance to investigate its decomposition by heat.

They point out the great difference of composition in the three graphitic oxides (C = 62.7 per cent., C = 56.2 per cent., C = 51.95 per cent. for cast-iron graphite, plumbago, and electric graphite respectively), and suggest that they may be regarded, either (1) as—



## PATENTS.

*A Method of and Apparatus for altering the Proportions of Mixed Gases.* S. Fox and E. Blass, Leeds. Eng. Pat. 6129, April 10, 1889. 6d.

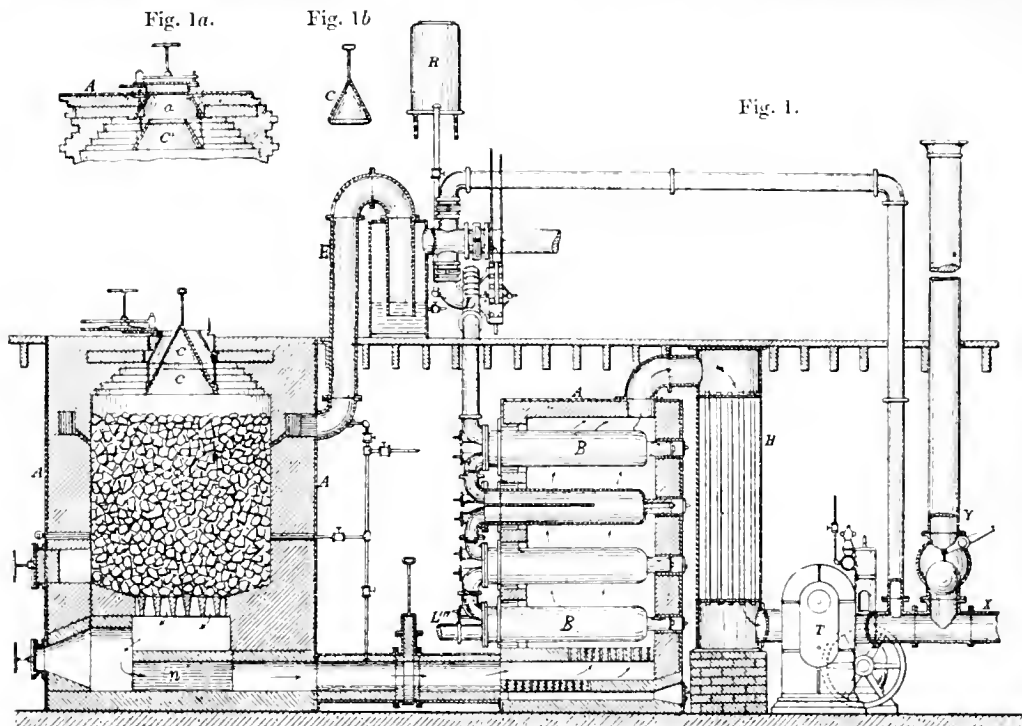
THE invention relates to a method of altering the proportions of the gases in a gaseous mixture by eliminating or removing from such a mixture definite quantities of the respective gases by compressing the mixture in presence of a suitable absorbent, such as water; the compressed gaseous mixture or absorbent is utilised, under pressure, to aid in driving the compressing pump, the rest of the power required for driving the pump being provided by any suitable motor.—F. S. K.

*An Improved Process of and Apparatus for Manufacturing Heating and Illuminating Gas.* B. Loomis, Hartford, Connecticut, U.S.A. Eng. Pat. 9279, June 4, 1889. 11d.

By this process a high-grade heating gas can be cheaply produced, and this gas may be carburetted or enriched with hydrocarbon oil or vapour, which is "fixed" by finally passing through heated retorts or brickwork flues.

The producer is, as usual, of firebrick (iron cased), but a special feature is that in making the producer-gas the air supply is drawn in at the top, the gas being drawn away at the bottom by means of an exhaustor. Many advantages are claimed for his. Dust and ashes are blown down, and the formation of clinker prevented, while the oily and tarry vapours from the top layers of fresh coal are drawn down into the heated coal below, and converted into valuable carburetted hydrogen gas. Fresh fuel can be more rapidly and uniformly charged, as the charger can work on the top and inspect the interior of the furnace. Waste heat from the gas is used to superheat the steam for the water-gas, while the producer-gas is led through, and, if desired, burned with air in the retort chamber, and then, after passing through a cooler, is drawn through the exhaustor. The water-gas is taken off some little way down from the top, which prevents the melting and caking of the upper fresh layers of coal. This water-gas is mixed with the oil or vapour by means of a spray and a rotating arrangement, and then led through the heated retorts or flues to be fixed, which heating, in this case, being gradual, ensures the gradual expansion and gasification of the oil vapour. This will be clearly explained by the illustration.

A is the producer and c the cone-shaped top (shown also in detail), F' is the main for producer-gas, n being brick-work to absorb heat for subsequent superheating of the steam. A' is the retort chamber, where the gas may be burnt with air, H the cooler, T the exhaustor, Y a ball valve leading to the chimney, which is closed if producer-gas is wanted at X for boilers or otherwise, E is the outlet for the water-gas, R the oil reservoir, and L the rotary mixer.



B B the retorts where the gas is finally heated, and L''' the outlet for the finished illuminating gas. There are four sheets of drawings and 19 claims are made.—D. A. S.

*Improved Apparatus for Generating and Consuming Combustible Gas.* G. Love, Quebec. Eng. Pat. 9454, June 7, 1889. 6d.

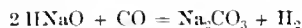
Two sets of hollow perforated bars or pipes are arranged in a suitable furnace in such a way that air can be forced through the one series till the fuel becomes incandescent, and then steam or vapour can be forced through the other series for the production of water-gas. An air-chamber is provided at the outlet of this generating furnace to supply air for the complete combustion of the gas so formed. The application of this furnace to a single-flued boiler is illustrated in the specification.—D. A. S.

*An Improvement in Regenerative Gas Furnaces.* R. S. Casson, Brierley Hill. Eng. Pat. 10,050, June 19, 1889. 6d.

See under X., page 627.

*Improvements in the Treatment of Water-Gas.* W. Crookes and F. J. Ricardo Seaver, London. Eng. Pat. 10,164, June 21, 1889. 4d.

THE authors propose rendering water-gas non-poisonous by removing carbonic oxide which as is well known it contains, in about equal proportions with hydrogen. The water-gas is passed over soda lime (preferably through several feet in a retort) at a good red heat; sodium carbonate is formed and hydrogen liberated—



which hydrogen takes the place of the carbonic oxide, and on account of its greater diffusiveness could probably be carburetted or enriched easier than water-gas if desired for illuminating purposes. One thousand cubic feet of mixed gas would require about 320 lb. of soda lime. The sodium

carbonate could be recovered by lixiviation and causticised, and the lime could be reburnt, thus enabling the same materials to be used over again.—D. A. S.

*Improvements relating to the Burning of Liquid Fuel in Boiler Furnaces and the like.* H. J. Haddan, London. From F. F. Amoroso, Venice, Italy. Eng. Pat. 10,392, June 26, 1889. 6d.

A CYLINDER fitted with radiating holders tipped with wick or asbestos is caused to rotate. The holders dip in turn into an open tank or tray containing oil which is kept to a constant level by means of an automatic arrangement. These holders, rising continuously into the fire, burn fiercely. It is claimed that this arrangement is noiseless, and secures the best distribution of the flame. Illustrations are given showing its application to several forms of boilers.—D. A. S.

*A New or Improved Producer for making Ordinary Producer-Gas and Water-Gas and Coke.* J. C. Johnson, Wednesbury. Eng. Pat. 10,581, June 29, 1889. 8d.

THIS producer consists of a firebrick chamber preferably wider at the top than at the bottom. At the base it is sealed by terminating in a trough of water down through which the coke can be drawn by means of a rake. There are two openings for the blast below the middle of the chamber, and there are two outlets for gas at the top. These are so arranged that the blast can be worked alternately from either side, so as to induce a cross current through the mass of fuel. If coke or water-gas be required, the coke is worked down by means of a rake into the water and drawn out, while the steam formed in quenching the hot coke passes up through the incandescent fuel, producing water-gas.

—D. A. S.

### III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

*Acids of Low Carbon Percentage occurring in Petroleum from Baku.* O. Aschan. Ber. 23, 867—875.

ABOUT two years ago the author procured for the purpose of this investigation, from Estlander of Baku, about 200 kilos. of crude naphtha acids which had been separated from the residue obtained in the purification of petroleum with caustic soda by adding water to the semi-fluid mass and heating to 90°—100° for 48 hours. The clear solution was then treated with sulphuric acid, and the acid thus obtained separated by decantation. The crude acid has a light yellow colour and petroleum-like odour, and shows a specific gravity of 0.9891 at 18°. When subjected to distillation at the ordinary pressure water and hydrocarbons begin to come over; at 200° the acid commences to boil with partial decomposition. The greater portion boils above 300°, only one-third distilling over below this temperature.

Hell and Meidinger some time ago isolated from crude Wallachian petroleum an acid having the formula  $C_{11}H_{13}O_2$ . Markownikow and Oglobin obtained two acids, to which they assigned the formula  $C_{10}H_{15}O_2$  and  $C_{11}H_{15}O_2$ , whilst Krämer has recently isolated an acid of the formula  $C_{15}H_{25}O_2$ . The extensive researches made by Markownikow appear to show that the acids belonging to the group represented by the general formula  $C_nH_{2n-2}O_2$  are the carboxylic acids of hydrocarbons of the  $C_nH_{2n}$  series, called naphthenes, which constitute the principal part of Caucasian petroleum. The same author has recently shown that the naphthenes are hexahydrides of benzene hydrocarbons, so that the corresponding acids must be regarded as hexahydroxybenzoic acid and its homologues. Of these acids those represented by simple formulae are of the greatest interest, as the higher homologues, which contain several isomers, cannot be isolated without considerable difficulty. The author has therefore separated and examined the lowest boiling acids. For this purpose the crude naphtha acid was subjected to distillation and the distillate up to 270° collected. This was then treated with a 5 per cent solution of caustic soda, and after standing for two days the petroleum layer was drawn off and the aqueous solution decomposed with sulphuric acid. A colourless oil was obtained which assumed a dark colour when exposed to light. 100 kilos. of crude acid gave 6 kilos. of low boiling products. In order to isolate the various acids the mixture was methylated and subjected to a long series of fractional distillations. In this manner the methyl ether of hexanaphthenecarboxylic acid was obtained. It boils between 165.5° and 167.5°; forms a colourless oil of 0.90547 sp. gr., having an agreeably fruity odour, which, however, eventually excites nausea. The free acid was prepared by dissolving the ether in alcohol, boiling the solution with a slight excess of potassium hydroxide, and decomposing the potassium salt thus formed with an acid. A yellow oil was obtained which after refining boiled at 215°—217°, and gave by analysis numbers agreeing with the formula  $C_7H_{12}O_2$ . Hexanaphthenecarboxylic acid is a colourless oil possessing a strong odour of valerian. It has a specific gravity of 0.95025 at 18.4°. Bromine has no action at the ordinary temperature, but on heating substitution takes place. Potassium permanganate in alkaline solution is gradually decolourised. Nitric acid causes slow oxidation. Hexanaphthenecarboxylic acid has strongly acid properties. It is isomeric with the synthetical methyl-pentanethylenecarboxylic acid (2:1) recently prepared by Perkin, jun., and Colman. The following salts have been prepared:—The potassium salt is readily soluble in water and alcohol, and is highly hygroscopic. The sodium salt is obtained from alcohol in the form of indistinct flat prisms, which effloresce on heating. The calcium salt is readily soluble in alcohol and water, and crystallises in two forms containing different amounts of water. The barium salt is readily soluble in alcohol, sparingly soluble in water, and forms two modifications. The amorphous salt contains water, and the crystalline salt is anhydrous. The silver salt is sparingly soluble in cold, more readily soluble in hot

water. The cadmium salt forms nacreous laminae sparingly soluble in cold, readily soluble in hot water. The ammonium salt cannot be obtained. On drying, ammonia is liberated and the acid reproduced. The author has also prepared the chloride, amide, and anilide. The chloride boils at 167°—169°. The amide melts at 123.5°, and is readily soluble in alcohol and water. The anilide crystallises in long needles which melt at 93°—94°. Of the homologues of hexanaphthenecarboxylic acid two acids represented by the formulae  $C_8H_{14}O_2$  and  $C_9H_{16}O_2$  have been prepared in a pure form; they boil at 237°—239° and 251°—253° respectively, and their properties will be described by the author in a future communication.—D. B.

*The Occurrence and Extraction of Ozokerite in Galicia.* J. Shotsky. Allg. österr. Chemiker-und Techniker Zeit. 1889, 614 and 651.

THE most important deposit of ozokerite is near Borislav, where it occurs in a marl containing salt. The wax is found in irregular lumps, sometimes of great size, or in layers which frequently die out completely. The neighbouring rock consists of clay-slate, marl and sandstone.

The centre of the basin is richest in wax; in some cases masses of such extent have been tapped that the miners have hardly had time to escape before the workings were filled with the plastic mineral. Such a deposit was recently found in the deepest shaft at Borislav, at a depth of 208 metres (227 yards). In general, however, the yield of wax varies at from 2 to 8 per cent. of the mineral extracted. Ozokerite was first found in this district in 1854 by Doms, who was in search of petroleum with which much of the ground is saturated. At first it was regarded as an unwelcome companion to the petroleum, as it frequently caused the timbering of the shafts to collapse. It was not until about 20 years later that this substance began to attain commercial importance, a method having then been discovered of producing from it a substance resembling bees wax and named cersin.

In 1865 ozokerite, which had previously been regarded as a Crown mineral, was declared free, and the consequence was that a number of shafts were sunk in the district, and much speculation ensued. The land being parcelled out in small plots, the shafts were sunk in the immediate neighbourhood of each other, and much waste and danger ensued. In 1886 the present law was passed, according to which the right of mining for ozokerite may be separated from the ownership of the land. The extraction is now carried out under official supervision.

In nearly every case the mineral is raised through vertical shafts or pits over which a wooden roof is erected. The section of the shafts in the first instance is 3 to 4 square metres (32 sq. ft. to 43 sq. ft.); but when the ozokerite formation is reached, an inner shaft 1 metre square (10.76 sq. ft.) is formed of timber, and the space between this and the timbering of the larger shaft is filled with a rich clay. This construction is adopted to exclude the surface-water, which is kept down by hand-pumps during sinking. From the bottom of the shafts levels are driven into the ozokerite ground, the richer portions being raised and the refuse used to fill up the old workings. The softer parts of the marl are dislodged by means of pick or wedge; but where the rock is hard, and the permission of the mining authorities can be obtained, dynamite is used. The mineral is raised by hand in skips or tubs holding 40 to 50 kilos. (88 to 110 lb.). Hand-ventilators are used for the purpose of ventilation, but explosions of gas are not uncommon, especially after Sundays and holidays. Fatal accidents are, however, rare. Safety-lamps are used in all the mines. The timbering of the shafts requires constant renewal and repairs; in some cases it is almost impossible to keep the shafts perpendicular.

The water is usually raised in tubs, and much difficulty is experienced in getting rid of it after it reaches the surface, on account of the numerous shafts and the broken nature of the ground. The mineral, when it leaves the tubs, is sorted by hand. The waste rock is picked out and tipped to spoil, lumps of ozokerite are specially selected, and the

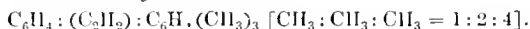
remainder of the rock, containing fragments of wax, is tipped into tanks full of water. On being well stirred most of the wax rises to the surface and is skimmed off. The residue still contains from 2 to 3 per cent. of wax. The quantity of waste mineral being considerable, and the distance between the shafts small, a special railway has been built to remove the residues from the immediate neighbourhood of the mines.

Only one attempt has been made to mine on a large scale for ozokerite. In this case a circular shaft, 2 metres (6 ft. 7 in.) in diameter and lined with iron tubing, was sunk outside the ozokerite zone, and the deposit was reached by galleries. Trucks holding 500 kilos. (1,102 lb.) were used, and both ventilation and pumping were done by steam-power. The operations on a large scale do not, however, seem to have been successful, the French company which carried them out having now ceased working.

The production of ozokerite in the Borislav district amounted in 1887 to 96 per cent. of the total output in Galicia, and was valued at 152,900*l.*

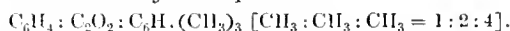
*On Homologues of Anthracene and Anthraquinone.* K. Elbs. J. prakt. Chem. **41**, 121—151. Conclusion. (See this Journal, 1890, 276—278.)

#### 1 : 2 : 4 Trimethylantracene—



In white fluorescent plates, melting at 244°, described by Gresly (J. Chem. Soc. 1886, 1028) by distillation of pseudocumene-phthalic acid with zinc dust. When oxidised by chromic acid in glacial acetic acid it yields 1 : 2 : 4 trimethylantraquinone, melting at 162°. An unstable *picric acid derivative* was obtained in red-brown needles melting at 145°.

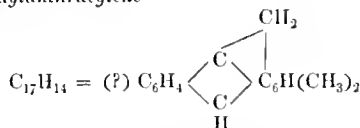
#### 1 : 2 : 4 Trimethylantraquinone—



In long yellow needles, melting at 162°—163°, and subliming without residue; soluble with difficulty in alcohol, more readily in ether, benzene, and xylene. A careful detailed description is given of its preparation from pseudocumene-phthalic acid. Warmed with zinc dust and alkali it becomes a blood-red liquid; with sodium amalgam and absolute alcohol a garnet red solution.

By reduction of 1 : 2 : 4 trimethylantraquinone with zinc dust and ammonia the author obtained a hydrocarbon in colourless plates, melting at 64°, becoming yellow and lowered in melting point, on exposure to air. (Compare this Journal, 1890, 277.) This he considers to be—

#### Trimethylantracylene—



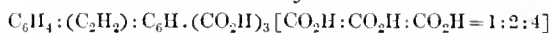
A *picric acid derivative* was prepared in dark-red needles melting at 134°, decomposing on exposure to air and light. The bromination of *Trimethylantracylene* gave a *dibromo derivative*,  $\text{C}_{17}\text{H}_{12}\text{Br}_2$ , crystallising from carbon disulphide in somewhat large yellow transparent prisms, with difficulty soluble in alcohol, and melting at 105°.

#### 1 : 2 : 4 Anthraquinone tricarboxylic acid—



In yellowish white nodules, almost insoluble in water, but easily soluble in alcohol; it neither melts nor decomposes at 320°. By oxidation of 1 : 2 : 4 trimethylantraquinone with dilute nitric acid it formed a red coloured solution with ammonia, but the salt was not obtained pure. Its *monosodium salt* (yellow, with 2 mols.  $\text{H}_2\text{O}$ ); its *disodium salt* (yellowish-red, with 3 mols.  $\text{H}_2\text{O}$ ); its *normal sodium* (blood-red), *calcium* (rose-red), *copper* (green), *lead* (yellow), and *silver* (red) salts are described. The *ethyl salt* occurs in small yellow lustrous plates, melting at 125°.

#### 1 : 2 : 4 Anthracene tricarboxylic acid—



A deep yellow precipitate, insoluble in water but soluble in alcohol, petroleum spirit, and benzene. It has no definite melting point, but at over 260° it sublimes, giving a red sublimate, which is probably the anhydride.

Prepared by reduction of the preceding acid with zinc dust and ammonia. The solutions of the *alkali salts* are greenish-yellow with blue fluorescence. The *silver salt* is greenish-yellow. By nitric acid 1 : 2 : 4 trimethylantraquinone three nitro-compounds are formed, two isomeric mono-compounds and one dinitro-compound.

The author terms the two former I. and II. respectively. 1 : 2 : 4 *Mononitro-trimethylantraquinone I.* is a red powder insoluble in petroleum spirit, alcohol, and ether, but soluble with difficulty in acetic acid, chloroform, carbon disulphide, and acetone, but easily soluble in boiling xylene and benzene, melting with decomposition at 195°—200°, and subliming in small yellow needles.

The 1 : 2 : 4 *nitro-antraquinone tricarboxylic acid* forms in yellow crystals insoluble in water, but soluble in alcohol, ether, &c. It melts with decomposition at 308°—310°. The three *sodium salts* (clear yellow, purple-red, and blood-red) are described, and also the normal *calcium*, *barium*, *strontium*, *nickel*, *cobalt*, *lead*, *copper* and *silver salts*. The *amido acid* was obtained in small deep-red plates, melting at 210°, soluble in boiling water and in alcohol.

*Mononitroanthraquinone II.* crystallises badly and could not be obtained pure. It is a pale red precipitate decomposing at 180°, insoluble in alcohol and ether but soluble in glacial acetic acid, benzene and xylene. The corresponding *tricarboxylic acid* formed yellowish-red crystals soluble with difficulty in water, easily in alcohol, ether, and benzene, and melting with decomposition between 360° and 370°. Its three *sodium salts* (all red) its neutral *calcium*, *barium*, *strontium*, *nickel*, *lead*, and *copper* (red needles), and *silver salts* are described. The *copper salt* dissolves in ammonia with a red colour, while the *copper salt* of the I. acid is green and dissolves in ammonia to an emerald-green colour. The *amidotricarboxylic acid* occurs in deep red plates melting at 255°, soluble in water and alcohol and in caustic potash to a red, and in hydrochloric acid to a yellow colour. The *amidoquinone* in deep red crystals melted at 154°—155°, and sublimed in lustrous dark red needles melting at 156°, soluble in alcohol and benzene.

1 : 2 : 4 *Dinitro-trimethylantraquinone* in deep yellow crystals, melting at 340°—360° with decomposition, distinctly soluble in water and easily in alcohol, &c. The *Tricarboxylic acid* was prepared and its *silver salt* (purple-red).

#### 2 : 1' : 4' Trimethylantracene—

$\text{C}_6\text{H}_5 : (\text{CH}_3)_2 : \text{C}_2\text{H}_2 : \text{C}_6\text{H}_3\text{CH}_3 [\text{CH}_3 : \text{CH}_3 : \text{CH}_3 = 2 : 1' : 4']$   
In colourless plates with green fluorescence subliming below 100°, and melting with decomposition at 227°, soluble in boiling alcohol ether, &c. (compare J. Chem. Soc. 1887, 941).

#### 2 : 1' : 4' Trimethylantraquinone—

$\text{C}_6\text{H}_5 : (\text{CH}_3)_2 : \text{C}_2\text{O}_2 : \text{C}_6\text{H}_3\text{CH}_3 [\text{CH}_3 : \text{CH}_3 : \text{CH}_3 = 2 : 1' : 4']$   
In yellow needles melting at 184° and subliming; sparingly soluble in alcohol but easily in glacial acetic acid. On oxidation a mixture of the *di-* and *tricarboxylic acids* resulted. The *silver salt* of the former is described.

#### 3 : 1' : 3' Trimethylantracene—

$\text{C}_6\text{H}_5 : (\text{CH}_3)_2 : (\text{C}_2\text{H}_5) : \text{C}_6\text{H}_3\text{CH}_3 [\text{CH}_3 : \text{CH}_3 : \text{CH}_3 = 3 : 1' : 3']$   
Melting at 222°, slightly soluble in alcohol, but easily in carbon disulphide; made by condensation of *di-m-xylol-ketone*. (Cosaek, Diss. Freiburg, B. 1889.)

On bromination *γ-dibromo 3 : 1' : 3' trimethylantracene* is produced in yellow plates melting at 142°.

#### 3 : 1' : 3' Trimethylantraquinone—

$\text{C}_6\text{H}_5 : (\text{CH}_3)_2 : \text{C}_2\text{O}_2 : \text{C}_6\text{H}_3\text{CH}_3 [\text{CH}_3 : \text{CH}_3 : \text{CH}_3 = 3 : 1' : 3']$   
In yellow needles melting and subliming at 190°, with difficulty soluble in alcohol, easily in glacial acetic acid. By oxidation of the preceding compound with chromic acid, its *Tricarboxylic acid* was prepared in yellow plates melting at over 300°, almost insoluble in water and only sparingly in the

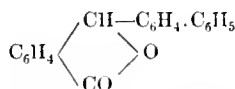


usual solvents. The *ammonium salt* (flesh coloured) and *barium salt* (cherry red) are described. Naphthanthracene,  $C_{18}H_{14} : (C_2H_2) : C_{10}H_6$ , by reduction of naphthanthraquinone, which in turn is the product by abstraction of water from naphthoyl-*o*-benzoic acid (see Ber. 19, 2209).

The author failed to obtain phenylanthraquinone by condensation of *phenylbenzoyl-o-benzoic acid*.

The last mentioned acid  $CO_2H.C_6H_4.CO.C_6H_4.C_6H_5$  was obtained in small yellowish-white needles melting at  $225^\circ$ , soluble with difficulty in petroleum spirit, but easily in alcohol, ether, &c. The *sulphonic acid* derivative and its *barium salt* were obtained. It was prepared by warming a mixture of diphenyl-phthalic-anhydride and aluminium chloride in petroleum spirit boiling at  $90^\circ$ – $100^\circ$ , which also is the best temperature for the reaction. After distillation with steam, the residual acid was dissolved in dilute soda solution from which it was precipitated by hydrochloric acid. The *nickel salt* (pale green), the *copper salt* (pale blue), the *lead salt* (brownish yellow), and the *silver salt* (white) were prepared from the *ammonium salt*, and are described.

A *lactone*, the inner anhydride of phenylbenzylhydriyl-ortho-benzoic acid—



is the chief product obtained by reduction of the phenylbenzoylorthobenzoic acid in alcoholic solution with zinc dust and ammonia. It forms colourless crystals melting at  $204^\circ$ , subliming in part when heated higher, the sublimate melting at  $205^\circ$ – $206^\circ$ . The *lactone* is soluble in the usual solvents. Further reduction produces *phenylbenzylorthobenzoic acid*,  $C_6H_5.C_6H_4.CH_2.C_6H_4.CO_2H$ , in colourless or slightly red crystals melting at  $184^\circ$ – $185^\circ$  without decomposition, soluble in alcohol and ether, but almost insoluble in water. Its *silver salt* (white) is described. The author concludes with a summary of the results of the foregoing communication.—D. A. S.

#### PATENT.

*Process for Cleansing and Bleaching Fibrous Substances and for Fixing Organic Colouring Matters thereon by Means of Acid and Alkaline Residues of Naphtha Manufacture, or of specially prepared Substances analogous thereto.* V. Schevelin, Bulakna, and P. Mindovsky, Moscow, Russia. Eng. Pat. 8107, May 15, 1889. *8d.*

See under VI., page 612.

## IV.—COLOURING MATTERS AND DYES.

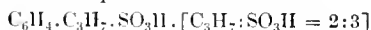
*Normal Cumene Sulphonic Acids.* A. Claus and O. Welzel. J. prakt. Chem. 41, 152–158.

PATERNÒ and Spica (J. Chem. Soc. 1877, 707) described the ortho and para ( $\alpha$  and  $\beta$ ) cumene sulphonic acids. The authors have since prepared these.

There is a marked difference in the solubility of these salts in water, the  $\alpha$ -sulphonates being soluble with difficulty and crystallise readily, while the  $\beta$ -sulphonates are very soluble in water and do not crystallise well. The authors, as the result of their research, find considerable difference between their acids and those of Paternò and Spica, and doubt if the latter obtained the true acids.

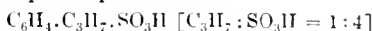
The acids were best prepared from their *nickel salts*; the  $\alpha$ -salt crystallised in light green lustrous needles on cooling the hot solution, while the  $\beta$ -salt did not crystallise even from a concentrated solution, but was obtained on evaporation as a yellowish-green powder.

#### *n-cumene-orthosulphonic Acid*—



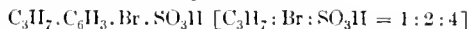
In colourless needles or small columns with one mol. of water. Soluble in alcohol and water. The *nickel salt*, the *chloride* and *amide* are described. *Orthobromocumene* was obtained as a yellow oil boiling at  $222^\circ$  (uncorr.); on oxidation with nitric acid this yielded orthobromobenzoic acid. The authors are still working on the bromosulphonic acids.

#### *n-Cumeneparasulphonic Acid*—



In needle-shaped crystals. Both acid and salts are more soluble in water and alcohol than the foregoing. The *amide* and the *p-bromo derivative* (boiling at  $220^\circ$  uncorr. and solidifying on cooling) were obtained. The latter was oxidised to *p-bromobenzoic acid*, melting at  $251^\circ$  (uncorr.)

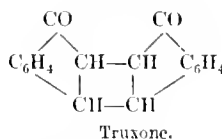
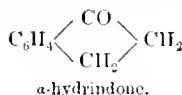
#### *o-bromocumeneparasulphonic Acid*—



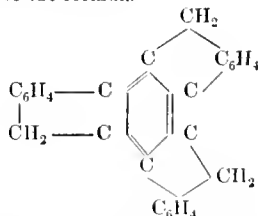
was prepared, and gave on treatment with superheated steam bromocumene, which in turn on treatment with dilute nitric acid gave pure *o-bromobenzoic acid* melting at  $147^\circ$  (uncorr.).—D. A. S.

*On Truxene and Truxene Derivatives.* C. Liebermann and O. Bergami. Ber. 23, 317–322.

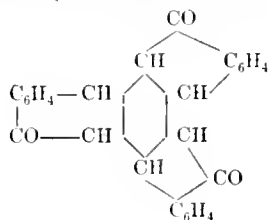
TRUXONE, which had been obtained by one of the authors (Ber. 22, 785) by treating  $\alpha$ -truxilic acid with concentrated sulphuric acid, yields on reduction the hydrocarbon ( $C_9H_6$ )<sup>2</sup>, which has a very high melting point, viz.,  $360^\circ$  C. Hausmann (Ber. 22, 2023), starting from a different product,  $\alpha$ -hydrindone, has also arrived at a hydrocarbon ( $C_9H_6$ )<sup>2</sup> (by means of dehydration with hydrochloric acid), which seems to be identical with truxene, which, however, had not yet been proved by Hausmann.



According to the formula—



which Hausmann gives to truxene, it ought to yield on oxidation the same tribenzoylenebenzene obtained by Gabriel and Michael (Ber. 21, 1007). This was confirmed. The body obtained showed only a slight difference on analysis, evidently due to traces of the hydrocarbon, from which it could not be freed entirely. The authors therefore feel inclined to assign a tri-molecular formula to truxone—

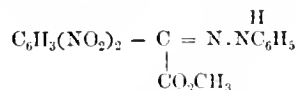


They have, however, not yet succeeded in producing tribenzoylbenzene by oxidation from above compound, some small quantities only being formed by melting with caustic potash. They could not investigate the subject any farther in this direction, on account of scarcity of material.

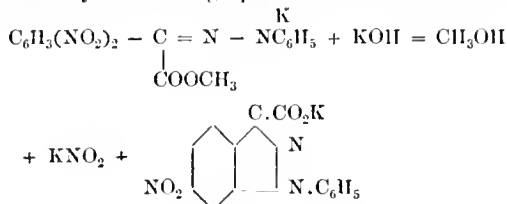
Truxone-oxime-anhydride and acetyl-truxone-oxime were also prepared by treatment with hydroxylamine. The potash melt yielded the body,  $C_{28}H_{16}O_3$ , dihydrodiphenylencoxy-anthraquinone, having a great similarity with the colouring matters of the anthraquinone group.—O. J. S.

*Notes on Indazole Derivatives.* H. Strassmann. Ber. **23**, 714—718.

VICTOR MEYER obtained by the action of diazobenzene-chloride on dinitrophenylacetic ether a compound—



which yielded with caustic potash a deep blue potash salt. This body is, however, unstable and undergoes change on standing a few minutes, whilst on boiling it changes immediately into a yellow salt, losing methyl alcohol, potassium nitrite being meanwhile formed. V. Meyer explains these reactions by the following equation—



and calls the new acid Nitrophenylindazole carbonic acid. This body, though a nitro-compound, *cannot be reduced*. Stannous chloride, tin, and hydrochloric acid, glacial acetic acid, and zinc dust are without any action upon it, even on continued ebullition. Ethyl alcohol acts on the methyl ether of this acid (m.p.  $192^\circ$ ) in presence of hydroxylamine hydrochloride after heating for eight hours at  $160^\circ$ — $180^\circ$  C., producing yellow needles melting at  $159^\circ$  C. They are the ethyl ether of the same acid. The same result is produced, if instead of hydroxylamine hydrochloride free hydrochloric acid be used.

The indazole compound dissolves in fuming nitric acid forming a mononitro-compound of the methyl ether  $C_{15}H_{10}N_2O_4(NO_2)$  melting at  $281^\circ$  C., which can be easily reduced to an amido-compound. Fuming sulphuric acid forms with the indazole a sulphonic acid. Some further experiments are described which, however, were not successful in explaining the constitution of these complicated compounds.—A. L.

*On Oreinol- and Resoreinol Colours.* R. Nietzki and H. Maackler. Ber. **23**, 718—726.

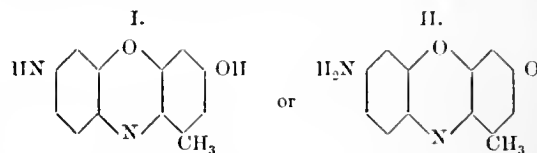
WESELSKY and Benedikt have treated the ethyl ethers of resoreinol with nitric acid containing nitrons acid in the same manner as described for the preparation of resazurinol (this Journal, 1890, 489). They obtained two substances, of which one separates from the ethereal solution, whilst the second is obtained after shaking the ether solution with caustic alkali and evaporating. The author observed on repeating these experiments that the diethyl ether does not yield any of the latter substance, whilst it can be readily prepared from the mono-ethylether. The formula is  $C_{14}H_{11}NO_3$ , and it is resorufinol-mono-ethyl ether,  $C_{12}H_6NO_3C_2H_5$ , melting at  $225^\circ$  C. The first body, described by Weselsky and Benedikt, the authors were unable to prepare. It is probably resazurinol ethyl ether.

Oreirufinol was obtained from oreinol by treating it like resoreinol in ethereal solution with fuming nitric acid. It forms dark brown needles soluble with a red colour in alkalis. It is a dimethylresorufinol  $C_{12}H_3(CH_3)_2NO_3$ . Bromine

changes oreirufinol into a blue colouring matter exhibiting strong fluorescence, very similar to Resorufinol blue. Acetic anhydride forms on heating with oreirufinol a monacetyl derivative, crystallising in orange-yellow needles melting at  $204^\circ$  C.

Liebermann described a colouring matter which he obtained by the action of sulphuric acid containing nitrons acid, on oreinol. This substance was identified by the authors as Oreirufinol. Oreirufinol may be further prepared by heating nitroso-oreinol with oreinol in sulphuric acid solution. Nitroso-oreinol can be readily prepared according to Fèvre's method; it forms yellow needles, exploding at  $157^\circ$  C.

*Action of Quinonedichloro-imide on Resoreinol and Oreinol.*—If Quinonedichloro-imide acts on resoreinol in concentrated sulphuric acid solution no formation of colouring matter takes place. Reaction sets in, however, when an alcoholic solution is taken. The yield of colouring matter is very small, and purification is extremely difficult on account of the abundance of by-products. The authors discovered that the action of quinonedichloro-imide on oreinol is far more successful. One molecule of quinonedichloro-imide dissolved in 20 parts of alcohol is gradually added to a molecular weight of oreinol in alcoholic solution. The mixture changes to a beautiful red with yellowish fluorescence. After heating for several hours on the water-bath, water and an excess of ammonia are added. A base is thus precipitated which is purified by means of its sulphuric acid salt which is with difficulty soluble. The base crystallises in small brown needles and has the formula  $C_{13}H_{10}N_2O_2$ . It is insoluble in water but soluble in alcohol with a red-violet colour. Its constitution is either—



The latter formula (II.) is probably the correct one. The body, which is of decided basic character, and insoluble in caustic soda, forms only a monacetyl derivative and a diazo-compound, which combines with naphtholsulphonic acid, yielding colouring matters. All these properties can only be explained by formula No. II. The authors propose to name this body *Oreirufamine*.

The compound prepared from resoreinol—*Resorufamine*—resembles very much the already described compound. Its preparation is, however, far more difficult.—A. L.

*On Amidochrysene.* R. Abegg. Ber. **23**, 792—793.

MONONITROCHRYSENE, prepared according to Schmidt's method, melting at  $205^\circ$  C., forms no amido-compound when treated with tin and hydrochloric acid or zinc and acetic or hydrochloric acid. On heating it with stannous chloride and hydrochloric acid in a sealed tube, a substance free from nitrogen was obtained.

The amido-compound is however produced by boiling a mixture of pure nitrochrysene, a little red phosphorus and hydriodic acid (sp. gr. 1.7) until the brown colour changes to a light grey. The powder obtained after washing and drying is the hydriodide of amidochrysene. It is purified by solution in alcohol, filtration, and precipitation with alcoholic caustic potash. Recrystallised from nitrobenzene, benzene, or alcohol, amidochrysene forms a yellowish brown powder, melting at  $201^\circ$ — $203^\circ$  C., and exhibiting in its solutions blue fluorescence.—A. L.

*On some New Diphenyl Derivatives.* E. Täuber. Ber. **23**, 794—798.

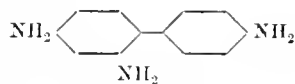
*m*-DINITROBENZIDINE is obtained by dissolving 28.2 grms. ( $\frac{1}{10}$ th mol.) of pure benzidine sulphate in 300 grms. of sulphuric acid at a temperature not exceeding  $50^\circ$ — $60^\circ$  C.

After cooling from  $10^{\circ}$ – $20^{\circ}$  C., 20.2 grms. of potassium nitrate are gradually added and the mixture is allowed to stand for several hours. It is then poured into three times its volume of water, filtered from some impurities and precipitated with sodium carbonate or ammonia. The precipitate is dissolved in hydrochloric acid, boiled with animal charcoal and reprecipitated with ammonia. The base is recrystallised from alcohol and forms yellow plates, melting at  $214^{\circ}$  C. It forms a diazo-compound which can be combined with amines and phenols; but the resulting colouring matters have no affinity for cotton fibre. The bodies obtained from naphthylamine sulphonic acids are bluer than the corresponding benzidine derivatives, whilst the combinations with naphthol sulphonic acid are redder than the benzidine colouring matters.

*m*-Mononitrobenzidine is obtained in the same way as above described, the only alteration being to use half the quantity of potassium nitrate. It forms long red needles like chromic acid, melting at  $143^{\circ}$  C. The colouring matters are in shade and in affinity to cotton, between the benzidine and dinitrobenzidine colours.

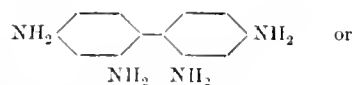
*m*-Diamidobenzidine was prepared by reducing the dinitro compound with tin and hydrochloric acid. After the usual purification the base was obtained in plates, melting at  $165^{\circ}$  C. It does not act on orthodiketones and forms brown colouring matters with nitrous acid. Its salts are with difficulty soluble in water.

*m*-Monamidobenzidine forms long colourless needles, melting at  $134^{\circ}$  C., is capable of producing colouring matters of the Chrysoidine and Vesuvine colours. Its constitution is—

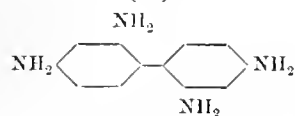


The diamidobenzidine can either be—

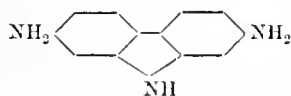
(I.)



(II.)



A body of the former constitution ought to yield by treating with hydrochloric acid under pressure a diamido-carbazol—



As the author did not succeed in obtaining this body, he concludes No. II. to be the correct formula.—A. L.

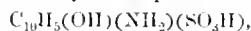
*On α-Hydroxynaphthoic-monosulphonic Acid.* K. König. Ber. 23, 806–810.

$\alpha$ -HYDROXYNAPHTHOIC-MONOSULPHONIC acid loses very easily its carboxyl and sulphonic groups. On boiling the acid with dilute sulphuric acid abundant quantities of  $\alpha$ -naphthol are formed. On heating the acid with water in a sealed tube only  $\alpha$ -naphthol was obtained. If, however, the well-dried acid sodium salt be heated to  $180^{\circ}$  C. the sodium salt of naphtholsulphonic acid is formed.

*Action of Nitric Acid on α-Hydroxysulphonaphthoic Acid.*—On gently heating this acid with an excess of dilute nitric acid dinitronaphthol, melting at  $138^{\circ}$  C., commonly called “Martius Yellow,” is formed. If, however,

hydroxysulphonaphthoic acid be suspended in five times its weight of glacial acetic acid, and 1 mol. of nitric acid sp. gr. 1.48, dissolved in four times its volume of glacial acetic acid, be gradually added, nitrohydroxynaphthoic acid is the only product of the reaction. It is identical with the acid described by Burkard as *p*-nitronaphthol-carboxylic acid. On heating with water to  $150^{\circ}$  C., mononitronaphthol, melting at  $164^{\circ}$  C., from this compound by reduction amidonaphthol, yielding on oxidation  $\alpha$ -naphthoquinone, were prepared. The author concludes that the sulpho group is in the para-position to the hydroxy group.

*Action of Diazobenzenechloride on Sulpho-α-hydroxynaphthoic Acid.*—Diazobenzene chloride combines readily with the sulphonic acid. The colouring matter is, however, nothing but the sodium salt of the benzene-azo- $\alpha$ -naphtholsulphonic acid, the carboxyl group being eliminated in the reaction. By reduction with stannous chloride and hydrochloric acid an amidonaphtholsulphonic acid—



was prepared. It is with great difficulty soluble in water and alcohol; its alkaline solutions soon change to a dark green, on acidulation the tint changing to red.

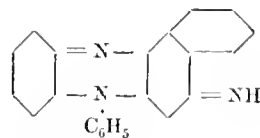
The author further prepared the colouring matter from diazobenzenechloride and Neville and Winther's  $\alpha$ -naphthol- $\alpha$ -sulphonic acid, and proved the complete identity of this dyestuff with the one above described.—A. L.

*On the Action of Chlorine on Pyrocatechol (Catechol) and o-Amidophenol.* Th. Zincke and F. Küster. Ber. 23, 812–831.

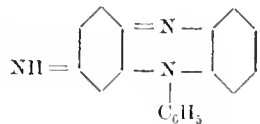
THE authors publish in an explicit manner their views on the theoretical aspect of these reactions, and describe finally  $\gamma$ : $\gamma$ -hexachloro-*R*-pentenehydroxy-carboxylic acid and its phosphoric ester.—A. L.

*On Indulines.* O. Fischer and E. Hepp. Ber. 23, 838–841.

THE simplest induline of the naphthalene series, viz., Rosinduline, has the constitution—

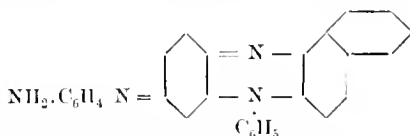


There ought to be therefore a product in the benzene series of the formula—

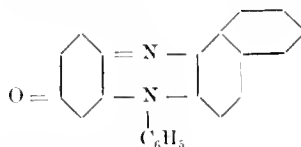
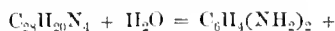


The authors think they have been successful in preparing this colouring matter. The Farbwerke vorm. Meister, Lucius, at Höchst, have obtained a patent for the production of an induline,  $C_{24}H_{18}N_4$ , soluble in water. It is formed in the amidoazobenzene melt, when this is heated only a short time in presence of an abundant quantity of aniline hydrochloride. The new product  $C_{18}H_{13}N_3$  is prepared in an analogous way. A mixture of 160 grms. of amidoazobenzene, 400 grms. of aniline, and 320 grms. of aniline salt are heated until a blue colouration begins to be formed. The reaction commences at  $130^{\circ}$  C. It is continued for 10 minutes at  $160^{\circ}$  C. The mixture is then rendered basic, neutralised with the theoretical quantity of sodium carbonate, and the aniline removed from it by distillation with steam. The residue was boiled with  $1\frac{1}{2}$  litres of acetic acid of 30 per cent. strength; the solution filtered hot and allowed to cool slowly. After several days' standing, a considerable quantity of a crystalline precipitate separated out, which contained

the acetate of the induline  $C_{23}H_{13}N_4$ . The mother-liquor yielded on addition of hydrochloric acid a blue amorphous precipitate, which consists of a mixture of hydrochlorides of  $C_{23}H_{13}N_4$  and  $C_{15}H_{13}N_3$ . The free bases were dissolved in hot benzene and the induline  $C_{23}H_{13}N_4$ , being soluble with far more difficulty in this menstruum, crystallised out first. After repeated recrystallisation the new product was obtained pure, in magnificent needles with a beetle-green lustre, and melting at  $135^\circ C$ . The product was soluble in ether, benzene, and alcohol, with a magenta colour. Its acetic acid solution is reddish violet and it dissolves in sulphuric acid with a blue-violet colour. The hydrochloride, sulphates and nitrates are easily soluble in hot water and dye fibres a reddish violet. Heated with hydrochloric acid to  $150^\circ C$ , it loses ammonia and a new colouring matter is formed, probably  $C_{15}H_{13}N_3O$ , which is redder in its solutions than the original colouring matter. The solutions of the base show a beautiful brownish red fluorescence. If the induline,  $C_{23}H_{13}N_4$ , could be reduced to phenazine the whole induline question would be solved, since all other indulines of the benzene series are derivatives of this, either containing the aniline, phenyl, or paraphenylenediamine residue. The latter acts with particular ease on all indulines, as is well known to manufacturers; and as there are considerable quantities of paraphenylenediamine formed in the process of making indulines, disappearing again if the reaction be continued a long time, there is no doubt that it serves for the formation of the *blue indulines*. In the above described process abundant quantities of paraphenylenediamine could be isolated. The authors further observed that in all processes for preparing indulines the addition of the diamine lowered the temperature of the reaction considerably. Azophenine, anilido-quinoneanilide, anilidonaphtho-quinoneanil, benzene-azo- $\alpha$ -naphthylamine, &c., were acted upon easily, when these bodies were heated in alcoholic solution with paraphenylenediamine and its hydrochloride from  $150^\circ$ — $170^\circ C$ . To decide in which way this base acts on indulines, phenylrosinduline was heated in alcoholic solution with two parts of phenylenediamine and  $\frac{1}{2}$  parts of its hydrochloride to  $160^\circ C$ . A new colouring matter was formed. Its free base was nearly black, crystallising from alcohol in dark needles melting at  $247^\circ C$ . It had the formula  $C_{23}H_{20}N_4$  and the constitution—



If heated with hydrochloric acid to  $180^\circ C$ , it forms paraphenylenediamine and Rosindone—



the latter crystallising in beautiful red plates with a golden lustre, melting at  $259^\circ C$ .

*p*-Phenylenediamine acts on benzene indulines probably in the same way. If it enters them, it changes the shade of the colouring matter from violet to blue.—A. L.

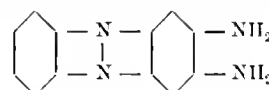
*On the Oxidation of o-Phenylenediamine.* O. Fischer and E. Hepp. Ber. **23**, 841—847.

THE authors discovered some time ago that the oxidation product of orthophenylenediamine formed with ferric chloride, is a diamidophenazine, which has its two amido groups either in one or two different nuclei. They now describe some salts, and the diformyl derivatives of the compound mentioned. The hydrochloride has the formula  $C_{12}H_{10}N_4 \cdot HCl + 3 H_2O$ , the sulphate the formula—



*Constitution of Diamidophenazine.*—Though the formation of the diacetyl- and diformyl-compounds is contrary to the hypothesis that both amido groups are in one ring, there are other characteristics which render it highly probable that these groups are in the ortho position to each other. Nitrous acid does not produce a diazo-compound; benzaldehyde acts on the hydrochloride at  $100^\circ C$ , with evolution of hydrochloric acid (Ladenburg's reaction for orthodiamines), and it forms with benzil a quinoxaline derivative. The latter product is prepared by boiling a mixture of 1 gm. of diamidophenazine, 2 grms. of benzil, and 20 grms. of alcohol for half an hour. Then 5 grms. of glacial acetic acid are added and the boiling is continued for another hour. The product of the reaction separates out after some time, and is purified by crystallisation from toluene. It forms small reddish-brown plates, soluble in concentrated sulphuric acid with a dark blue colour, and has the formula  $C_{20}H_{16}N_4$ .

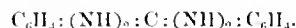
The constitution of diamidophenazine must therefore be—



*Dihydroxyphenazine hydrochloride* is obtained by heating the diamido-compound with 10 parts of concentrated hydrochloric acid in a sealed tube to  $200^\circ C$ . The free base is prepared from the sulphate by addition of sodium carbonate. It crystallises from dilute alcohol in reddish-yellow needles with  $\frac{1}{2}$  mol. of  $H_2O$ , and has the composition  $C_{12}H_8N_2O_2 + \frac{1}{2} Aq$ .

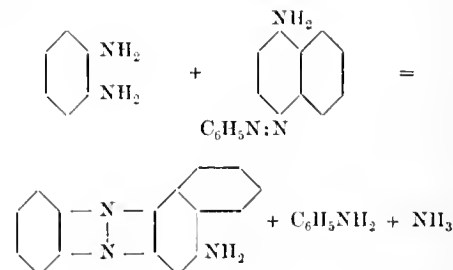
*Diacetyldihydroxyphenazine* is prepared by heating one part of dihydroxyphenazine with 15 parts of acetic anhydride. Light yellow leaves melting at  $230^\circ C$ , and soluble in benzene.

Hübner and Frerichs studied the action of *iodocyanide* on orthophenylenediamine, and obtained a yellow base which they believed to be—



The authors, however, proved that this body is diamidophenazine.

By the action of amido-azo bodies on orthophenylenediamine, monamidophenazines or eurhodines are formed. By the action of 1 mol. of the diamine on 1 mol. of benzene azo- $\alpha$ -naphthylamine, a base  $C_{16}H_{11}N_3$ , was obtained, soluble in alcohol, and melting at  $264^\circ C$ . Its solution exhibits a yellowish-green fluorescence. With an excess of acetic anhydride, it yields an acetyl compound  $C_{18}H_{13}N_3O$ , and on heating with hydrochloric acid in a sealed tube at  $180^\circ$ — $200^\circ C$ , a hydroxy derivative  $C_{16}H_{10}N_3O$ , ammonia being eliminated. On distillation of this body with zinc dust naphthophenazine, melting at  $142^\circ C$ , is formed. The reaction between orthophenylenediamine and benzene-azo- $\alpha$ -naphthylamine must be expressed by the following equation:—



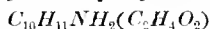
—A. L.

*On Alicyclic and Aromatic Tetrahydro- $\beta$ -naphthylamine.* E. Bamberger and M. Kitchelt. Ber. **23**, 876—884.

FIFTEEN grms. of naphthylamine are reduced with 20 grms. of sodium. The solution of the mixture of bases in petroleum spirit is treated with carbonic acid, or instead of repeating this operation several times, the carbonates are dissolved in acetic acid of 5—8 per cent. strength; the aromatic bases

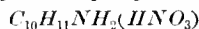
remain undissolved as a brown oil, which can be easily separated. If on addition of diazobenzenesulphonic acid no colouration takes place, the separation is complete.

*Alieyclic Tetrahydro-β-naphthylamine acetate—*



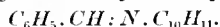
melts at 155.5°—156° C.

*Alieyclic Tetrahydro-β-naphthylamine nitrate—*



melts at 210°—212° C.

*Alieyclic Benzylidenetetrahydro-β-naphthylamine—*

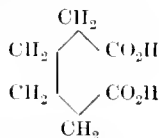


obtained from the base and benzaldehyde at the ordinary temperature, forms tablets or prisms melting at 51.5°—52° C. It is soluble in petroleum spirit, ether, benzene, and boiling alcohol, but insoluble in water. Whilst very stable towards alkalis it is easily decomposed by acids into the base and the aldehyde.

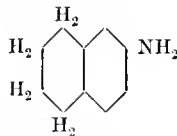
By the oxidation of the base with potassium permanganate a mixture of phthalic acid and orthocarboxylic hydrocinna-monic acid was obtained. If, however, the alieyclic β-tetrahydronaphthylamine be oxidised with potassium bichromate and sulphuric acid, α-naphthoquinone, melting at 123.5° C. and α-naphthol, melting at 95° C. were obtained, besides phthalic acid. This is indeed exceedingly surprising, considering that a pure β-derivative was the material which the authors used.

*Aromatic Tetrahydro-β-naphthylamine* is formed to the extent of about 3 to 4 per cent. on the reduction of β-naphthylamine. To the mixture of crystals of the hydrochlorides remaining after distilling off the amylalcohol caustic soda is added until the mixture becomes alkaline. Thus the aromatic bases are set free, and may be distilled off with steam. They are converted into salts and concentrated. After further addition of caustic soda the base is then extracted with ether, the solution exhibiting a blue fluorescence. On evaporation of the ether the residue is dissolved in petroleum spirit, and this solution is then treated with moist carbonic acid. The filtrate from the solid carbonate is distilled and a product is obtained between 275°—277° C., solidifying and melting after several recrystallisations from hot petroleum spirit at 38° C. It forms salts with mineral acids, can be diazotised, and the diazo-compound yields colouring matters in combination with phenols and amines.

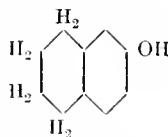
On the oxidation of the base with potassium permanganate adipic acid was obtained, melting at 148° C. and solidifying at 144° C. Its constitution is—



and the base must therefore be—



*Aromatic Tetrahydro-β-naphthol—*



was obtained from the diazo compound of the base above described, by boiling it with dilute sulphuric acid. It melts at 58° C., and is identical with that prepared by reducing β-naphthol.—A. L.

*On Aromatic Tetrahydro-β-Naphthol.* E. Bamberger and M. Kitschelt. Ber. 23, 885—887.

The reduction product of 48 grms. of β-naphthol is distilled at a pressure of 55 mm. until the temperature rises to 196° C. The distillate is treated with caustic soda, which dissolves the aromatic compound. Some, however, of the alieyclic body is mixed with it, and to produce separation, steam is passed through the alkaline solution until no more oil insoluble in alkali distils over. The remaining alkaline solution is acidified, steam again passed through the distillate, saturated with sodium chloride and shaken with ether. On distilling the latter β-tetrahydronaphthol was obtained in white needles, melting at 58° C. It is soluble in ether, alcohol, benzene, chloroform, and boiling petroleum spirit, little soluble in cold, more so in hot water. Calcium hypochlorite separates from its aqueous solution white flakes; ferric chloride colours it a bluish-green, and on heating a brownish-yellow precipitate is produced; chloroform and caustic soda colour its solution greenish-yellow. If aromatic β-tetrahydronaphthol be dissolved in concentrated sulphuric acid and sodium nitrite be added, the solution is coloured pink, the tint on heating changing to a beautiful violet. β-naphthol treated in the same way produces in the cold a reddish-brown solution, which on heating turns to a violet-brown, combined with *p*-diazobenzene sulphonic acid a chert-red colouring matter is formed.—A. L.

*On Quinone-imides and Amido-quinones.* F. Kehrman. Ber. 23, 897—907.

QUINONE-IMIDES are bodies of the typical formula—

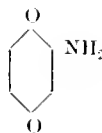


Quinone-imide.

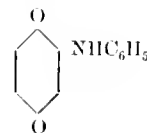


Quinone-di-imide.

Amido-quinones the author considers to be constituted as follows:—



Amido-quinone.

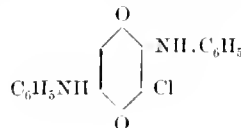


Anilido-quinone.

These classes of bodies have been closely studied of late, and Fischer and, at the same time, Bandrowsky have discovered that azophenine is dianilidoquinonedianilide—



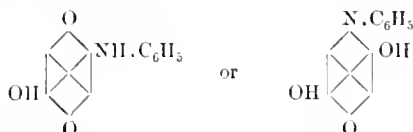
The author studied the action of aniline on monochloro-*p*-dihydroxyquinone. If the latter be treated with an alcoholic solution of aniline, the liquid is at first coloured dark brown and separates somewhat suddenly large brown crystals of a blue violet lustre. This compound proved to be identical with Niemeyer's Chlorodianilidoquinone—



obtained by the action of alcoholic aniline on metadihydroxyquinone.

If, however, to a boiling solution of chlorodihydroxyquinone in glacial acetic acid so much aniline acetate be

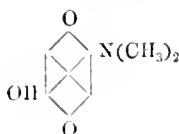
added that the whole of the quinone is converted into the aniline salt separating in red crystals, and the boiling be continued for five minutes, the salt re-dissolves, and on cooling greenish-black needles with a blue lustre are obtained isomeric with chlorodiamidoquinone, melting after recrystallisation from alcohol at 240° C. with some decomposition. The compound is insoluble in water, with difficulty soluble in alcohol and ether, easily in boiling benzene and glacial acetic acid. The solution in concentrated sulphuric acid has a beautiful green colour, changing on dilution with a little water to blue, and precipitating the anilide on mixing with much water. This is soluble in caustic alcoholic potash with a dark red colour. On boiling with dilute caustic potash until the solution is coloured light red, a body is obtained which is either—



Anilidoxyhydroxyquinone. Dihydroxyquinone mono-anilide.

If the boiling be continued until no more smell of aniline can be perceived, the solution contains the potassium salt of monochlorodihydroxyquinone. The author discusses the constitution of these compounds at great length without, however, arriving at any definite formulae.

On treating anilidoxyhydroxyquinone-anilide or anilidoxyhydroxyquinone with very dilute caustic potash until the evolution of aniline ceases, the potassium salt of Nietzki's paradihydroxyquinone is formed. The same body is obtained from diamidoquinone by treating it with alcoholic sulphuric acid from tetramethyldiamidoquinone by treatment with caustic alkalis. If, instead of alkalis, hydrochloric acid be used, dimethylamidohydroxyquinone—



is the first product of the reaction.—A. L.

*On some Derivatives of 1:3 Dichloronaphthalenes.* P. T. Clève. Ber. 23, 954—958.

THE most convenient method for the preparation of this compound is to form it from dichloronaphthylamine. It is purified by distillation with steam, and boils at 289° C., at a pressure of 775 mm.

If a solution of this body in chloroform be saturated with chlorine at ordinary temperatures, and then treated with alcohol, a quantity of white needles are precipitated, melting after crystallisation from hot alcohol at 92° C. The trichloronaphthalene, C<sub>10</sub>H<sub>7</sub>Cl<sub>3</sub> [1:3:4] is identical with the compound formerly obtained from dichloronaphthol and phosphorus pentachloride.

On oxidation of dichloronaphthalene with 2½ parts of chromic acid, monochloronaphthoquinone, melting at 115° C. and crystallising in long yellow needles, and phthalic acid, were obtained. The author prepared from the quinone, anilidomonochloronaphthoquinone (dark red needles, melting at 207° C.), Chloronaphthoquinone-oxime, C<sub>10</sub>H<sub>7</sub>ClO.NOH (yellowish needles melting and decomposing at 200° C.).

Ordinary nitric acid, sp. gr. 1.2, does not act on this dichloronaphthalene; a mixture of nitric acid and sulphuric acid forms two dinitro-compounds; with an excess of nitric acid trinitrodichloronaphthalene is produced.

*Dinitrodichloronaphthalene I.*, C<sub>10</sub>H<sub>4</sub>(NO<sub>2</sub>)<sub>2</sub>Cl<sub>2</sub>.—If the mixture of the nitration product be crystallised from glacial acetic acid, fine nearly colourless needles are obtained, containing acetic acid of crystallisation. The compound melts at 150° C., and is soluble in benzene. From the

mother-liquor the isomeric *Dinitrodichloronaphthalene II.*, crystallises in small white needles, melting at 158° C. Both compounds yield on further nitration—

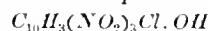
*Trinitrodichloronaphthalene*, C<sub>10</sub>H<sub>3</sub>(NO<sub>2</sub>)<sub>3</sub>Cl<sub>2</sub>.—Pale yellow needles, melting at 178° C. It forms with ammonia,

*Anidochlorotrinitronaphthalene*, C<sub>10</sub>H<sub>3</sub>(NH<sub>2</sub>)Cl(NO<sub>2</sub>)<sub>3</sub> (yellow needles melting at 252° C.) and with aniline,

*Anidochlorotrinitronaphthalene*—



(red flakes melting at 230° C.), whilst alcoholic caustic soda converts it into *trinitrochloronaphthol*—

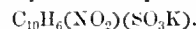


crystallising from glacial acetic acid in pale yellow needles, melting and decomposing at 156° C.—A. L.

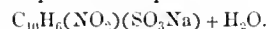
*1:4 Nitronaphthalenesulphonic Acid.* P. T. Clève. Ber. 23, 958—961.

On titration of the sodium salt of naphthalene-α-sulphonic acid, three isomerides are obtained: 1:5, 1:8, and 1:4 nitronaphthalenesulphonic acids. The latter yields on reduction naphthionic acid. To prepare these bodies the author proceeded in the following way: 1 kilo. of the sodium salt is gradually added to a mixture of 750 grms. of nitric acid of sp. gr. 1.4, and of 500 grms. of nitric acid of sp. gr. 1.5. After dilution the calcium salts are formed, and their solution is filtered whilst hot. On cooling, the calcium salt of the 1:5 acid crystallises out. The mother-liquor is concentrated, and the calcium salts decomposed with sulphuric acid. On evaporation small crystals of the 1:5 acid are obtained. The mixture of the remaining acids is converted into their potassium salts and then by means of phosphorus pentachloride into sulphonic chlorides. The first fractions yielded nearly pure 1:5 nitronaphthalenesulphonic chloride, whilst the remaining formed a mixture of the 1:5 and 1:4 chlorides, from which the latter was obtained by crystallisation from benzene and petroleum spirit, and melted at 99° C. The 1:8 acid forms the most soluble salts, and is contained in the mother-liquor after the 1:5 and 1:4 salts have crystallised out. The author describes the following compounds of the 1:4 acid:—

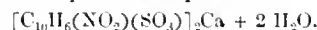
*Potassium Nitronaphthalenesulphonate*—



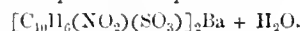
*Sodium Nitronaphthalenesulphonate*—



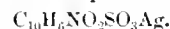
*Calcium Nitronaphthalenesulphonate*—



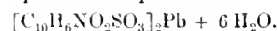
*Barium Nitronaphthalenesulphonate*—



*Silver Nitronaphthalenesulphonate*—



*Lead Nitronaphthalenesulphonate*—



*The Ethyl Ester*, C<sub>10</sub>H<sub>6</sub>NO<sub>2</sub>SO<sub>3</sub>C<sub>2</sub>H<sub>5</sub>, melting at 98° C.

*The Methyl Ester*, C<sub>10</sub>H<sub>6</sub>NO<sub>2</sub>SO<sub>3</sub>CH<sub>3</sub>, melting at 117° C., and the amido compound, C<sub>10</sub>H<sub>6</sub>NO<sub>2</sub>SO<sub>2</sub>NH<sub>2</sub>, melting at 188° C. The latter yields on reduction with glacial acetic acid, hydriodic acid and phosphorus, and on precipitation of the solution with ammonia, the amide of naphthionic acid, melting at 206° C. Its formula is C<sub>10</sub>H<sub>6</sub>NH<sub>2</sub>(SO<sub>2</sub>NH<sub>2</sub>). —A. L.

*On 1:8 Chloronaphthalenesulphonic Acid.* P. T. Clève. Ber. 23, 962—963.

On treating the potassium salt of 1:8 nitronaphthalenesulphonic acid with phosphorus pentachloride a very

violent reaction sets in. To moderate the reaction, the salt was diluted with carbon bisulphide, and then phosphorus pentachloride added. Nitrous acid vapours escaped, and 1 : 8 chloronaphthalenesulphonic chloride, melting at  $101^{\circ}\text{C}$ ., was obtained. On boiling with barium hydrate, the barium salt,  $(\text{C}_{10}\text{H}_6\text{ClSO}_3)_2\text{Ba} + 2\text{H}_2\text{O}$ , was formed. There are further described the *Ethyl ester*,  $\text{C}_{10}\text{H}_6\text{ClSO}_3\text{C}_2\text{H}_5$ , melting at  $67^{\circ}\text{C}$ .; the *Methyl ester*,  $\text{C}_{10}\text{H}_6\text{ClSO}_3\text{CH}_3$ , melting at  $70^{\circ}\text{C}$ .; and the *amide*,  $\text{C}_{10}\text{H}_6\text{ClSO}_2\text{NH}_2$ , melting at  $196^{\circ}\text{--}197^{\circ}\text{C}$ .—A. L.

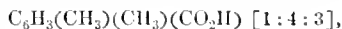
*On Ethylxylenes.* J. Stahl. Ber. 23, 988—991.

By the action of ethyl bromide and sodium on the monobromo derivatives of the three isomeric xylenes, there were obtained—

*Ethylmetaxylene*,  $\text{C}_6\text{H}_3(\text{CH}_3)(\text{CH}_3)(\text{C}_2\text{H}_5)$  [1 : 3], boiling at  $185^{\circ}\text{--}186^{\circ}\text{C}$ ., and forming a sulphonic acid on treatment with concentrated sulphuric acid. With a mixture of nitric and sulphuric acid, *Trinitro-ethylmetaxylene*,  $\text{C}_6(\text{NO}_2)_3(\text{CH}_3)_2\text{C}_2\text{H}_5$ , melting at  $127^{\circ}\text{C}$ ., is prepared.

*Tribromo-ethylmetaxylene*, melting at  $94^{\circ}\text{--}95^{\circ}\text{C}$ ., is the product of the action of an excess of bromine on the hydrocarbon. On oxidation with dilute nitric acid, *Nitrylic acid*,  $\text{C}_6\text{H}_3(\text{CH}_3)(\text{CH}_3)(\text{CO}_2\text{H})$  [1 : 3 : 4], melting at  $125^{\circ}\text{C}$ ., is formed.

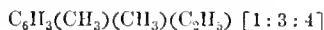
*Ethylparaxylene* boils at  $185^{\circ}\text{C}$ ., and yields a sulphonic acid, which, on fusing with caustic potash, is converted into *Ethylparaxyleneol*, melting at  $37^{\circ}\text{C}$ ., and boiling at  $245^{\circ}\text{C}$ . Ferric chloride does not act on its aqueous solution, but produces in its alcoholic solution a deep green colouration. On oxidation of the hydrocarbon, isoxilylic acid,—



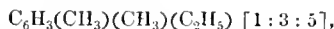
was obtained.

*Ethylorthoxylene*,  $\text{C}_6\text{H}_3(\text{CH}_3)(\text{CH}_3)(\text{C}_2\text{H}_5)$  [1 : 2 : 4], boils at  $187^{\circ}\text{--}188^{\circ}\text{C}$ ., and is easily converted into a sulphonic acid, forming a *trinitro derivative*, melting at  $121^{\circ}\text{C}$ ., also a *tribromo compound*, melting at  $93^{\circ}\text{C}$ ., and on oxidation *paraxilylic acid*,  $\text{C}_6\text{H}_3(\text{CH}_3)(\text{CH}_3)\text{CO}_2\text{H}$  [1 : 2 : 4], melting at  $163^{\circ}\text{C}$ .

By the action of ethyl bromide on metaxylene in presence of aluminium chloride, two isomerides are obtained: the one before described, ethylmetaxylene—



and—



boiling at  $188^{\circ}\text{C}$ . It forms a sulphonic acid, and, on oxidation, *mesitylenic acid*.—A. L.

*On Amidochrysene.* E. Bamberger and C. Burgdorf.

Ber. 23, 1006—1007.

THE authors obtained amidochrysene by reducing nitrochrysene with tin and hydrochloric acid, thus obtaining a tin double salt.—A. L.

*Dyewoods and Extracts, their Use and Abuse.* L. Bruehl. Textile Colourist, 1889, 125, et seq. (This Journal 1889, 612—618.)

*Fustic*.—Fustic is the product of the tree, *Morus tinctoria*, which is indigenous to the West India Islands, especially Jamaica and Cuba, Mexico and Brazil. It contains two different dyestuffs, viz.: morin or moric acid, and maclurin or moritannic acid. Maclurin, or moritannic acid, is readily soluble in water: 6 parts cold water, or 2·18 parts boiling water dissolve 1 part maclurin. Morin is nearly insoluble in water (1 part morin dissolves in 4,000 parts cold water or in 1,060 parts boiling water), readily soluble in caustic alkalis and in alkaline borates and phosphates, and is

precipitated from such solutions by acids. Morin is also readily soluble in alcohol and ether.

The kinds of fustic most frequently offered in the European market are: Tampico fustic, Tuspan fustic, Yellow Pernambuco wood, East India fustic, San Domingo fustic, which denomination applies to all yellow woods from the West Indies besides; finally, Cuba fustic.

The commercial classes of fustic greatly differ from each other in their contents of dyestuff, the brands Cuba, San Domingo and Tampico being considered the best. The so-called East India fustic of commerce is very poor in extract, and scarcely pays for working; it is, therefore, principally used for fine cabinet work.

It is generally held that Cuba wood, especially the brand "Cuba Coast" is to be considered the best. It has been found that the quantity of extract contained in the wood is very variable, and that in the different logs from the same cargo of wood as many different percentages of dyestuff extract may be found, which with good wood range from 6 to 18 per cent. The average yield of the wood ought to be 10—12 per cent., and only seldom woods are encountered which in industrial working give a yield of 15—18 per cent. Considering the structure of the wood it is particularly necessary with fustic, on taking samples, to be very cautious in judging it, because different parts of the same tree often yield very different quantities of extract. In the interior of the wood often deposits of a yellow powder are found, and of a red resinous matter, which substances are very rich in colouring matter, indeed, they are nearly pure dyestuff. If it happens in taking a sample, that a portion of the wood is struck, where there is such accumulation of colouring matter, a yield of over 40—60 per cent. extract is easily obtained. A light yellow colour and numerous red veins appearing in the structure of the wood, are regarded as general characteristics of a good fustic.

The analyses of various brands of fustic which follow, illustrate the difference between the quantity of extract and quantity of colouring matter.

Name of Wood.	Water.	Extract.	Dyestuff.	Ash.
	PerCent.	PerCent.	PerCent.	PerCent.
Tampico fustic, 1884 .....	15·2	14·6	11·32	1·4
Cuba fustic, 1884 .....	14·3	15·35	12·71	0·96
Maracaibo fustic, 1885 .....	14·31	13·64	10·91	1·33
San Domingo fustic, 1887 ..	12·73	12·1	10·46	0·99
Cuba fustic, 1886 .....	10·8	13·17	9·09	1·37
Cuba coast, 1887 .....	10·3	14·32	11·4	1·45
Cuba coast, 1885 .....	9·7	15·81	13·67	1·03
Tuspan fustic .....	14·12	13·5	11·68	1·00
East India fustic .....	14·6	6·19	4·00	2·38

This table shows very distinctly that the quantity of extract obtained from a wood is not sufficient evidence as to the tinctorial value of the latter, since there are woods found which with a yield of extract of 13·64 per cent. represent a quantity of 10·91 per cent. contained therein (Maracaibo), while Cuba 1886, yielding 13·17 per cent. extract represents 9·09 per cent. dyestuff. On the other hand it can also be observed that there are differences between different crops of the same kind of wood. If we compare the different cuttings of Cuba fustic, we find the following:—

Cuba.	Extract.	Colouring Matters.
	Per Cent.	Per Cent.
1884	15·35	12·71
1885	15·81	13·67
1886	13·17	9·39
1887	14·32	11·14



These figures demonstrate plainly how the difference between the quantities of extract matter and of colouring matter contained in a wood operates in practice. Persons who have not all the facilities at their disposal to carry out an accurate examination of the wood, ought never to estimate the tinctorial value from the yield of extract; they will arrive at a more correct estimate by making a dye-trial.

The *morin* of fustic being nearly insoluble in water, it is unable to diffuse in the dye-baths and thus to be utilised. For dyeing practice, therefore, only the quantity present of *maclurin* is of importance, and this ranges between 6 and 12 per cent. Only with few woods a higher tinctorial value in *maclurin* is obtained, unless they are lixiviated by the aid of substances which are solvents for *morin*. In this case the yield of the wood will appear a little higher; in the dyeing process, however, such wood extracts will not be able to give results corresponding with the yield of extract, but will always, in trial dyes with equal quantities of extract, give inferior results with extracts which are rich in *morin*.

After carrying out the processes most used in Germany and England with various classes of wood and extracts, the author arrived at the general conclusion that the addition of borax in the extraction, in other words, the presence of borax in the extract, is of no use whatever.

Of equally doubtful value is the addition of carbonate of soda to the extraction liquids, which is practised in some extract works. The author has often pondered the utility of this addition, and on undertaking experiments in this direction has obtained no results which would practically justify the addition of soda. The extracts produced with the addition of soda show a little redder colour. It is not the alkaline carbonates, but only the caustic alkalis which can effect a more rapid solution of the fustic dyestuffs.

While the extract manufacturer buys his fustic in sticks or logs, the product is offered to the dyer in chips or in powder. As regards the way of chipping, there are sold in Germany cross-cut chips, that is, such as are produced by cutting the log in a perpendicular direction to its axis (across the grain) and long-cut chips, produced by cutting parallel to the axis, in the direction of the structure of the fibre (with the grain). Many dyers prefer the long cut for good reasons, and about the usefulness of the long cut there can be said at least as much *pro* as *contra*. The advantage of the long cut is, that it is softer to the feel and gives not so much powder as the cross cut. The pieces and yarns dyed with such wood are easier to clean from the adherent particles of wood; and there is no danger of tearing the pieces upon the washing machines, which can very readily occur with cross-cut chips, unless they are very fine. A disadvantage of the long cut, however, and which is of a very serious character, is that it is difficult to extract, and when the thickness of the chip is over 0.5 mm., considerable quantities of residual colouring matters remain in the chip, which represent no inconsiderable loss in dyestuff for the dyer. The manufacturers of extract, therefore, always use cross-cut chips for extraction.

There are inserted here the results of a comparative experiment of extraction with cross-cut and long-cut chips. The extraction was carried out in a high pressure apparatus with only  $\frac{1}{2}$  atm. pressure. The quantity of water was calculated in the proportion of 1:3.

#### EXTRACTION OF CROSS-CUT CHIPS. 10 KILOS. WOOD.

Operation.	Concentration of Decoction in 1° B.	Extract in Grms.	Residue in the Wood.	
1	Extract 1° B.	666	Unknown.	
2	Extract 1° B.	666	Unknown.	
3	Extract 0.5° B.	333	Unknown.	
4	Extract 0.2° B.	133	42.6 grms.	Per Cent. 9.426
	Total .....	1532	Yield ....	15.32
	Total contents of the wood .....			18.13

#### EXTRACTION OF LONG-CUT CHIPS. 10 KILOS. WOOD.

Operation.	Concentration of Decoction in 1° B.	Extract in Grms.	Dissolve in the Wood.	
1	Extract 1° B.	666	Unknown.	
2	Extract 0.9° B.	600	Unknown.	
3	Extract 0.3 B°.	200	Unknown.	
4	Extract 0.1° B.	66	281 grms.	Per Cent. 2.81
	Total .....	1,798	Yield ....	17.980
	Total contents of the wood .....			18.406

After the lixiviation an average sample was taken from each sample of wood and dried, and 100 grms. of each completely extracted with distilled water in Schroedler's extractor, when the cross cut still gave 0.426 per cent. residue, and the long cut 2.81 per cent., whereby the total yield of one wood (cross cut) amounts to 18.406 per cent., that of long cut to 18.13 per cent. Considering the unavoidable losses occurring by the extraction in the experimental apparatus, in evaporating the decoctions, &c., the wood was therefore considered of equal value.

Herefrom results a difference of 2.374 per cent. from the extractions which are obtainable in industrial working, that is, for the dyer who uses long-cut chips, a loss of 2.374 kilos. extract for every 100 kilos. chips which, taking the price of extract at 140 mk. per 100 kilos. extract, represents in cash a loss of 3 mk. 32 d. Besides, the price of long-cut chips is higher than that of cross cut. Cuba long-cut fetching, in 1888, 15.50 mk. per 100 kilos., the loss of 3.32 mk. represents about 20 per cent. of the total value of the wood, which loss, considering the present low dyeing prices, is a matter which requires due consideration.

Similar to the case of logwood is that of fustic which is sold in the form of chips. The quality and purity of these woods is very variable, and principally depends upon the price. Fustic in a finely reduced condition has also the property of absorbing water in considerable quantity. The normal proportion of water contained in the wood ranges from 12 to 15 per cent., while chips may often contain 18–24 per cent. of water and no noticeable alteration of it can be discovered, particularly when presented in the shape of long-cut. It is easy to understand that this property of the woods is largely taken advantage of to the disadvantage of the consumer, who must buy and pay gross for net according to the weight indicated by the bill of lading. Another largely practised adulteration is that with woods of inferior quality, such as chestnut wood, waste of quercitron bark, turmeric powder, and finally with fustic chips which have already been extracted. Of the latter adulteration the most extensive use is made where the object is to blind the consuming dyer with a low price and fine cut. The author has met with cases where the chipped wood was mixed with about 40 per cent. of extracted chips and about 5 per cent. turmeric powder. Such a mixture was then sprinkled with water containing about 5 per cent. of soda and left in a pile for several days, using the precaution to several times shovel it over. By this treatment the wood assumes a uniform colour and the lixiviated chip could only with difficulty be distinguished from the non-lixiviated even by means of the microscope.

It is impossible, even with the most careful extraction of the woods, to obtain the whole quantity of colouring matter contained in the wood, but there always remains in the chips a part of the dyestuff soluble in water and, with a well conducted extraction, about 0.5 to 1 per cent. must be allowed for this loss. This residue, however, does not for the extract-maker represent 0.5 or 1 per cent., but with an effective percentage of 15 per cent. extract contained in the wood, a loss of 3.3 per cent., or otherwise 6.6 per cent. of the value of his wood in extract; it is, therefore, a matter of course, that ways and means are sought for to reduce this loss to a minimum. Besides the additions of borax and soda which, as stated, do not answer the purpose, the method of lixiviating under high pressure is applied. As

in the case of logwood, in this case also higher yields in extract are obtained, but, unless this extraction is carried out with great caution, it will be at the cost of the quality of the extract. It is an undisputed fact that by working with high pressure, the last traces of the dyestuff soluble in water can be extracted; but there enter also into the extract other extractive matters present in the wood together with particles of wood mechanically carried over, which do not contribute to the increase of the tinctorial value of the extract. A comparative trial dye with extracts obtained with high pressure in greater amount, as against such as had been produced without high pressure, showed in the shades the same graduation but in inverse proportion to the pressure, that is, it became manifest that the extract which had been produced without pressure gave the best results, and the extracts prepared with pressure gave dyes which were weaker in the same ratio as the pressure and yield increased. The experiment of treating the respective extracts with alcohol was now tried, the residue which had been left was dissolved in water and allowed 48 hours to settle, when a sediment was deposited which proved to be cellulose; and cellulose could also be discovered, both in the aqueous and alcoholic extract, and that in quantities increasing proportionally to the pressure applied in the extraction.

**Sumac.**—The commercial sumac is obtained from the various species of *Rhus*, such as *Rhus coriaria*, *Rhus cotinus*, *Rhus glabrum*, *Rhus canadensis*, *Rhus typhinus*, *Rhus pentaphyllum*, and *Rhus uva ursi*. The principal species sold in the European markets and most valued are the Sicilian sumac (*Rhus coriaria*) and that from Central Italy (*Rhus cotinus* and *Rhus coriaria*), which plants are not only collected in Central Italy, but also in Dalmatia, Istria, and Montenegro, and are commercially known as Fiume sumac. Besides, there is Spanish sumac and French sumac. Of the latter, four brands are distinguished which, however, belong all either to *Rhus coriaria myrtifolia* or *Rhus pentaphyllum*, according to the regions where found, as Fauvis sumac (Département du Var), Redoul sumac (Gironde), Pudis sumac (South France), and Dongère sumac (Rhône). Finally, American sumac is obtained from various species of *rhus*, the best being that derived from *Rhus glabrum*, *Rhus canadensis*, and *Rhus typhinus*. More recently another tannin-bearing plant is often offered in the market as sumac under the name of Swedish sumac. Although of very inferior quality, this product, obtained from *Arbutus uva ursi* and *Vaccinium vitis*, is fraudulently sold for sumac, the deception being easy to carry out, as both leaves and twigs of these plants very much resemble those of the various species of *rhus*. Only a close examination shows the difference.

The principal value of sumac consists in the proportion of real tannin which it contains, or rather a tannic acid which, though not identical with the tannic acid of nutgalls, produces similar reactions as the latter, or possesses similar properties. The quantity of dyestuff contained in sumac plays in practice only a subordinate part.

It seems that there are several tannic acids contained in sumac, some of which resemble the tannic acid of nutgalls (tannin), while the others are similar to gallotannic acid. The former are designated as actual value in tannin of sumac, the latter give the so-called gallotannic value. Of actual value for the tanner and dyer is only the quantity of tannin contained in sumac, because only this class of tannic acid compounds has the property of forming insoluble salts with metallic oxides, and thus serving for the fixation of mordants, and as mordants for aniline and other dyestuffs. About the value of gallotannic acid in dyeing, opinions are greatly divided.

The tannic acids readily soluble in water are pretty completely precipitated from their solutions by various reagents, as by sulphuric acid, many salts and solution of glue. Of all these reagents, however, only the solution of glue has been adopted for practical use according to the propositions of Meunier and Warrington, who invented a method of determining tannin by titration. This method was declared insufficient by G. Mueller, who demonstrated that an exact quantitative analysis by this process is impossible, as the precipitate formed does not perfectly separate out, and the

solution remaining turbid prevents the observation of the end of the reaction. Although an addition of alum in some degree removes this difficulty, this method, despite all modifications added to it by Mueller, F. Schulze, and others, remains inaccurate. Loewe showed that many tannic acid bearing materials, sumac included, contain besides it, other substances soluble in water, which act like tannin, and thereby render the correctness of the analysis problematical.

Since thus the method of precipitating tannic acid with glue was done away with, scientists turned their attention to the use of metallic salts capable of precipitating tannic acids as insoluble precipitates, and H. Fleck pointed out the property of acetate of copper of completely precipitating tannic acid as well as gallotannic acid. Since cupric gallotannate is perfectly soluble in a solution of ammonium carbonate, while cupric tannate is insoluble in this reagent, the road towards exact examination again seemed to have been found. But this method was also controverted by many chemists, and later on modified by Eder, Hayer, and others, though finally rejected as insufficient.

Hardtke introduced ferric acetate instead of the cupric salt as precipitating agent, but this method of titration as well as that of Gerland (with solution of tartar emetic) proved inaccurate. Only Hammer succeeded in finding the correct way to determine tannin. He based his method upon the property of tannic materials to give insoluble precipitates with the animal skin. Although Hammer's original process was very complex, it gave in combination with other methods the basis for the determination of the tannins.

The final decisive step towards improvement was taken by Loewenthal. This chemist based his method upon the oxidisability of the tannins by potassium permanganate or chloride of lime, using a surplus of indigo carmine as indicator. This method, combined with that of Hammer, forms at present the principal basis of all determinations of tannin, and the improvements introduced in the combined (or, as it is called, improved) method of Loewenthal only concern the way of proceeding, but not its principles. (This Journal, 1884, 82 and 524; also 1885, 263.)

In order, now, to determine the value in tannin, a certain quantity of the substance to be examined is weighed off, say 5 grms. sumac, perfectly extracted with boiling water, and the decoction made up to 1 litre. For the test, 20 ccm. of the decoction are placed in a beaker, and 750 ccm. distilled water, 10 ccm. of 1·10 normal sulphuric acid, and 20 ccm. solution of indigo carmine (of known percentage) are added. Then the oxidising liquid, in this case permanganate of potash (3 litres water and 4 grms.  $\text{KMnO}_4$ ) is introduced from a burette until the original blue colour of the solution to be titrated is changed to straw yellow. According to the quantity of solution of permanganate the total value in tannin and gallotannic acid is then calculated.

The author calls attention to the fact that it is an error to believe that the tannin of sumac is identical with the tannin of nutgalls or of other tanners' plants. On the contrary, nearly every tannin-bearing plant has a specific tannin of its own, which resembles the tannic acid of nutgalls only in the properties of giving with certain metallic salts precipitates which are insoluble in water, and of tanning the animal skins to be precipitated by the latter.

It was found that from the yield of extract of a certain kind of sumac no conclusion as to its value in tannin can be drawn. While a yield of 30 to 40 per cent. extract is by no means a rare occurrence, no sumac was discovered that contained over 25 per cent. actual tannin. Good sumac averages 10—15 per cent. actual tannin, its value in extract ranging from 30—40 per cent. It is therefore a great mistake if, for the valuation of a given kind of sumac, simply a decoction of it with a certain quantity of water is made, its specific gravity found, and the estimation of the value of the test-sumac made accordingly. This method may have special value for the extract maker, but to the dyer and tanner it affords absolutely no reliable indication of the value of the article; and yet this method of valuation is practically applied in many dyehouses and tanneries.

In the commercial sumacs, besides sand and yellow earth (loam) sometimes up to 15 per cent. refuse of various tanning barks of inferior value, such as chestnut wood,

quebracho wood, also quercitron bark and waste of fustic, are found as adulterants. As regards the latter two adulterations, it seems that their object is to produce the colour peculiar to the concomitant dyestuff which is contained in sumac, especially when the sumac in question either naturally contains but little of this accompanying dyestuff, or when its colour has been degraded by other additions or by fermentation of the leaves in a damp store. But as the principal value of sumac, not to say its only value, depends upon its content of tannin, the additions of other colouring bodies appears to be a useless waste of the latter. Sand and loam go mostly together with waste of chestnut wood used for adulteration; probably the tannin contained in chestnut wood (which, besides, is about 50 per cent. cheaper than sumac) is to compensate the deficiency in tannin which is produced by the admixtures of loam and sand. What trouble such admixture of another tannic acid bearing material together with a dark colouring companion dyestuff may cause the dyer, anyone may judge by contrasting a dye with chestnut or quebracho with a sumac dye.

#### PATENTS.

*Improvements in and relating to the Manufacture of Colouring Matters.* H. H. Lake, London. From A. Leonhardt and Co., Mühlheim-on-the-Maine, Germany. Eng. Pat. 4387, March 29, 1886. Amended December 8, 1887. (Second Edition.) 6d.

THE subject-matter of this patent has already been abstracted (this Journal, 1887, 507). In the original specification the acid obtained by the action of caustic soda and zinc dust on *p*-nitrotoluenesulphonic acid was designated a "new anidosulpho acid." This is amended to a "disulpho acid of diamidostilben." The other amendments provide for varying the shades of the dyestuffs obtained, either by combining the tetrazo-compound from diamidostilbene disulphonic acid with dissimilar phenols or amines, or by mixing the various dyestuffs themselves.

—T. A. L.

*The Manufacture of Azo-Dyes and of Materials therefor.* B. Willcox, London. From "The Farbenfabriken vormals Friedrich Bayer and Co.," Elberfeld, Germany. Eng. Pat. 7802, May 9, 1889. 8d.

THE *o*-benzidine disulphonic acid, obtained by reducing *m*-nitrobenzene sulphonic acid in an alkaline solution and transforming the hydrazo-compound into the diamido-compound, gives tetrazo dyestuffs which do not dye unmordanted cotton from an alkaline bath. It has, however, been discovered that by heating the disulphonic acid with caustic alkalis at a pressure of 30–40 atmospheres and at 150°–300° C., it is converted into diamidodiphenylene oxide, which when diazotised and combined with phenols and amines or their sulphonic acids gives direct dyestuffs for cotton. In like manner the *o*-tolidine disulphonic acid obtained from *o*-nitrotolylene-*m*-sulphonic acid can be converted into diamidoditolylene oxide.—T. A. L.

*Manufacture of a New Colouring Matter of the Induline Series.* O. Imray, London. From "The Farbwerke vormals Meister, Lucius und Brüning," Höchst, Germany. Eng. Pat. 7857, May 10, 1889. 4d.

A METHOD for obtaining from the induline melt a bluish-violet colouring matter having the formula  $C_{21}H_{18}N_4$ , dependent upon the fact that only this base is capable of forming a stable acetate. 5 kilos. of aniline, 3.9 kilos. of aniline hydrochloride, and 2 kilos. of amidoazobenzene are heated together. The melt is then boiled two or three times with a large quantity of water and filtered. The filtrates are treated with hydrochloric acid and the precipitate thus obtained filtered, pressed, and dried at 70°–80° C. This mixture is then heated with sodium acetate under pressure, in which case the acetate of the base  $C_{21}H_{18}N_4$  remains in solution, and after filtration can be dried down. As an alternative method the hydrochlorides obtained above are decomposed with dilute caustic soda, washed, and the mixture of the bases whilst still moist boiled in an aqueous

solution with acetic acid, filtered and evaporated to one-fourth of its volume, when the acetate crystallises, forming blue leaflets with a brown lustre. In place of acetic acid, tartaric, citric, or levulic acid may be employed.—T. A. L.

*Improvement in the Manufacture of Amidophenols.* H. H. Lake, London.—From A. Leonhardt and Co., Mühlheim-on-the-Maine, Germany. Eng. Pat. 8156, May 16, 1889. 4d.

A PROCESS for producing *m*-amidophenol and its alkylated derivatives by heating resorcinol with ammonia or a fatty amine at about 200° C. under pressure in an aqueous solution.—T. A. L.

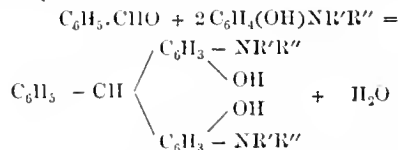
*Improvements in or connected with the Manufacture of Azo-Colouring Matters for Printing and Dyeing.* B. Willcox, London. From "The Farbenfabriken vormals Friedrich Bayer and Co.," Elberfeld, Germany. Eng. Pat. 8299, May 18, 1889. 8d.

A PROCESS for combining (1) the diazo-compounds of amido carboxylic acids, such as the amido benzoic acids, amido hydroxybenzoic acids, amido phthalic acids, and the sulphonated amido cresol carboxylic acids, &c., with 1:8 and 1:2 di-oxy-naphthalenes and  $\alpha$ - and  $\beta$ -naphthol carboxylic acids; (2), the diazo-compounds from the amido benzoic acids, the amido phthalic acids, and the amido anisic acids with salicylic acids and cresol carboxylic acids; and (3) the diazo-compounds of the amido carboxylic acids referred to in (1) with one molecule of  $\alpha$ -naphthylamine, rediazotising the amido azo-compounds and combining them with amines, phenols, and their sulphonic and carboxylic acids. The dyestuffs thus formed are to be used for dyeing and printing, generally with the aid of mordants, although some of them are capable of dyeing wool directly. The colours are said to be absolutely fast to soap, milling, and sunlight. The shades obtained vary from a bright yellow and brown to a deep blue-black according to the components.—T. A. L.

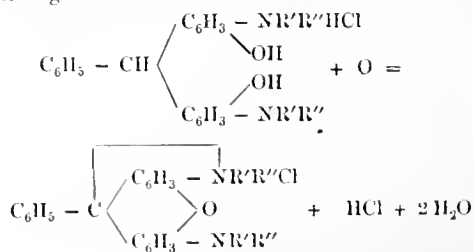
*Improvements in the Manufacture of Colouring Matter and of Leuco Bases employed therein.* B. Willcox, London. From "The Farbenfabriken vormals Friedrich Bayer and Co.," Elberfeld, Germany. Eng. Pat. 8673, May 24, 1889. 8d.

THE red colouring matters formed according to this invention are obtained from *m*-amidophenol, its alkylated derivatives and hydroxydiphenylamine, by heating them with a variety of substances with or without a dehydrating agent. The patent is divided into three parts, as follows:—

I. Leuco bases of the triphenyl methane series are obtained by condensing benzaldehyde and its derivatives with the substances mentioned above according to the general equation—



The substance thus obtained when heated with a dehydrating agent is represented as decomposing in the following manner—



This equation is represented somewhat differently in the provisional specification. The products obtained dye wool and mordanted cotton the same shade as rhodamine and show a brilliant fluorescence on silk.

II. Red basic colouring matters are formed by heating methylene chloride, bromide or iodide, acetone, or chloroform with two molecules of benzil or nitrobenzil and four molecules of an alkylated *m*-amidophenol.

III. Similar colouring matters are also obtained by heating the di-alkylated *m*-amidophenols with phosgene under pressure at 180°, and treating the resulting tetra-alkylated di-hydroxydiamidobenzophenones with dehydrating agents.

Finally, a number of substances are mentioned, which when heated with an alkylated *m*-amidophenol at various temperatures yield red basic colouring matters, such as paraldehyde, unsymmetrical diphenyl-trichloro-ethane or diphenyl-dichloro-ethylene, benzal chloride, benzotrichloride, formic acid, or a mixture of glycerol and oxalic acid.

—T. A. L.

*Improvements in the Manufacture of Basic Colouring Matter.* B. Willcox, London. From "The Farbenfabriken vormals Friedrich Bayer and Co.," Elberfeld, Germany. Eng. Pat. 8750, May 27, 1889. 8d.

BLUE colouring matters are obtained, according to this invention, by combining diazo-compounds or their sulphonie acids with the alkylated derivatives of *m*-amido-phenol and subsequently heating these products with  $\alpha$ -naphthylamine or an alkylated derivative thereof in presence of a suitable solvent at 150°–200° C. The reaction may also be performed in another way by combining the diazo-compounds with  $\alpha$ -naphthylamine or its alkylated derivatives and heating these compounds with alkylated *m*-amido-phenols as above. The second phase of the reaction which takes place is expressed as follows:—"One molecule of benzene-azo-dimethyl-*m*-amido-phenol + one molecule of  $\alpha$ -naphthylamine gives aniline + blue colouring matter." The following example illustrates the process. About 12 kilos. of benzene-azo-di-methyl-*m*-amido-phenol, 6 kilos. of hydrochloric acid of 33 per cent. HCl, 9.5 kilos. of  $\alpha$ -naphthylamine hydrochloride, and 9 kilos. of glycerol are stirred into a paste and heated to 160°–165° C. The melt finally turns blue, gas is evolved and the colouring matter separates out in lustrous green laminae. The whole melt is then poured into about 150 litres of water, and the colouring matter which precipitates is purified by crystallisation from water in which the hydrochloride is sparingly soluble in the cold. It is tolerably soluble in cold alcohol with a blue colour, having a dark red fluorescence, and on the addition of an alkali gives a base soluble in alcohol and ether with a red colour and a yellowish brown fluorescence. The sulphate is readily soluble in hot water.—T. A. L.

*Colouring Matter of the Oxyketone Group, suitable for Dyeing and Printing with the Aid of Mordants.* J. Y. Johnson, London. From "The Badische Anilin und Soda Fabrik," Ludwigshafen, Germany. Eng. Pat. 9427, June 5, 1889. 4d.

THIS is an extension of Eng. Pat. 8373, of 1889 (this Journal, 1890, 497), and describes the preparation of tetra-hydroxy-phenyl-naphthyl ketone. About 100 parts by weight of gallic acid are dissolved in about 400 parts by weight of  $\alpha$ -naphthol at 230° C. and 4 parts by weight of zinc chloride are carefully added to the solution. After the reaction has ceased the mass is allowed to cool to about 100° C., when the excess of  $\alpha$ -naphthol is extracted with benzene. The dyestuff is then dissolved in alcohol and precipitated by pouring into water, when it separates in yellowish green scales. The substance does not melt below 200° C., and dyes cotton mordanted with alumina a greenish yellow, and wool mordanted with alumina a brownish yellow.—T. A. L.

*A New or Improved Manufacture of Beta-Naphthol Carbon Acid.* J. Y. Johnson, London. From F. von Heyden Nachfolger, Radebeul, Germany. Eng. Pat. 9612, June 11, 1889. 4d.

The production of a  $\beta$ -naphthol carboxylic acid has already been described in Eng. Pat. 6134 of 1885 (this Journal, 1885, 531), and Eng. Pat. 8155 of 1886 (this Journal, 1886, 482). The acid thus obtained melting at 157° C. is very unstable and decomposes easily into  $\beta$ -naphthol and carbonic acid. It has been found that by the action of carbonic acid under pressure on the alkaline salts of  $\beta$ -naphthol at 200°–250° C. a stable acid of a yellow colour is produced melting at 216° C.—T. A. L.

*Production of Oxy-sulphonic Acids of Naphthalene.* O. Imray, London. From "The Farbwerke vormals Meister, Lucius und Brüning," Höchst, Germany. Eng. Pat. 9642, June 11, 1889. 6d.

THE  $\beta$ -naphthol disulphonic acids R and Y (described in Eng. Pat. 1715 of 1878) and the  $\beta$ -naphtholtrisulphonic acid of Eng. Pat. 2544 of 1882, when heated with caustic alkalis, yield oxy-sulphonic acids of naphthalene, which form azo-dyestuffs when combined with diazo-compounds.

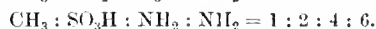
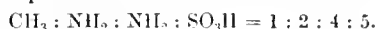
—T. A. L.

*Improvements in the Production of Colouring Matters from Oxy-sulphonates of Naphthalene.* O. Imray, London. From "The Farbwerke vormals Meister, Lucius und Brüning," Höchst, Germany. Eng. Pat. 9643, June 11, 1889. 4d.

VIOLET to bluish-black colouring matters for wool are produced by combining the dioxynaphthalene disulphonic acid described in Eng. Pat. 9642 of 1889 (see preceding abstract) with the diazotised amido azo-compounds obtained by combining diazotised amidosulphonic acids with  $\alpha$ -naphthylamine. In the example given, *o*-toluidine sulphonie acid is diazotised and combined with  $\alpha$ -naphthylamine. The product thus obtained is re-diazotised and combined with dioxynaphthalene disulphonic acid in presence of sodium acetate. Still bluer shades are obtained by substituting naphthylamine sulphonie acids for the *o*-toluidine sulphonie acid.—T. A. L.

*Improvements in the Production of Colouring Matters.* H. H. Lake, London. From K. Oehler, Offenbach-on-the-Maine, Germany. Eng. Pat. 11,000, July 9, 1889. 6d.

Two series of brown colouring matters are obtained (1) by combining the diazo-compounds of the three toluylene diamine sulphonie acids—



with *m*-phenylene diamine and *m*-toluylene diamine; and (2) by combining the so formed intermediate compounds with diazotised amidosulphonic acids. The following example gives the quantities employed: 15 kilos. of the sodium salt of toluylene diamine sulphonie acid—

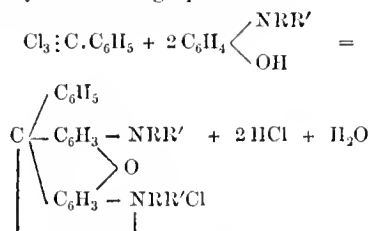


and 6.9 kilos. of sodium nitrite are dissolved in 500 litres of water, cooled to 0°, and 36 kilos. of hydrochloric acid of 22.5° B. are run in. This diazo solution is then added to 1,000 litres of water containing 21 kilos. of *m*-phenylene diamine sulphate and 25 kilos. of calcined soda. The precipitate forms a dark brown mass, which is filtered, pressed, and dried. It forms a reddish-brown powder, soluble in water with a light yellow-brown colour, and dyes cotton brown from an alkaline bath. As an example of the second series of colouring matters 100 kilos. of the compound obtained above are mixed while still damp with 184 kilos. of diazosulphanilic acid in a sodium acetate solution. The formation of the dyestuff proceeds slowly, and can be accelerated by gently warming. When the reaction is finished the solution is neutralised with soda, boiled, filtered, and precipitated with salt. The dyestuff

forms a blackish powder with a greenish hue. It dissolves in water with a brown colour, which is little affected by alkalis.—T. A. L.

*Manufacture of New Red and Blue Colouring Matters.* O. Imray, London. From "The Society of Chemical Industry in Basle," Switzerland. Eng. Pat. 11,613, July 20, 1889. 6d.

THESE colouring matters are produced by the action of benzotrichloride on the alkylated and phenylated derivatives of *m*-amido-phenol, the former giving reds to violets soluble in water, and the latter bluish-violets to blues, but insoluble in water and requiring to be sulphonated. The reaction is expressed by the following equation:—



where R, R' represent the radicles methyl, ethyl, amyl, benzyl, phenyl, tolyl or naphthyl. A red colouring matter is produced by heating a mixture of 10 kilos. of benzotrichloride, 13 kilos. of dimethyl-*m*-amidophenol and 50 kilos. of sand to 50°–60° C., and finally up to 100°–120° for some hours. The mass is distilled with steam to expel volatile products, and the residue is extracted with 50 kilos. of boiling water and 2 kilos. of concentrated hydrochloric acid. The base is separated by the addition of 20 kilos. of caustic soda lye of 38° B., and purified by redissolving in hydrochloric acid, from which it is precipitated by the addition of zinc chloride and salt, forming long green needles. The base is nearly insoluble in water but easily soluble in alcohol, ether, benzene and toluene. It dyes wool and silk crimson without a mordant, silk showing a yellowish-red fluorescence. Cotton requires a mordant, and is then dyed a rather bluer shade than wool.—T. A. L.

*Manufacture of Blue Colouring Matter.* O. Imray, London. From "The Society of Chemical Industry in Basle," Switzerland. Eng. Pat. 11,848. July 25, 1889. 6d.

TANNIN when heated with aromatic amines forms with them crystalline products (see Schiff, Ber. 15, 7591). These condensation products on heating with nitroso-dimethylaniline hydrochloride give blue colouring matters soluble in alcohol. When heated with sodium bisulphite and alcohol they yield light green needles soluble in hot water, and dye cotton mordanted with tannin, alum or chromium blue to bluish-violet. The condensation products before treatment with bisulphite may be sulphonated, and can then be employed for dyeing blue shades on wool.

—T. A. L.

*Improvements in the Manufacture of Colouring Matters.* H. H. Leigh, London. From R. G. Williams, Albany, U.S.A. Eng. Pat. 3095, February 26, 1890. 6d.

AN extension of Eng. Pat. 12,767 of 1889 (this Journal, 1889, 981; see also this Journal, 1890, 173), in which cotton colours are produced by combining naphthalene-azobiphenylamines and their sulphonie acids with tetrazo bodies, to form intermediate compounds, which can be combined with phenols and amines. In the present invention it is proposed to substitute for the naphthalene-azobiphenylamines the bodies obtained by combining one molecule of diazobenzene, its homologues, or sulphonie acids with one molecule of  $\alpha$ - or  $\beta$ -naphthylamine or its sulphonie acids. The following is one of the examples given: 17.3 lb. of sulphianilic acid are diazotised with 7 lb. of sodium nitrite and 12 lb. of hydrochloric acid of 22° B. The diazobenzene sulphonie acid thus produced is combined with 25 lb. of sodium naphthionate in presence of sufficient

sodium acetate to neutralise the mineral acid present. After some time a brownish-red wool dye is formed, which is combined with tetrazoditolyl from 32 lb. of tolidine sulphate and 14 lb. of sodium nitrite. When all the tetrazo compound has combined, 25 lb. of sodium naphthionate are added. The formation of the colour proceeds slowly, and the whole should be stirred for several days. When dye-tests show no improvement, the temperature is raised to the boil, and sufficient soda lye added to form the sodium salt, which is then precipitated from the liquor by the addition of salt, filtered, pressed, and dried. The colouring matter dyes unmordanted cotton bright red from an alkaline or soap bath, giving a shade similar to that produced when diazo- $\alpha$ -naphthalene sulphonie acid is substituted for the diazo-benzene sulphonie acid mentioned above. Regarding this similarity of shade by substituting benzene for naphthalene, see Eng. Pat. 10,653 of 1889 (this Journal, 1889, 538), and Eng. Pat. 16,493 of 1887 (this Journal, 1888, 619).—T. A. L.

## V.—TEXTILES: COTTON, WOOL, SILK, Etc.

### PATENT.

*Improvements in the Method of and in Apparatus for Washing or Cleansing Wool and other Textile Fibres, also applicable to the Washing of Clothes.* W. Eastwood, Leeds, and A. Ambler, Bradford. Eng. Pat. 15,153, September 26, 1889. 1s. 1d.

THE wool or other textile fibre which is to be washed is passed through feeding rollers into an open trough, which is preferably set at a slight inclination. A second smaller trough, or a number of such, is fitted into the open one, and by means of an eccentric or other mechanical arrangement, a reciprocating motion is imparted to the same in such a way that the material being washed receives a forward impulse down the open trough towards the squeezing rollers. The smaller troughs are preferably provided with lids, and are perforated at the bottom and sides. Below the washing trough or "sud bowl" there is placed a tank for the reception of the liquor, which, after allowing the sand, &c. to deposit, is pumped again into the trough. It is stated that by the use of this machine the fibres "are not matted or clogged together, as is the case more or less with all fork machines."

The original specification is illustrated with five sheets of drawings.—E. B.

## VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

*The Action of Chlorine on Hematoxylin and the Extractive Matter of Logwood.* W. W. Macfarlane and P. S. Clarkson. J. Franklin Inst. 1890, 129, 247–255.

TO increase the dyeing power of logwood liquors and extracts, oxidising agents such as bleaching powder, chlorates, &c. are often added, their action being supposed to consist in the conversion of the hematoxylin into hematein. The authors using the proportions given by Avery (Eng. Pat. 7630 of 1885; this Journal 1885, 585), viz.,  $\text{C}_{16}\text{H}_{11}\text{O}_6 + \text{O} = \text{H}_2\text{O} + \text{C}_{16}\text{H}_{12}\text{O}_6$ , could obtain no satisfactory result; with bleaching powder the result was invariably a dull grey shade of black.

The dyeing power of logwood liquors is however gradually increased to a maximum by exposure to free chlorine, the efficiency of a "42° extract" of dry cut wood was increased 150 per cent. by the action of 9 per cent. of its weight of

chlorine, but any further increase in the quantity of chlorine produced a dull grey shade. The extract obtained from eured wood is similarly affected. If the temperature of the solution be maintained at about 80° C. during the absorption of the chlorine, a smaller quantity of the latter will produce the maximum effect. When a solution of bleaching powder is used the addition of *half* the theoretical quantity produces a marked improvement in the colour obtained. Worst dyed black on chromium mordant with the chlorinised extract and exposed to the light for six weeks does not undergo more change in shade than a sample dyed with ordinary eured wood.

The dyeing power of hæmatoxylin is likewise increased by exposure to chlorine, the greatest development being produced when four atoms of chlorine are supplied for each molecule of hæmatoxylin; this is equivalent to 47 per cent. of the latter. When bleaching powder is employed, the same effect is produced when the proportion of available chlorine to hæmatoxylin is only 23.5 per cent.

Erdmann (J. prakt. Chem. 26, 202), Reim. (Ber. 4, 329), and Dralle (Ber. 17, 372; this Journal, 1884, 242) were unable to isolate hæmatein from the product of the action of chlorine on hæmatoxylin.

By dissolving the hæmatoxylin in hot water, allowing to cool, treating with chlorine water containing the calculated amount of chlorine, and repeatedly washing with ether containing a small quantity of alcohol, the authors obtained on evaporation and drying a brownish resinous mass completely soluble in alcohol. The residue on treatment with chloroform yielded a small quantity of a white crystalline substance soluble also in ether, water and acetic acid. By treating the residue with ether two bodies were separated, one having a brownish, resinous appearance, the other a bright greenish metallic lustre. The former appears to be a chloro-derivative of hæmatein or hæmatoxylin, while the latter possesses physical and chemical properties identical with hæmatein.

Hæmatein is also found in the extract from eured wood, and appears to exist in about the same proportion as in the chlorinised hæmatoxylin, the colours obtained in comparative dye tests being similar in depth and shade. Hæmatein has only a special value in wool dyeing where the bath is neutral or slightly acid; in this case the shade given by hæmatein is at least twice as full as that obtained from hæmatoxylin. In "speck dyeing," however, where the solution is strongly alkaline, the reverse is the case, hæmatein being of little practical value. From this it appears that when alkaline solutions are used in the application of logwood, the extract or decoction should be made of dry eured wood, whilst for wool dyeing much better results follow from the use of eured wood.—S. B. A. A.

*Note on some Hypochlorite Solutions used for Bleaching.*  
Osterberger and Capelle. Bull. Soc. Ind. Ronen, 1889, 375—378.

UNTIL the year 1876, bleaching soda, or "can de Javel," was made by passing chlorine gas into a dilute solution of sodium hydrate or carbonate. Plancau, about this period, succeeded in making a more concentrated solution by adding dry bleaching-powder to caustic soda liquor of 19° B. Since then there has appeared in the market a number of preparations stated to possess remarkable properties. One of the first of them was a liquor which attracted considerable attention under the name of "chlorozone." Its discoverer, Count Brochocki, manufactured it by forcing with an air-blast chlorine, liberated from bleaching-powder by the action of dilute sulphuric acid, into two vessels filled with dilute caustic soda; as the caustic soda in the first vessel became neutralised, the hypochlorite, which had been first formed, was decomposed, hypochlorous acid being liberated; the action in the second vessel was only allowed to proceed as far as the formation of hypochlorite. The contents of the two vessels were mixed forming "chlorozone." According to Brochocki, chlorozone contains oxygenated compounds of chlorine, such as chloric acid and chlorine peroxide. On the other hand, Lunge and Landolt, who have examined the product, find it to be merely a dilute solution of hypochlorous acid in bleaching-soda liquor.

Owing to the commercial success of the original chlorozone, numerous manufacturers have been led to make imitations. Some of them have not scrupled to sell, under the names of chlorozone and chlorogene, ordinary bleaching-soda, which they guaranteed to be free from caustic, but which is of a composition such as is shown by the following example:—

	Grms. per Litre.
Active chlorine .....	115.68
Inactive chlorine .....	29.96
Sodium carbonate .....	17.31
Sodium hydrate .....	19.58

The density of this particular sample was 24° B.—E. B.

*Nigrisine.* T. Baumann. Bull. Soc. Ind. Mulhouse, 1890, 65—68.

NIGRISINE is the commercial name of a basic grey dyestuff recently discovered by E. Ehrmann. It comes into commerce in the form of a black powder, soluble in water and acetic and hydrochloric acids. 1 litre of water at 100° dissolves 80 grms. of the dyestuff; it is rather more soluble in acetic acid of 6° B., but dissolves most easily in a mixture of equal volumes of such acid and water; it is almost insoluble in 90 per cent. alcohol, and but slightly soluble in ethyl-tartaric acid and acetic. Its chief reactions are as follow:—In water it dissolves with a reddish-grey colour, which is changed to a blue-grey by the addition of acid; concentrated sulphuric acid dissolves it with a pure grey colour, which on the gradual addition of water, changes successively to red-grey and blue-grey; nitric acid added to an aqueous solution oxidises the dye, producing a red-brown solution; stannous chloride and hydrochloric acid reduce it, a yellow solution being obtained, which when diluted with water, deposits a brown precipitate, the solution remaining pale yellow; zinc dust and acid reduce the dye, also forming a yellow solution, which, on exposure to the air or by oxidation with ferric chloride, recovers its original tint; potassium bichromate produces a brown precipitate, soluble in acetic or tartaric acid with a grey-blue, in hydrochloric acid with a red-blue colour; a characteristic test consists in adding sodium hydrate to a solution of the dye, and extracting the precipitated base with ether or light petroleum, which are coloured cherry-red. On separating the coloured layer and shaking it up with acetic acid, the latter becomes coloured greenish-blue.

Nigrisine is distinguished by its purity of shade, strong colouring power, fastness, and the variety of compound shades, which, in admixture with other dyestuffs, it is capable of yielding.

The following examples of colour-mixtures will serve to show its mode of application in calico-printing:—

	Dark Grey.	Medium Grey.
	Parts,	Parts,
Nigrisine .....	40	10
Acetic acid of 6° B. ....	200	200
Water .....	200	250
Gum tragacanth paste .....	500	500
Tannic acid .....	80	20
Tartaric acid .....	15	15

Increase of the proportion of tartaric acid renders the shade bluer and more regular, but endangers the strength of the cotton. A mixture made without tannic acid, printed and steamed, yields a rather reddish-grey, which is as fast as when fixed with tannic acid, and its fastness may even be slightly increased by chroming after steaming. Such a mixture, in which the tannic acid is omitted, serves equally well for cotton, wool, and silk, or mixed goods.

Nigrisine does not require a mordant for dyeing bleached cotton. Full shades are obtained with 1 to 3 per cent. of the dye. Cotton mordanted with antimony tannate takes the dye well. A method of dyeing, suitable in this case,



consists in padding the pieces in a solution containing from 2 to 10 grms. of the dye per litre, drying, and steaming, or the pieces may be passed, without drying, through a 0.5 per cent. solution of potassium bichromate at a temperature of about 60°, when an insoluble chromate of the colour-base is formed.

The many advantages of this dye, combined with its low price, should, in the opinion of the author, secure its employment in dyeing and calico-printing.—E. B.

*A Mishap in Cotton Bleaching.* A. Scheurer. Bull. Soc. Ind. Mulhouse, 1820, 78—90.

THE author enumerates some of the causes of bad work experienced on starting a new bleachworks, and then recounts his own experiments made some time ago for the purpose of discovering the cause of a troublesome defect, which frequently appeared in the printed cloth in the works with which he is connected. After the prints had been steamed there were frequently noticeable broad stripes, of 60 to 80 cm. width, extending across the cloth, and occurring regularly at intervals of 7 metres. On these stripes the colours were defective. There was, however, a central portion of 8 to 10 cm. in the stripe where the colours were normal. The defect manifested itself in the following way: the extract reds and pinks bled, forming "auroles"; the extract purple was of a reddish hue. Attention was at first directed to the hanging rails in the steaming machine, the back-greys, and the preparation with oleine, but no fault could be found with any of these. Eventually the cause was traced to the presence of aluminium chloride in the water of the bleach-house washing machine, the distance of 7 metres mentioned above corresponding to the length of cloth lying between two successive "nips" in the same.

The aluminium chloride was formed by the sours draining from a heap of cloth on a stillage, escaping through the sides of an asphalt gutter, through the soil, and thence into a canal from which the wash-water was taken. The mortar in the bricked wall of the canal was dissolved away and aluminium chloride formed. It was found that the defect was produced in the cloth contained in the washing machine during the dinner hour, when the machine was stopped, the cloth out of the water becoming drier, and the mordant fixing on the exposed parts, whilst the portion pressed between the rollers remained moist, and, consequently, unmordanted. It was further found that cotton had the power of attracting and fixing alumina even from a very acid solution of aluminium chloride, and that the alumina contained in rosin and soda can give rise to a light mordanting.—E. B.

#### PATENTS.

*Improvements in Machines for Sizing and Painting Cloth or Fabrics.* A. J. Boulton, London. From D. A. Page, R. C. and M. Bird, Chicago, U.S.A. Eng. Pat. 7185, April 30, 1889. 1s. 1d.

THE improvements claimed relate to mechanism for sizing and painting fabrics, such as window curtains.

The fabric is taken from a roll at one end of the machine, sized, according to its quality, on one or both sides, dried, painted upon both sides, again dried, and finally rolled up at the other end of the machine, all the operations being continuous. Drawings are given in the original specification to illustrate the details of construction and mode of working.—E. B.

*Process for Cleansing and Bleaching Fibrous Substances, and for fixing Organic Colouring Matters thereon by Means of Acid and Alkaline Residues of Naphtha Manufacture or of Specially Prepared Substances analogous thereto.* V. Sehevelin, Balakhna, and P. Mindovsky, Moscow, Russia. Eng. Pat. 8107, May 15, 1889. 8d.

SULPHO castor oil-acid, sulphopyroterebene-acid, sulpho-oleine-acid, and the "soda and acid residues" obtained in the washing of mineral and coal-tar oils, when neutralised

with alkalis, effect the cleansing of textile fibres more energetically than does soap. The fats contained in greasy wool are readily dissolved by the "residue" solution, a more complete removal of the grease being so obtained than with the usual methods of washing. Treatment of the alkaline solutions with sulphur dioxide to acid reaction causes their bleaching action to be increased. Further, it is claimed, fabrics composed of vegetable fibres, after being boiled in the solutions of the substances above mentioned, can be dyed with organic colouring matters, without further mordanting. (See also this Journal 1890, 488.)—E. B.

*Improvements in Apparatus for Dyeing and otherwise Treating Wool.* S. Mason, jun., Manchester. Eng. Pat. 9149, June 3, 1889. 8d.

THIS is an arrangement for dyeing or otherwise treating wool in the form of "tops" in a keir or closed vessel. The apparatus cannot be properly described apart from the drawings given with the original specification.—E. B.

*Improvements in Solutions for Removing Rust Stains from Cloth.* A. Buecher, Heidelberg, Germany. Eng. Pat. 9600, June 11, 1889. 4d.

RUST stains in cloth are removed by treatment with the following mixture:—15 grms. of oxalic acid, 20 grms. of chloride of tin, 1.5 litres of water, and 50 cc. of a 0.5 per cent. solution of benzoic acid.—E. B.

*Improvements in the Method of and Apparatus for Dyeing and similarly Treating Yarns and Textile Materials in all Stages of Preparation.* W. E. Heys, Manchester. From C. Vandermeirsche, Paris, France. Eng. Pat. 9836, June 15, 1889. 8d.

THIS invention relates to a mechanical arrangement (described in detail with the aid of drawings in the original specification) for the purpose of carbonising, washing, scouring, mordanting, and dyeing woollen or other textile material. The material to be treated is placed in pots, which have their upper and lower ends perforated and removable, and which are caused intermittently to rotate in a bath of the liquor corresponding to a particular operation, a stream of the same liquor being injected through the material in the pots during the periods of rest.—E. B.

*An Improved Copying Ink.* W. S. Conrad and H. T. Lilley, Southsea. Eng. Pat. 2011, February 7, 1890. 4d.

By this ink a clear copy of a written document can be rapidly obtained "by simply blotting the writing with any ordinary copying paper," both original and copy being left dry. The ingredients used are a solution of gall nuts in water, sulphate of iron, aniline blue black, chloride of magnesium, and "glycerin-indigo" (prepared by adding one-third of an ounce of indigo-carmin to each pound of glycerol).—E. G. C.

*Improvements in or relating to the Dyeing of Yarn.* A. J. Boulton, London. From A. and M. Koblenzer, Augsburg, Germany. Eng. Pat. 4683, March 25, 1890. 4d.

IN dyeing cotton yarn in the cop, a thorough penetration of the dye is effected by the employment of a mordant such as primuline, applied under a pressure of 2 to 3 atmospheres, the cops being afterwards rinsed in ice-cold water, then treated with an acidulated solution of sodium nitrite, again rinsed, and dyed with "naphthol or naphthylamine" under a pressure of 2 to 3 atmospheres.—E. B.



## VII.—ACIDS, ALKALIS, AND SALTS.

## On the Substitution of Salts in Complex Solutions.

A. Etard. *Compt. Rend.* **110**, 186—188.

I. The author has previously investigated, by a special method, the complete curves of solubility for a mixture of potassium and sodium chloride made under the special conditions enumerated below (III.) (this Journal, 1890, 290), and has thus determined the variation with temperature in the proportion of two metals present, which share the same metalloïd. He now investigates the problem when the metal of the two dissolved salts is the same and the metalloïd different.

II. The solubility  $S$  (defined as the percentage of salt in a saturated solution) of potassium iodide may be represented as a function of the temperature,  $t$ , by three straight lines, extending the first from  $-22^\circ$  to  $0^\circ$  (insufficiently determined to be represented by a formula), the second from  $0^\circ$  to  $108^\circ$  (angular coefficient =  $0.1257 t$ ), the third (No. 1 on diagram), from  $108^\circ$  to  $220^\circ$  (angular coefficient =  $0.0603$ ). If this last segment be produced by extrapolation, its "limiting point" is found to occur at  $637^\circ$ ; the melting point of potassium iodide being 639, according to Carnelley. (The "limiting point" has been defined in a previous paper as the theoretical temperature at which, supposing that its strength continue to increase at a constant rate with rise of temperature the solution would contain the limiting amount of salt, i.e., 100 per cent.)

From the examination of a large number of complete curves of solubility it appears that they are composed of segments of straight lines, the angles which these lines make with the temperature axis successively decreasing with increase of temperature. Most perturbations take place below  $130^\circ$ , a temperature at which in all likelihood hydrates no longer exist. And it seems probable that the lines least inclined to the temperature axis, which have been determined

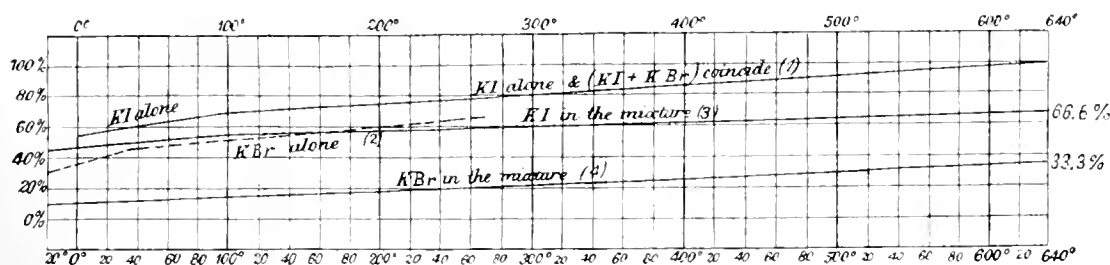
in many cases experimentally as far as  $260^\circ$ , represent the phenomenon of solution much beyond this temperature. Chemical decompositions, the occurrence of the critical point of solvents, &c., however, would prevent the lines representing the facts as far as the "limiting point." But they bear a real relation to this "limiting point," which, in many cases, coincides with the melting point of the salt, and hence possesses a precise physical signification.

III. If a small quantity of water be added to an excess of potassium iodide and potassium bromide, the two salts being mixed in any proportion, it is found:—

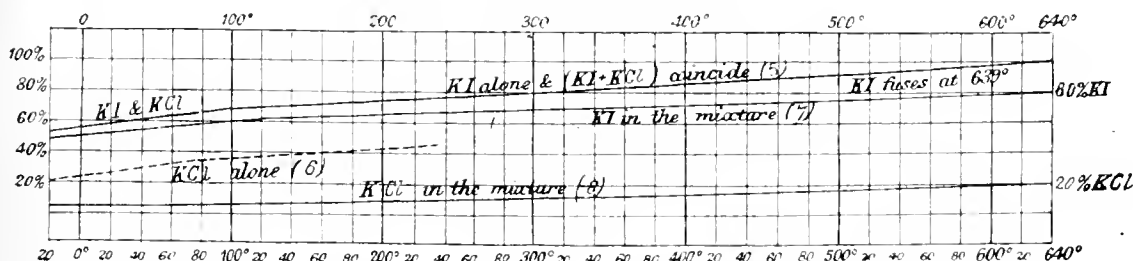
- (1.) That the total weight of salt dissolved is the same at all temperatures as if potassium iodide alone had been used; the line (1) represents solubility both of potassium iodide alone and of the mixture of potassium iodide and potassium bromide. Hence the "limiting point" for  $KI + KBr$  solution is 639, the melting point of  $KI$ .
- (2.) From the analysis of the solutions it follows that in the mixture the potassium bromide follows the law of solubility of potassium iodide and not its own. It is as if bromine were substituted for iodide.
- (3.) It may be calculated by extrapolation that at the limiting point the salt would consist approximately of two-thirds (66.4 per cent.) of potassium iodide and one-third (33.6 per cent.) of potassium bromide; that is to say, if the experimental lines (3) and (4) be considered separately, and produced to their limiting points, the ordinates at these points correspond to the figures given above, which, as will be seen, add up to 100.

IV. The line for a mixture of potassium iodide and potassium chloride (5) coincides in the same way with the line for potassium iodide. At the limiting temperature the salt would consist of four-fifths of iodide and one-fifth of chloride (see curves (7) and (8)). The couple, potassium bromide and potassium chloride, gave similar results, the

SOLUBILITY OF A MIXTURE OF (KI + KBr).



SOLUBILITY OF MIXTURES OF (KI + KCl).



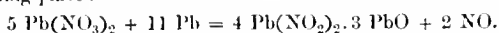
limiting composition being calculated to be three-quarters bromide and a quarter chloride. The research is in progress. —P. J. H.

The Preparation of Alkaline Nitrites. G. A. Le Roy. *Bull. Soc. Ind. Rouen*, 1889, 363—370.

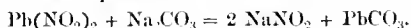
THE author enumerates the various processes known for the preparation of nitrites.

1. *Decomposition of Nitrates.*—This is the oldest method, and consists in heating a nitrate till a portion of its oxygen has been evolved as in the equation:  $\text{NaNO}_3 = \text{NaNO}_2 + \text{O}$ . If the heating be continued complete decomposition ensues, more oxygen, together with nitrous gases, being evolved. This method is not employed for manufacturing purposes on account of the difficulty of controlling the reaction and the poor yield obtained.

2. *From Lead Nitrate*.—Basic lead nitrite is first prepared by boiling a solution of one part of lead nitrate with one or two parts of lead in thin sheets, the following reaction taking place:—



The excess of lead oxide is precipitated by a current of carbon dioxide, and the solution of normal lead nitrite so obtained is decomposed with an alkaline carbonate—

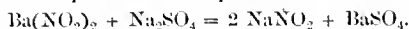


3. *Method with Nitrogen Dioxide*.—Gay-Lussac stated that pure alkaline nitrite was formed by absorbing a mixture of four volumes of nitrogen dioxide and one of oxygen in a concentrated solution of alkali. Berthelot has since shown that when the nitrous acid formed is absorbed as it is produced, the relative proportions of the two gases are immaterial, but that, otherwise, nitrogen tetroxide is produced, and then, on absorption, a mixture of nitrate and nitrite is obtained, thus—



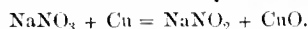
4. *Method with Nitrous Acid*.—Nitrous acid, obtained by acting on arsenious oxide, starch, &c., with nitric acid, is passed into a solution of alkali. To obtain the gas nearly free from nitrogen tetroxide, Lunge recommends the employment of one part of arsenious oxide or starch to every 10 parts of nitric acid of sp. gr. 1.30 to 1.35.

5. *Method with Barium Dioxide*.—This method, due to H. Debray, consists in passing nitrous oxide over gently-heated barium dioxide, the gas being absorbed with the formation of barium nitrite:  $2 \text{ NO} + \text{BaO}_2 = \text{Ba(NO}_2)_2$ . The product is treated with water, filtered, any barium hydrate present removed by carbon dioxide, and the solution decomposed with a sulphate—



The author of this process incidentally remarks that lead dioxide cannot be used instead of barium dioxide in this process.

6. *Method with Copper*.—Persoz has proposed to partially deoxidise alkaline nitrates with finely-divided copper—



The copper for the purpose is obtained by igniting cupric acetate in closed vessels. Metallic copper may be regenerated from the oxide by a current of hydrogen. Müller and Pauly obtain the copper in the necessary fine state of division by reducing a solution of copper sulphate with zinc.

7. *Method with Lead*.—Hampe or, according to H. Rose and Fresenius, Stromeyer has the credit of originating this process. It consists in fusing a mixture of a nitrate and metallic lead, the following equation representing the reaction which takes place:  $\text{NaNO}_3 + \text{Pb} = \text{NaNO}_2 + \text{PbO}$ . The mass is dissolved out with water and treated with carbon dioxide and hydrogen sulphide or ammonium sulphide to eliminate lead; the solution is then filtered, evaporated to dryness, the residue heated to destroy hyposulphites, again dissolved in water, and crystallised.

8. *Method with Litharge*.—Burton, in his process, commences with lead oxide, and makes use of the deoxidising powers of the same in becoming converted into lead tetroxide:  $\text{NaNO}_3 + 3 \text{ PbO} = \text{NaNO}_2 + \text{Pb}_3\text{O}_4$ .

A mixture, in proper proportions, of lead nitrate, sodium carbonate and litharge, is heated, and the sodium sulphate and nitrite formed are dissolved in water and separated by crystallisation.

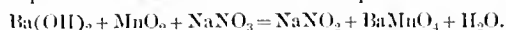
9. *Method with Carbon*.—This method, known since 1828, is of no industrial value, as it is impossible to avoid the simultaneous production of alkaline carbonate. Finely powdered charcoal, starch, or, best of all, graphite, is heated with the nitrate  $\text{NaNO}_3 + \text{C} = \text{NaNO}_2 + \text{CO}$ .

10. *Method with Alkaline Sulphites*.—Etard suggested the heating together of molecular equivalents of a nitrate and sulphite of the same base, sulphate and nitrate being formed thus:  $\text{NaNO}_3 + \text{Na}_2\text{SO}_3 = \text{NaNO}_2 + \text{Na}_2\text{SO}_4$ .

The two salts are separated by crystallisation.

11. *Method with Zinc*.—Stahl-schmidt has described a wet method, in which, in an ammoniacal solution, the nitrate is reduced to nitrite by zinc dust, the temperature being kept below 50°. When the reaction is complete, the solution is boiled to expel ammonia, treated with carbon dioxide to precipitate zinc, nitric acid added to decompose the alkaline carbonate present, and the nitrite crystallised out.

12. *Method with Manganese and Chromium Oxides*.—C. Huggenberg, in 1887, patented the following method: manganese oxide, chromium oxide, or arsenious oxide, is fused with a nitrate and an alkali or alkaline earth; an example of the reaction is shown in the equation—



13. *Miscellaneous Methods*.—Such are the reduction of the fused nitrate by a current of hydrogen, the saponification of alkyl nitrates, and the reduction of alkaline nitrates by microbes, a curious fact discovered by Gayon and Dupetit.

14. *Method with Barium Sulphide*.—This method has been proposed by the author. It has the advantage of yielding a nitrite free from impurities requiring special modes of separation. The alkaline nitrate is heated with barium sulphide in the proportions required in the equation:  $4 \text{ NaNO}_3 + \text{BaS} = 4 \text{ NaNO}_2 + \text{BaSO}_4$ .

It is necessary to employ barium sulphide free from carbon. When the mineral contains barium sulphate it is rather an advantage than otherwise, as it serves to moderate the reaction. The barium sulphate produced can be easily reconverted into sulphide (this Journal, 1889, 933).

—E. B.

#### *Manufacture of Ammonium Chloride from By-Products in Gas Manufacture and Metallic Chlorides.* Dubose and Heuzey. Bull. Soc. Ind. Rouen, 1889, 438—442.

IN the manufacture of gas from coal, ammoniacal liquors are produced which yield three commercial products.

1st, the crude liquor; density 4° to 5° B. containing about 4 per cent. of  $\text{NH}_3$ .

2nd, the concentrated liquor; density 10° to 12° B., containing 12—14 per cent. of  $\text{NH}_3$ .

3rd, crystallised ammonium sulphate containing 20 per cent. to 21 per cent. of nitrogen, corresponding to 25 per cent. of  $\text{NH}_3$ .

These three products may be treated with metallic chlorides so as to yield ammonium chloride, the process varying with the product treated.

(1.) *Treatment of the Crude and Concentrated Ammoniacal Liquors*.—These liquors contain besides ammonium sulphate, free ammonia, ammonium chloride and cyanide, a considerable amount of ammonium carbonate and sulphide which precludes the possibility of conversion into chloride by the direct action of hydrochloric acid, which would give rise to large volumes of carbon dioxide and sulphuretted hydrogen. The authors base their new process on the precipitation of the sulphur and carbonic acid by double decomposition with a mixture of the chlorides of iron and calcium in proper proportions; neither the sulphides nor carbonates of these metals being soluble in presence of ammonium chloride. The originality of the method consists in using the mixture of iron and calcium chlorides; it has the advantage of (1) complete desulphurisation, which is not possible with the use of calcium chloride alone, owing to the slowness with which calcium sulphide is formed; and (2) rapid precipitation of the carbonates as  $\text{CaCO}_3$ , which does not take place in the presence of iron alone, as the colloidal carbonate of iron is then formed.

The process is carried out as follows:—The gas liquors are freed from tar by being left for 48 hours in cisterns, and are then pumped up into a tank, into the bottom of which the necessary quantity of the mixed chlorides has been already introduced. The mixture is stirred mechanically and left to settle for 12 hours. The upper two-thirds of the mother-liquor are then perfectly clear, and absolutely free from sulphide and carbonate. After being slightly acidified, if necessary, this portion is conveyed directly by pipes to the concentrating pans. The precipitate, consisting

of the mixed carbonates and sulphides of iron and calcium, is filtered on horizontal filters or, preferably, in the filter-press. The dried residue forms an excellent purifying mixture for coal-gas, analogous to Laming's mixture.

The density of the liquors thus obtained is generally  $7^{\circ}$  B., and they must be evaporated down to  $13^{\circ}$  B. For this purpose the authors use flat iron pans made of strong sheet or cast iron, containing 2,000 litres. A close grained cast-iron wears best. The fire and pans are covered in entirely by a hood of silicated and stuccoed wood. All the joints are made tight so that the vapours may be entirely carried off by a large flue into the main chimney shaft, where any sulphuretted hydrogen, due to possible imperfect desulphurisation, is burnt by a special arrangement. When the density reaches  $13^{\circ}$  B. the liquid is drawn off and left to crystallise in wooden troughs. It takes 15 days to crystallise completely, the crust being broken each day. When acicular crystals of the ammonium chloride are to be produced, the operation is carried out exactly as above. If cubes be desired, 5 per cent. of a solution of perchloride of iron (density  $35^{\circ}$  B.) must be added to the mother-liquors; this seems to act merely "by presence" and remains in the mother-liquors. For certain chlorides used in galvanising, manganese salts or zinc salts, or fatty matters are added. When the crystallisation is complete the residual liquid is drawn off and pumped back into the evaporating pans, and the crystals are taken out, drained, and then either sent to a drying chamber whose temperature is  $50^{\circ}$ — $60^{\circ}$ , or fritted in a reverberatory oven. Slaked lime is used to give the brownish-yellow colour required in commerce, and the product is then packed in barrels.

To produce the refined salt, the solution is only concentrated to  $11^{\circ}$  B., then pumped into wooden troughs, and the metals it contains precipitated with ammonium sulphide. The liquid is then concentrated in vessels made of enamelled cast iron, and crystallised in earthenware pans; the crystals are drained and dried in the drying chamber. As the enamel frequently splits off the cast-iron pans, it must be replaced by a cement composed of dried calcium carbonate and potassium silicate, made insoluble by the action of hydrofluosilicic acid.

(2.) *Treatment of Ammonium Sulphate by Metallic Chlorides.*—The treatment varies with the nature of the chloride added.

1st. When the metallic chloride used yields an insoluble sulphate (e.g. Ca) on addition to the solution of ammonium sulphate:—

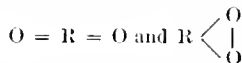
The ammonium sulphate is dissolved in the smallest quantity of water possible; it is then pumped up into a trough placed at a certain height, into which the necessary amount of chloride has been previously introduced. The mixture is stirred constantly, and when the reaction is complete, it is sent to a special filtering apparatus, or to a filter-press in which thorough washing is possible. The filtered liquid is then concentrated in the manner previously described. The residue of calcium sulphate may be used for agricultural purposes.

2nd. When the metallic chloride ( $\text{NaCl}$ ,  $\text{FeCl}_2$ ,  $\text{ZnCl}_2$ ,  $\text{MnCl}_2$ ) yields a soluble sulphate:—

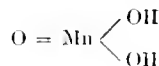
Any of the chlorides mentioned might be used, but the one actually employed is ferrous chloride. A solution of the crude salt (such as is produced in cleaning iron surfaces) of a density  $24^{\circ}$  B. and containing 25 per cent. of substance, is neutralised and pumped into cauldrons, where it is heated to over  $60^{\circ}$  C., and then the necessary amount of ammonium sulphate is added. Double decomposition ensues, and as soon as the surface begins to froth the mother-liquor is sent to the crystallising vessels where 95 per cent. of the iron sulphate crystallises out. The iron crystals are washed and the wash-water added to the ammonium chloride mother-liquors. To obtain the crude ammonium chloride these liquors are simply evaporated in flat pans; to obtain a refined salt, ammonium sulphide and barium chloride are added, and after filtration the liquors are concentrated in enamelled vessels as before.—P. J. H.

*On the Constitution of Manganese Peroxide.* W. Spring and M. Lucien. *Bull. Soc. Chim.* 1890, **3**, 4—8.

THE authors begin their paper by enumerating the theories which have been proposed to account for the behaviour of the various peroxides. Traube (*Ber.* **21**, 821) considers them as containing oxygen in the molecular state,  $\text{O} = \text{O}$ . Richarz has opposed these views (*Ber.* **21**, 1675), and returns to the old theory of Schönbein on ozonides and antozonides, for which the formulae would be—



The formula for manganese dioxide would, according to Richarz, be  $\text{O} = \text{Mn} = \text{O}$ ; and would thus agree with Frankes' proposal (*J. prakt. Chem.* **36**, 451) to denote by a formula—



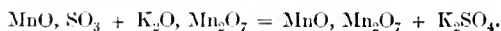
the hydrated dioxide formed by the action of water on the sulphate  $\text{O} = \text{MnSO}_4$ , which is itself produced in the decomposition of  $\text{Mn}_2(\text{SO}_4)_3$  (Fremy, *Compt. Rend.* **82**, 475).

It has, however, often been proposed to regard  $\text{MnO}_2$ , not as a peroxide, but rather as a manganate or permanganate of manganese either  $\text{MnO}$ ,  $\text{MnO}_3 (= 2 \text{ MnO}_2)$ , or  $3 \text{ MnO}$ ,  $\text{Mn}_2\text{O}_7 (= 5 \text{ MnO}_2)$ .

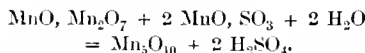
(See Guyard, *Bull. Soc. Chim.* [2], **1**, 88; and Volhard, *Annalen*, **198**, 318.)

Objections have to these views been raised by O. T. Christensen (this *Journal*, 1883, 346), but the authors think that the latter formula accounts best for the formation of manganese peroxide by the action of potassium permanganate on manganous salts,—

1.



2.



The saline and sometimes basic properties of manganese peroxide are better expressed by the formula  $3 \text{ MnO}, \text{Mn}_2\text{O}_7$ , which also brings out the relation of this body to the more basic compound  $4 \text{ MnO}, \text{Mn}_2\text{O}_7$ . (See Gorgeu, *Ann. Chim. et phys.* [3], **66**, 160; and Weldon, *Chem. News* **20**, 109 (1869), and *Monit. Scient.* 1874, **4**, 891).

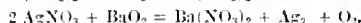
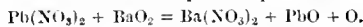
In order to elucidate the question, the authors have made the following experiments:—

1st. Barium dioxide was ground with crystallised manganous chloride in slight excess. A reaction immediately set in and oxygen was evolved. A black mass was produced which after being freed from the barium chloride produced and the excess of manganous chloride, proved to be  $\text{Mn}_2\text{O}_7$ . Hence it follows that manganese dioxide has a constitution unlike that of  $\text{BaO}_2$ .\*

2nd. Pure barium manganate was treated with manganous sulphate. Double decomposition takes place, but only slowly; after heating for a week on the water-bath, 10 grms. of a substance were obtained, which corresponded to the formula  $\text{Mn}_2\text{O}_7$ , and which may in this case be represented as manganous manganate,  $\text{MnO}, \text{MnO}_3$ . The colour of the body obtained differs slightly from that of ordinary manganous dioxide, but no sufficient evidence was obtained to show that it is really an isomeric substance.

3rd. Sodium manganate was treated with manganous carbonate, but double decomposition did not take place in the same way as in the preceding experiment. The reaction only sets in at a high temperature, and  $\text{Mn}_2\text{O}_7$  is obtained instead of  $\text{Mn}_2\text{O}_3$ . Probably  $\text{Mn}_2\text{O}_3$  is first formed, and this reacts with  $\text{MnO}$  formed by the decomposition of  $\text{MnCO}_3$  by heat ( $\text{MnO}_2 + 2 \text{ MnO} = \text{Mn}_2\text{O}_7$ ).

\* Lead nitrate and silver nitrate react on barium dioxide in the dry state, but in neither case is a peroxide produced:—



The authors are led to this explanation from the fact that no oxygen was evolved during the reaction.

4th. Manganese peroxide was heated with soda in an atmosphere of nitrogen, in order to verify the hypothesis that it is a salt. After washing and drying the products of the reaction, also in an atmosphere of nitrogen, a residue consisting of  $Mn_2O_3$  was found. The authors think this proves that  $MnO$  was formed at some stage of the reaction, and  $Mn_2O_3$  then produced according to the equation  $MnO + MnO_2 = Mn_2O_3$ .

Fremy obtained a residue of  $MnO_2$  from this operation: but he performed it in air, not in nitrogen.

5th. Pure  $Mn_2O_3$  was heated with dilute nitric acid, and it was found that manganese dioxide is formed, not by reduction of the nitric acid, but simultaneously with manganous nitrate,  $Mn_2O_3 + 2 HNO_3 = MnO_2 + Mn(NO_3)_2 + H_2O$ .

The authors conclude that manganese peroxide must be regarded either as  $MnO$ ,  $MnO_3$ , or else as  $3 MnO$ ,  $Mn_2O_7$ .

Possibly the body exists under isomeric states. The authors intend to continue their investigation.—P. J. II.

*On the Native Oxides of Manganese. First Part. The Psilomelanes and Wads.* A. Gorgeu. Bull. Soc. Chim. 1890, 3, 248—255.

Compare this Journal, 1890, 508.

*On the Technical Value and Preparation of Magnesia.* Ann. für Gewerbe und Bauwesen, 35, 1889, 139; and Proc. Inst. Civil Eng. 1890, 53.

MAGNESIA, formerly chiefly valued on account of its medicinal properties, has recently risen into great commercial importance owing to its infusibility and its employment as a lining for converters used in the basic process of steel manufacture. Caron, whose process was in the first instance followed, used calcined magnesite. This was made up with one-sixth of its weight of tender-burned magnesia, and from 10 to 15 per cent. of water, into a plastic state; it was then compressed into bricks in iron moulds, and burned at a dull red heat. Ehrenworth has pointed out that, if the refractory properties of the magnesia are to be evoked to the full, it is of the utmost importance that the whole of the magnesia should be dead-burned; the process, moreover, being carried so far as not only to expel the whole of the carbonic acid, but also to cause the full amount of shrinkage which this material is capable of attaining. This extreme amount of calcination is very difficult to effect, owing to the tendency of the magnesite to fly into splinters, and to drop to pieces when subsequently touched, and in consequence of its being such a bad conductor of heat the stone is very hard to burn in large pieces.

Recently dolomite, which is a double carbonate of lime and magnesia, has been used instead of magnesite. In order to utilise this material there are two processes before the public, that of Closson and that of Scheibler. Under the former plan, the calcined dolomite is mixed with chloride of magnesium, the chlorine in which separates from the magnesia and combines with the lime, yielding a soluble calcium chloride which can readily be washed out, leaving behind the insoluble magnesia. Under the process of Scheibler the calcined dolomite is treated with dissolved sugar, leading to the formation of sugar of lime, and leaving the magnesia. The solution of sugar of lime is then exposed to carbonic acid gas, which separates the lime as a carbonate, leaving the sugar ready for re-use. Both these systems of producing magnesia have the advantage of relative cheapness in their favour, owing to the low price of dolomite. Professor Frank, of Charlottenburg, has advocated the use of magnesia as a substitute for plaster of Paris for casts, and Grundmann has recently shown the advantage of employing a mixture of magnesia and powdered marble for this purpose. The author states that he has found, following the directions given by Hirzel, that a mixture of benzene

and magnesia is the very best possible substance for the removal of grease from drawings, or from any other material.

*Pick's System of Manufacturing Salt in Vacuo.* Perry F. Nursery. Iron, 35, 1890, 488—489.

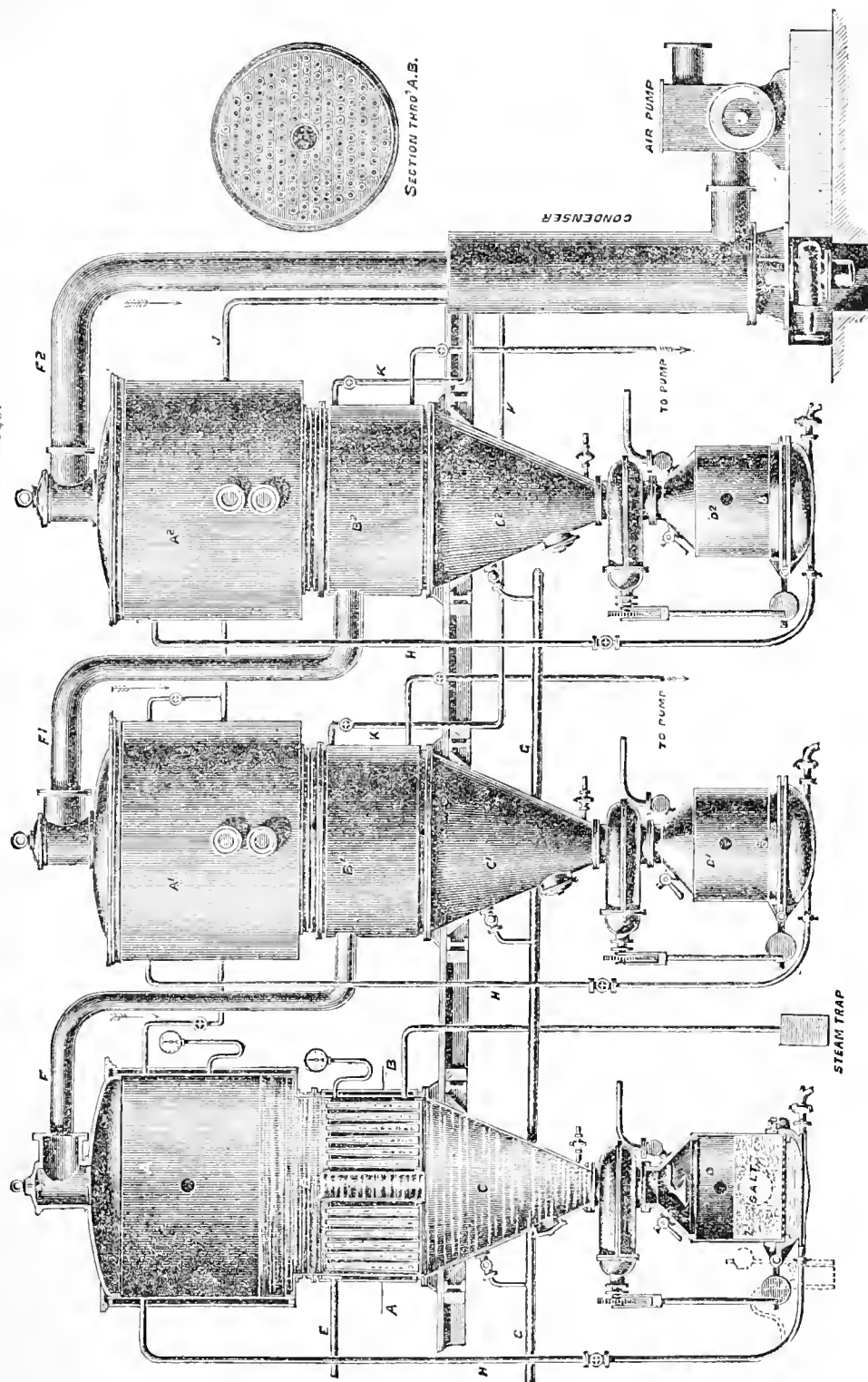
In the process in ordinary use wrought-iron evaporating pans, which have an area of from 600 to 1,000 superficial feet, are employed. Their usual form is oblong, and their depth ranges from 12 to 16 in. There are three or more fires under each pan, and there is usually a separate pan-house to each pan.

The disadvantages of the ordinary system of salt-making are numerous as well as serious. In the first place, although the salt is raked out regularly, it is found impossible to keep the bottom plates of the pans quite free from it. This deposit, together with the salts of lime and magnesia present in the brine, however carefully the operations be conducted, forms a scale upon the plates which causes them to rapidly burn away and buckle or bend. Repairs are consequently constantly going on in salt works, and indeed this item of maintenance is one of the most serious in a salt manufactory. The finer the salt produced, the heavier are the repairs, and the more frequently the pans require cleaning, the scale having to be frequently chipped by chisels from the plates and damaged plates to be replaced by new ones. These pans cover a large area, and they can never be kept tight. And it does not take long to disable them either, as may be imagined when it is stated that with the most careful working, and the best occasional patching, the life of a brine-pan cannot be put at more than about three years.

Besides repairs to the pans, there is the heavy fuel bill. In order to maintain the brine at the boiling point, and to secure the proper rate of precipitation of the salt according to the quality required, a large consumption of fuel is necessary. Another evil is the production of noxious gases, the effects of which are readily recognisable in the vicinity of salt works. As soon as scale begins to form in the pans the buckling action commences, the rivet seams open, and the brine leaks from the pans into the fire-holes, producing gases which are alike deleterious to animal and vegetable life. The ordinary process of salt making is thus slow and costly, and the output is comparatively small in proportion to the area occupied by the works and the quantity of fuel consumed.

Dr. Pick in his process introduces principles similar to those upon which the Rillieux or triple effect system depends, which system is, as is well known, in use for the evaporation of liquids in certain other manufactures, and he engrafs upon the Rillieux apparatus, the vacuum filter, which contributes much to the success of the method. As will be seen from the illustration, Dr. Pick's apparatus consists of three separate but similar sections, each section consisting of four main and closely connected parts. These are the boiling chamber A, the heating chamber B, the collecting chamber C, and the filtering chamber D. The three sections are placed side by side a few feet apart, and they are connected together by pipes as shown. The heating chamber B of the first section is placed in communication with a steam boiler, or with the exhaust steam from an engine, by means of the pipe E. The boiling chamber A of the first section is placed in communication with the heating chamber B of the second section by means of the pipe F, the boiling chamber A of the second section communicating in its turn with the heating chamber B of the third section by the pipe G. This latter section has its boiling chamber placed in communication with a jet condenser and air pump as shown. G is the brine inlet pipe to the various sections which is in communication with the brine tanks, the brine being raised by vacuum and supplied automatically to the several sections. H is a pipe for automatically conducting the brine from the filtering chambers D, D', and D'', to the boiling chamber of each section, under the special conditions which will be hereafter explained. J is a small pipe which connects the boiling chamber of the first and second sections with the condenser, and is used for assisting in maintaining a vacuum in each of those chambers. In like manner K is a small pipe for assisting the vacuum in the heating chambers of the second and third sections by clearing them of surplus air.

PICK'S APPARATUS FOR THE MANUFACTURE OF SALT IN VACUO.



The boiling chamber of each section is simply a cast-iron cylinder, of larger diameter than the heating chamber beneath it. The object of the increased diameter is to enable the chamber to contain a large quantity of brine with a minimum of depth and a maximum of evaporating surface. The usual level of the brine is seen in the first section, which in fact is a sectional view of a single apparatus, the second and third sections being shown in elevation. The heating chamber consists of a series of conical tubes of comparatively small diameter surrounding a central tube of larger diameter. The whole of the tubes are inserted in a tube plate at top and bottom, and enclosed in a cylindrical chamber, into which steam is admitted in the first section by the pipe E, and after imparting its heat to the brine it is condensed, and passes away to a steam trap as shown. In the second and third sections, the condensed water is drawn off by pumps. The reason for having the tubes conical is to prevent sealing, or, should sealing take place, that it may be easily removed, the larger diameter of the tubes being at the bottom. The author's experience of the working of the system, however, leads him to believe that sealing will not take place, so moderate is the heat applied, so rapid the circulation of the brine, and so powerful the ebullition maintained. It is to promote and increase the rapidity of the circulation that the central enlarged tube is employed. In operation, the brine heated by the small tubes passes upwards through them to the boiling chamber. It then makes for the centre, and returns downwards through the large central tube by reason of the lower temperature prevailing within that tube. The proportion of the volume of brine to the heating surface being much greater in the large tube than in the smaller tubes causes a reduced temperature within it, and the rapidity of the circulation is thereby increased in accordance with the natural law that heated fluids or gases ascend, while those of a lower temperature descend.

The settling chamber, immediately beneath the heating chamber, serves for collecting the salt as it is precipitated. It settles readily, as no movement takes place in the brine at that point. It is, of course, in direct communication with the upper or boiling chamber through the tubes of the heating chamber. This collecting chamber terminates in a sluice valve, and is in this way connected with the vacuum filter beneath it, which forms an important and essential feature of Pick's system. Each filter consists of an upper fixed portion and a lower hinged portion, the filtering medium being attached to the lower portion of the filter at its junction with the upper part. The upper part is fitted with an air inlet cock and a water pipe, ending in a rose for washing the salt if necessary. The lower part of the filter is connected with the boiling chamber by a tube, the lower portion of which, as far up as the valve, is flexible, and yields when the filter is open, as will be seen from the dotted lines.

The method of operating Pick's system is briefly as follows:—Each of the three sections having been charged

with brine to the proper level, which is that indicated in the boiling chamber A, steam is admitted to the heating chamber of the first section, in which the highest temperature is maintained. The brine in that section becomes quickly heated, and the steam given off from that brine enters the heating chamber of the second section, heating the brine in that section. The steam given off from the brine in the first section, after doing its work in the heating chamber of the second section, condenses and produces a vacuum in the boiling chamber of the first section, which vacuum is aided, if necessary, by opening the valve on the connexion with the vacuum pump. The pressure being reduced, the brine in the first chamber enters into violent ebullition at a comparatively low temperature. The same process is repeated in the second section, the steam chamber of the third section acting as a condenser, and producing a vacuum in the boiling chamber of the second section. The steam generated in the third section is drawn off by the vacuum pump, and condensed by the jet condenser as shown. It will be seen that the highest vacuum and the lowest temperature exist in the third section, while the highest temperature and the lowest vacuum occur in the first section. As the salt is precipitated it settles in the collecting chamber, and at stated intervals the sluice valve is opened and the salt and brine are admitted into the filtering chamber. After settling there for a few seconds, the sluice valve is closed and the air cock on the filter is opened. The valve on the ascension pipe H is then opened, and in a few seconds more the whole of the brine, in which the salt lies as in a bath, is automatically transferred to the vacuum chamber, leaving the charge of salt resting on the filtering medium and perfectly free from brine. The valve on the ascension pipe is then closed, the filter opened, and the charge withdrawn. The filter is then closed ready for another charge of salt.

It is stated that during the operation of letting down the charge of salt and withdrawing it from the vacuum filter, it is not necessary to stop working, the processes of evaporation and production being thus rendered simultaneous and continuous, and thus automatic.

The finest salt has been and is still being made equally well in one section, as in three sections, although of course not nearly so economically as if the complete set of three were adopted.

*Twenty-sixth Annual Report on Alkali, &c. Works by the Chief Inspector. Proceedings during the Year 1889. Price 8½d.*

THE report, which deals separately with Scotland (this part will be considered in its proper place), fills 98 pages.

The number of works under inspection was 903, of which 116 were alkali, and 787 works carrying on other processes. There is thus a diminution of three under the former heading and of 20 under the latter. The works were distributed as follows amongst the different districts:—

NUMBER OF REGISTERED WORKS.

	Dist. I. Ireland.	Dist. II. North of England.	Dist. III. Cheshire, N. Wales, and part of Lan- cashire.	Sub-Dist. Widnes.	Dist. IV. East Lan- cashire and York- shire.	Dist. V. South Mid- land.	Dist. VI. South- West of England and South Wales.	Dist. VII. South- Eastern Counties.	Sub-Dist. VIII. Eastern Counties.	Total.		
										1889.	1888.	1887.
Alkali works.....	2	18	15	18	34	10	8	1	10	116	119	116
Other works .....	47	87	75	13	131	124	125	87	98	787	807	811
Total .....	49	105	90	31	165	134	133	88	108	903	926	927

The above table, however, only gives the actual number of works; but inasmuch as many processes are sometimes carried on in one works, the total number of processes

under inspection is much greater than that of the works, as will be seen from the following table, which also gives the distribution of the processes in the different districts:—

TABLE OF SEPARATE PROCESSES UNDER INSPECTION.

	Dist. I. Ireland.	Dist. II. North of England.	Dist. III. Cheshire, N. Wales, and part of Lan- cashire.	Sub-Dist. Widnes.	Dist. IV. East Lan- cashire and York- shire.	Dist. V. South Mid- land.	Dist. VI. South- West of England and South Wales.	Dist. VII. South- Eastern Counties.	Sub-Dist. VII. Eastern Counties.	Total.	
										1889.	1888.
Alkali .....	2	13	14	16	12	5	4	1	2	69	69
Hydrochloric acid (cylinder) .....	..	..	..	..	13	3	..	..	8	24	29
Copper (wet process) .....	..	4	3	4	2	2	4	..	..	19	19
Cotton carbonising (alkali) .....	..	..	..	..	7	..	..	..	..	7	8
Sulphuric acid .....	11	24	22	18	41	27	42	4	28	217	221
Chemical manure .....	17	35	15	2	24	45	41	7	46	232	230
Gas liquor .....	..	1	2	3	18	4	..	..	..	28	28
Nitric acid .....	..	2	6	1	40	21	3	5	16	94	91
Sulphate and muriate of ammonia ..	21	35	24	7	55	58	60	36	38	334	320
Chlorine and bleaching powder ...	2	9	17	20	5	2	1	..	1	57	61
Sulphur recovery .....	..	1	2	6	1	2	..	..	..	12	..
Salt .....	10	8	55	1	..	4	..	..	..	78	77
Cement .....	2	13	1	1	7	7	9	40	13	93	88
Total .....	65	145	161	79	225	180	164	93	152	1,264	1,241

During the whole of the year 1889 there were 4,921 visits paid, and 4,808 tests made, by the different inspectors. In the Widnes sub-district alone there were 1,225 of the latter

taken and 737 visits made. A statement of the average escape of acid for the different districts is then subjoined, and is as follows:—

TABLE OF AVERAGE AMOUNT OF ACID GASES ESCAPING FOR EACH DISTRICT.

	Dist. I. Ireland.	Dist. II. North of England.	Dist. III. Cheshire, N. Wales, and part of Lan- cashire.	Sub-Dist. Widnes.	Dist. IV. East Lan- cashire and York- shire.	Dist. V. South Mid- land.	Dist. VI. South- West of England and South Wales.	Dist. VII. South- Eastern Counties.	Sub-Dist. VII. Eastern Counties.	Average.		
										1889	1888.	1887.
Hydrochloric acid in chimney gases. Grains per cubic foot	0.10	0.105	0.06	0.104	0.067	0.092	0.087	..	0.086	0.088	0.089	0.100
Hydrochloric acid. Amount escaping compared with that produced. Per cent.....	1.82	2.75	0.88	2.18	1.65	3.19	1.31	..	1.768	1.915	1.960	1.990
Acid gases escaping from sulphuric acid chambers given as SO <sub>3</sub> . Grains per cubic foot.....	1.23	1.29	1.38	1.43	1.10	1.31	1.83	1.61	1.16	1.370	1.490	1.500
Acidity of chimneys given as SO <sub>3</sub> . Grains per cubic foot..	0.985	0.63	0.69	0.65	0.51	0.47	0.91	..	0.89	0.715	0.737	0.740
Acidity of gases from manure works given as SO <sub>3</sub> . Grains per cubic foot.....	0.25	0.115	0.48	0.69	0.20	0.47	0.28	..	0.311	0.349	0.250	0.440

\* This figure was given in error in last year's report as 1.32.

It will be noticed that the escape of hydrochloric acid is less than half of that allowed under the Act, whilst the ratio between that which is produced and that which escapes is only as 100 : 1.94 instead of as 100 : 5.00 as allowed by the Act. Though in many cases there were escapes exceeding the legal limits, yet these were almost all assignable to disturbing causes, and were rapidly got under, there being but one prosecution throughout the whole year. This was of a firm in West Ham for failing to adopt the best means for preventing the escape of sulphuretted hydrogen gas in the manufacture of sulphate of ammonia. A fine of 10*l*. and costs was imposed. In the manufacture of salt there

has been, as compared with 1888, a falling off of about 12 per cent., the amount raised in the year 1889 being obtained from the following areas:—

	Tons.
Cheshire .....	1,154,649
Worcestershire .....	221,322
Staffordshire .....	6,500
Derham .....	392,300
Ireland .....	10,600
Scotland .....	7,419
Total in 1889 .....	1,792,790



There has been, as will be noticed, a large increase, about 50 per cent., in the output from Durham. The amount of the soda compounds manufactured is steadily on the increase from year to year, and of this the proportion which is produced by the ammonia-soda process is rising continuously. A comparison between the two rival processes in their consumption of salt brings the following results to light:—

	1889.	1888.	1887.
	Tons.	Tons.	Tons.
Leblanc process .....	584,293	585,498	577,381
Ammonia-soda process .....	219,279	212,181	158,636
Total .....	803,482	797,679	736,017

Besides the salt used in the actual manufacture of alkali, some 56,500 tons are employed in the well-known wet process, as it is termed, for extracting copper from burnt pyrites, bringing up the total consumption of salt to 640,703 tons.

In the production of sulphate of ammonia there has also been a continuous increase during the last few years. The amounts obtained from various sources are here given:—

#### AMOUNT OF SULPHATE OF AMMONIA PRODUCED IN THE UNITED KINGDOM.

	1889.	1888.	1887.
	Tons.	Tons.	Tons.
Gas works .....	100,711	92,896	85,022
Iron works .....	6,115	5,280	5,098
Shale works .....	23,953	22,072	21,098
Coke and carbonising works.	2,735	2,537	2,678
Total .....	133,604	122,785	113,896

The value of the sulphate of ammonia is thus about a million and a half if the price of the bulk be taken as being about 12*l.* per ton. But even this output, large as it is, is not one-tenth of the amount which might be obtained if all the ammonia evolved from coke ovens, iron works, &c., was saved. Very much of the sulphate of ammonia made here is exported to Germany to be employed in the cultivation of beetroot.

In the manufacture of the sulphate a considerable quantity of sulphuretted hydrogen gas is given off; this is either burnt or passed through ordinary absorbers holding oxide of iron, the latter being much the better plan. In this case the so-called continuous process of manufacture is better employed, as the strain put upon the absorbers at any given time, especially at the commencement of any operation, is not then so excessive. In the case of sulphuretted hydrogen gas it is understood that the whole of it must be absorbed, and that none is to be allowed to escape into the atmosphere.

In the number of works using the Leblanc process there is a further diminution in spite of the aid which the Chance-Claus sulphur recovery process is affording, though possibly in some of the works it came too late to be of service. There are no less than 12 installations of the Chance process now at work, and as many more are in the hands of the builders. The amount of sulphur now being recovered is about 400 tons per month, and this amount will shortly become about 1,000 tons weekly, or some 45,000 tons per annum. By this amount, therefore, will the output from the Sicilian sulphur mines be probably reduced. The total quantity exported from Sicily annually is about 360,000 tons, of which England takes 37,000 tons, and America half of the remainder.

As in the Chance process, the sulphur is recovered in the form of sulphuretted hydrogen; the volumes of gas to be manipulated are enormous. Thus, in one works alone some four million cubic feet of sulphuretted hydrogen are being produced weekly, and it is estimated that when the amount of sulphur recovered reaches 60,000 tons per annum, the quantity of sulphuretted hydrogen to be dealt with will touch 1,508 million cubic feet annually. Great difficulty is experienced in removing the last traces of the gas, much of it after passing through the Claus kiln existing and escaping as sulphurous acid gas. To produce as little of this latter gas as possible it has been found to be advantageous to keep the former in slight excess over the air admitted for the partial combustion of the gas and to keep the temperature of the kiln low.

In the different works much more efficient apparatus and appliances are being introduced. Thus, in the manufacture of mineral phosphate the mixing is conducted in a closed vessel provided with a stirrer driven from the outside, and the mixture is then sent into a closed chamber or "den" where it remains until the discharge of acid vapour has ceased, the gases evolved being passed through wash towers. After such washing there should not be more residual acid gas than is equivalent to 0.2 grains of sulphuric anhydride ( $\text{SO}_3$ ) per cubic foot. In the case of fetid gases the use of a cremator or combustion chamber for their decomposition has been recommended. This plan has been adopted by the Manchester Corporation for destroying the noxious gases given off in the boiling down of the liquid obtained by them in their pail system of removing house sewage.

With regard to the question of the evolution of black smoke, a committee has been formed in Manchester for the purpose of testing smoke appliances. It will pay attention to such points amongst others as (1) practical freedom from smoke; (2) reasonable amount of duty; (3) economy of fuel, &c. Experiments on and tests of the various appliances submitted to it will also be made. In this connexion may be mentioned the method of producing gas used by Messrs. Brunner, Mond, and Co. The distillation is conducted at such a low temperature that the ammonia is not destroyed whilst a smokeless gas is produced from the coal. Per ton of coal about 66 lb. of sulphate are yielded. A description is given of the usual method for boiling down brine, and of the damage which is done by the evolution of acid vapours to the surrounding vegetation. A new method, that of Dr. Pick, for the evaporation of the brine by triple or multiple effect, is referred to and illustrated by a diagram (see page 617). By this method the waste steam evolved from one pan is not allowed to escape to the atmosphere at once, but passes through a coil of pipes of the next in series, and thence to the third. This latter is connected up with an air pump which produces a partial vacuum causing "a brisk ebullition to take place at a low temperature; thus condensing the steam from an adjoining vessel sets up an ebullition there, and so on."

To this report is added the separate reports of each inspector, from which a few extracts are taken.

In district No. 1. there were 49 works under inspection in which were carried on 65 different processes, the amounts of material treated being as follows:—

	Tons.
Salt decomposed .....	6,728
Bleaching powder made .....	2,565
Pyrites burned .....	24,928
Mineral phosphates and bones dissolved	33,579
Sulphate of ammonia made .....	2,511
Salt produced .....	10,600
Cement produced .....	7,487

In district No. 11. attention is called to the fact that both the sludge from the carbonators in the Chance process and the exit gases from the Claus kiln promise to be sources of annoyance, and complaints have been made on both these points. In the Widnes sub-district the inspector reports that from the former source no nuisance has occurred. The

average escape from the various wet copper extraction works was as follows:—

	Average of four preceding Years.	1889.
Average HCl in cubic foot of smoke.....	0.125	0.155
Average total acidity of cubic foot of smoke.	1.380	1.500

Tables are also given of the average exit gases of the different works, such as alkali and chemical manure, both as regards HCl and SO<sub>2</sub>.

It is stated that in one works where sulphate of ammonia was being made that the escape of hydrocyanic acid from the decomposition of cyanides present in the gas liquor was so great that on two occasions it was found to be necessary to stop work. 382 tons of sulphate were obtained from the ammoniacal liquor of the patent coke ovens.

In district No. III., including Widnes and Runcorn, there were 121 registered works made up thus:—

Alkali works.....	33
Sulphuric acid works.....	9
Chemical manure works.....	11
Gas liquor works.....	5
Nitric acid works.....	5
Sulphate of ammonia works.....	28
Chlorine works.....	2
Salt works belonging to the Salt Union, Limited...	27
Salt works belonging to independent owners.....	2
Cement works.....	1
	121

Most firms are now adopting the Chance sulphur recovery process. The amount of waste being treated in the St. Helens district is about 1,400 tons weekly. The total quantity of pyrites burnt in the district (excluding Widnes and Runcorn) amounts to 90,365 tons. No brimstone is burnt. Total sulphate of ammonia equals 4,160 tons. The amount of white salt produced is 1,154,649 tons. Very full tables of the various acid escapes are inserted.

In the Widnes sub-district there are 31 registered works, *i.e.*:—

Alkali.....	18
Sulphuric acid.....	2
Chemical manure.....	1
Sulphate and muriate of ammonia.....	4
Gas liquor.....	1
Chlorine.....	2
Nitric acid.....	1
Salt.....	1
Cement.....	1
	31

A cement works has been started for the utilisation of the lime mud from the caustic soda and Chance sulphur recovery processes. The following return is of interest:—

	Tons.
Pyrites burnt for sulphuric acid or salt cake...	132,426
Salt made into salt cake.....	209,587
Salt used in wet copper extraction.....	18,720
Bleaching powder made (including chlorate of potash).....	64,927

It has been found that, although the average of the various exit escapes is satisfactory, yet that there have been many cases of excessive escapes of usually short duration. In this district also the Chance process is making rapid progress, some 150,000*l.* having been already spent on it during the past 18 months, and 10 works are either erecting or now possess the necessary plant. Here again the main source of annoyance is not from the Chance process, but from the exit gases from the Claus kiln, these containing from 8 to 15 grains of sulphur per cubic foot of the waste gases, the loss being thus considerable. To minimise this

loss the temperature of the kiln should, as already stated, be kept as low as possible. The gases after leaving the *dry* flowers chamber should be passed through a *wet* scrubber to remove SO<sub>2</sub> and finely-divided sulphur, and thence through vessels containing milk of alkali waste to absorb the SH<sub>2</sub>, this solution then passing to the carbonators in the usual way. There are 10 Claus kilns 25 ft. in diameter in the district, and 18 or 20 more designed, and each capable of producing 15 to 20 tons of sulphur per week.

In district No. IV. tables of the various exit escapes are given. Still less brimstone than last year is used for acid making. The total amount of ammonia in its various salts manufactured during the year 1889 was 22,483 tons.

Of this amount 89 per cent. is made by the continuous process and 11 per cent. by the intermittent.

In district No. V. there are 134 registered works and 180 processes. Tables giving the averages of the various escapes are added. In the Chance process it is recommended that large kilns of 30 ft. in diameter should be employed, thereby securing a lower temperature of decomposition and a better yield of sulphur. In the manufacture of sulphate of ammonia most of the sulphuretted hydrogen evolved is treated by the open oxide of iron purifiers. In one works per ton of sulphate of ammonia produced 100 lb. of sulphur have been obtained: each ton of coal yielded 27 lb. of sulphate; thus, for every ton of coal carbonised, about 1½ lb. of sulphur have been recovered in the Claus kiln.

In district No. VI. it is reported that many complaints have been made of the fumes evolved, *i.e.*, arsenic, oxides of lead and zinc, &c., in metallurgical processes, which do not fall within the boundaries of the Alkali Act. On this point, therefore, some of the sanitary authorities have decided to petition the Local Government Board. A new process for the manufacture of ammonium sulphate is that of Mr. E. Bowen's. The ammonia evolved is passed into the waste pickle from tin works. Oxide of iron is precipitated, which may be used in the manufacture of Venetian red, and the filtrate on concentration deposits crystals of the sulphate.

In district No. VII. the results of the tests made and of improvements effected in the works are tabulated.

In sub-district No. VII. there were 108 works and 152 processes. In this district occurred the only prosecution under the Act. Six works burn brimstone and four use spent oxide as a source of sulphur. In the latter case the utilisation of the material appears to be very difficult, there being rapid and unaccountable changes taking place in the chambers. A table giving the yield of sulphate of ammonia from various coals is given in full:—

#### CONTINUOUS PROCESS.

No.	Yield of Sulphate in Pounds per Ton of Coals Carbonised (Sulphate 2½ per Cent. NH <sub>3</sub> ).	Description of Coal used.
1	25.48	Chiefly Ravensworth Pelaw with one-sixth South Yorkshire silkstones.
2	25.46	Ravensworth Pelaw, Pelton, Pelton Main, and Waldrige.
3	24.37	Chiefly Newcastle district coal.
4	23.00	Wharfedale silkstones.
5	22.80	Chiefly Aldwarke main, Stafford, Thorncliffe, Barrow and Dodsworth Collieries with one-sixth Madeley.
6	22.42	East Pontop (Newcastle).
7	21.47	Barnsley silkstones and North Staffordshire.
8	19.50	Hutton and Mandin seams, Bolden Pit Co., Durham.
9	18.00	New Pelton (Newcastle).
	22.50	Average yield.

## INTERMITTENT PLANTS.

No.	Yield of Sulphate in Pounds per Ton of Coals Carbonised (Sulphate 24½ per Cent. $\text{NH}_3$ ).	Description of Coal used.
1	24.00	Chiefly Tawkesley (Thorncliffe) with small quantity of Wigan.
2	22.00	One half Pelaw Main; equal quantities Aldwarke Main and Normanton.
3	19.00	Equal portions Ravensworth Pelaw, and South Yorkshire silkstones.
4	18.95	Equal portions Aldwarke Main and Pelaw Main.
5	18.88	Equal parts Orrell (Wigan) and Barrow (Yorks).
6	18.60	Rockingham Colliery (South Yorkshire).
7	18.50	Equal portions of Hoyland and Wharcliffe silkstones.
8	16.84	Madeley and Lyeett Collieries.
9	11.45	Equal portions of Staffordshire and Wigan.
10	9.72	Barnsley silkstones and North Staffordshire.
11	6.81	Warleigh gas coal (South Yorkshire).
	16.79	Average yield.

In Scotland the number of registered works was 129, of which 16 were alkali works, the number in 1888 being 131, of which 16 were, as before, alkali. Of separate processes there were 175, distributed thus:—

Alkali (Leblanc process) .....	11
Muriatic acid (cylinder process) .....	4
Muriatic acid (copper extraction process) .....	2
Sulphuric acid .....	32
Chemical manure .....	49
Nitric acid .....	7
Sulphate, &c. of ammonia .....	51
Chlorine .....	8
Salt .....	11
	<u>175</u>

During the year the visits paid were 567 and 280 tests were taken.

The following quantities of materials were used and produced:—

Salt decomposed:—	Tons.	Tons.
In alkali works.....	33,376	
In copper works.....	7,110	
		40,486
Pyrites burnt .....		79,665
Brimstone burnt. ....		2,180
Bleaching powder manufactured .....		8,950
Bones and phosphates used in chemical manure works.....		47,434
Sulphate of ammonia manufactured:—		
From gas liquor works .....	9,907	
From blast furnace gases .....	5,645	
From distillation of shale .....	23,953	
		39,505
Salt manufactured.....		10,600
Nitric acid manufactured.....		3,173

Whilst the production of salt and of bleaching powder has thus diminished the consumption of pyrites has increased

10 per cent., the acidity of the exit gases from the sulphuric acid works was 1.67 grains  $\text{SO}_3$  per cubic foot, or considerably less than one-half of the limit under the Act, whilst in the alkali works the average escape was 1.46 grains  $\text{SO}_3$ . The average escape of  $\text{HCl}$  per cubic foot of the chimney gases was 0.088 grains, a figure identical with that obtained for last year. From the manure works the escape was equal to 0.25 grains  $\text{SO}_3$  per cubic foot. The manufacture of sulphate of ammonia has increased by about 6 per cent., the sources whence the ammonia is derived being noteworthy when compared with the corresponding sources in England. In this report also the Chief Inspector insists upon the importance of devising means for the prevention of the production of black smoke, a source of annoyance over which the inspectors have no power. On examination it has been found that in many cases the damage done to vegetation has been in reality caused by the clouds of smoke constantly poured forth, not by any noxious gases present in that smoke.—S. G. R.

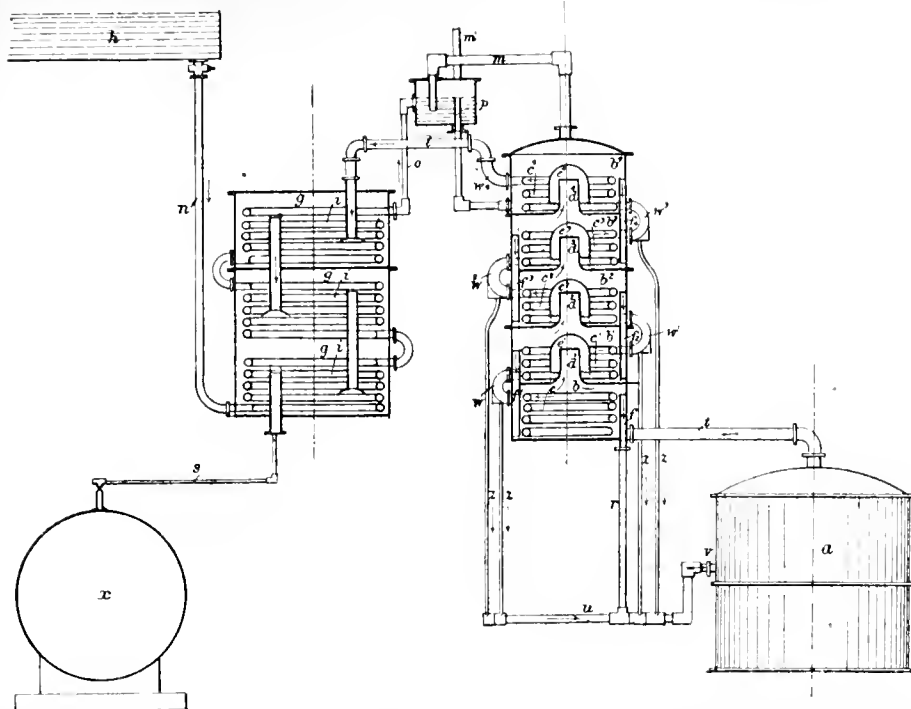
## PATENTS.

*Improvements in the Production of Magnesium Chloride and of Magnesium Oxychlorides.* F. M. Lyte, London. Eng. Pat. 6172, April 10, 1889. 6d.

THE object of the present invention is to provide a means of producing magnesium oxychloride without the usual drying and consequent loss of hydrochloric acid (see this Journal, 1887, 775—790). Anhydrous magnesium chloride is first prepared by igniting ammoniacal magnesium chloride, and is then mixed with the desired proportion of moderately calcined magnesia, which may be in powder or fashioned into balls or other shapes. By a second method, the dry double chloride of ammonium and magnesium is powdered and mixed with the dry magnesia, also in powder, and the mixture is heated carefully to a temperature not exceeding  $300^\circ \text{C}$ ., whereby all the ammonia is evolved in the free state, and the residue consists of anhydrous magnesia chloride or oxychloride, according to the proportion of magnesia added.—G. H. B.

*Improvements in and connected with the Distillation and Condensation of Ammoniacal and other Liquids, and in Apparatus therefor.* Solvay and Co., Brussels, Belgium. Eng. Pat. 8163, May 16, 1889. (Internat. Conv. October 19, 1888.) 8d.

IN the distillation of ammoniacal liquors with lime no provision has up to now been made for a preliminary separation of impurities such as carbonic acid and sulphuretted hydrogen and the like. On the contrary, all volatile products are distilled off together and then re-condensed. The apparatus shown in the figure has been devised to effect both a satisfactory elimination of the impurities and simultaneously to act as a concentrator. The working of the apparatus can be best gathered from a description of the plant itself. The weak ammoniacal liquor is run from the reservoir *h* by the pipe *n* through the coiled pipes *i* where it is heated by waste heat to about  $40^\circ \text{C}$ .; it passes thence to the washer *k* and then by the pipe *p* to the chamber *b*<sup>1</sup> and then by the pipes *f*<sup>1</sup>, *f*<sup>2</sup>, &c., to the lowest chamber *b*; from *b* it is carried by the pipes *r* and *v* to the distillation apparatus *a*; the vapours evolved now escape at *l* and then pass upwards through the coolers *c* to *c*<sup>1</sup>, where it will be best to leave them for the moment. Of the chambers *b*<sup>1</sup> to *b*, it will be clear that the lowest *b* must be the hottest, as the gases from the still are passing directly into it, and *b*<sup>1</sup> the coolest. Any carbon dioxide or sulphuretted hydrogen present in the liquors begins to be evolved in *b*<sup>1</sup> and the elimination is complete in *b*. The ammonia which is also given off in *b* is gradually re-absorbed, as it passes upwards through the cooler chambers by the "plunger caps" *e*<sup>1</sup> to *e*<sup>4</sup>. Finally the gases pass by the pipe *m* through the washer *k*, which removes the last traces of ammonia, whilst the residual gases, carbon dioxide and sulphuretted hydrogen, escape by *m*<sup>1</sup> to the exterior. Returning again to the ammoniacal distillate, this passes by



the pipes *w* and *l* to the cooler *g*. As it gives up its heat, so the weak liquors in the worms *i* as already mentioned are gradually warmed. The vessel *x* receives through the pipe *s* the purified concentrated ammoniacal liquor.—S. G. R.

*Process for the Manufacture of Chlorine which may be Employed in Combination either with the Ammonia-Soda Process or with the Leblanc Soda Process.* J. J. Alsberge, Ghent, Belgium. Eng. Pat. 8779, May 27, 1889. 6d.

THE mother-liquors of the ammonia-soda process are evaporated to dryness, the solid residue of the sodium chloride and ammonium chloride heated to 300° C. and the vapours of ammonium chloride are brought into contact with manganese dioxide at a temperature of 300° C. The chlorine combines with the manganese forming the chloride, oxygen and ammonia being evolved. The ammonia, together with the sodium chloride which was mixed with the ammonium chloride, is returned to the ammonia-soda process. The manganese chloride on gently heating with an excess of dioxide, manganese, and nitric acid yields its chlorine and is converted into manganese nitrate from which manganese dioxide and nitric acid are easily recovered by heating. In the Leblanc process the hydrochloric acid from the salt-cake furnaces may be led direct into stills containing manganese dioxide and nitric acid.—O. H.

*Improvements in the Manufacture of Nitrate of Ammonium and of a resulting Bye-product.* C. A. Burghardt, Manchester. Eng. Pat. 8841, May 28, 1889. 4d.

EQUIVALENT quantities of lead nitrate and ammonium sulphate are dissolved separately in water, and on mixing the solutions, lead sulphate is precipitated and ammonium nitrate is left in solution. The lead sulphate is separated by subsidence or other known means, and the solution is evaporated to the crystallising point, for the production of ammonium nitrate.—G. H. B.

*Improvements in the Method of and Apparatus for Condensing or Absorbing and Utilising Sulphuric and Sulphurous Acid Fumes.* W. Thorp, London. Eng. Pat. 8862, May 28, 1889. 4d.

CERTAIN sulphates and sulphides when heated evolve vapours of various sulphur acids. These are led together with a supply of air through a series of towers made of brick or stone or wood lined with lead, and filled with scrap-iron, down which water or a solution of sulphate of iron is kept running. In this way the iron is attacked by the vapours, and iron sulphate produced. When the towers are not working at a temperature of about 120°–160° F., a jet of steam must be introduced.—S. G. R.

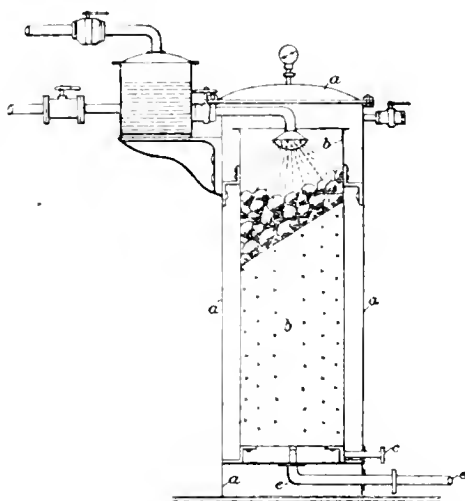
*Improvements in or connected with the Manufacture of Sulphate of Ammonia, and Apparatus therefor.* T. and G. Wilton, Beckton. Eng. Pat. 19,225, November 29, 1889. 8d.

THE improvement consists in the employment of a steam jet pump of specified construction by which the deposit of crystallised sulphate of ammonia formed in the saturators is removed to the drainers or vessels for allowing the mother-liquor to drain away. The same form of pump is also used for circulating the absorbing liquid through the saturators, which may be of the ordinary construction.—G. H. B.

*Improvements in the Production of Solutions of Alkaline Silicate or Water-Glass, and in Apparatus therefor.* A. J. Boulton, London. From P. Sievert, Dresden, Germany. Eng. Pat. 4021, March 14, 1890. 6d.

WHEN water-glass is dissolved in water, the solution obtained is usually not clear, and must, therefore, be clarified before use. In order to avoid the inconvenience of this, it is usual to buy the material already in solution, and hence a loss arises from the increase of warehouse room required for storage. The inventor partly dissolves the lumps of alkaline silicate by the action of a current of steam, and then by washing with a spray of water or a suitable alkaline lye. The solution then flows over more of the material, and

when it has acquired the requisite strength, is constantly drawn off. The process is carried out in the apparatus shown in the figure. *a* is the outer reservoir, in which is



placed the perforated vessel *b*, within which are the lumps of water-glass. Steam is injected either by the pipe *c* or the rose *d*, and through this latter is subsequently run the water or alkaline liquid which, after passage over the silicate, issues by the pipe *e*.—S. G. R.

*Improvements in Purifying Brine.* J. C. Mewburn, London. From C. C. Peck, Warsaw, U.S.A. Eng. Pat. 4652, March 25, 1890. 8d.

Brine is first heated to boiling, to decompose the bicarbonates of calcium and magnesium. Sodium carbonate is then added to decompose calcium sulphate and the chlorides of calcium and magnesium. The precipitate is removed by filtration or subsidence.—G. H. B.

## VIII.—GLASS, POTTERY, AND EARTHENWARE.

### PATENTS.

*Improvements in and relating to Decorative Pottery Ware.* W. H. Hollinshead and H. Griffiths, Burslem. Eng. Pat. 7245, May 1, 1889. 4d.

AFTER the article has been glazed and fired it is covered with a ground layer of dry colour and again fired. In order to obtain a dull gold surface, "liquid gold" may now be applied instead of "best gold," and the article again fired. Further ornamentation may be secured by scratching away the gold to form a design and then filling in with various other colours.—S. G. R.

*An Improved Process for Depositing Metal on Glass, Porcelain, and other similar Ware.* O. P. Erard and B. J. Round, Birmingham. Eng. Pat. 7761, May 9, 1889. 6d.

IN carrying out this invention a flux of the following composition is first prepared: calcined borax, 7 parts; sand, 3 parts; oxide of lead, 4 parts; nitrate of potash, 1 part; phosphate of lime, half a part; and white arsenic, 2 parts. To every  $4\frac{1}{2}$  parts of the flux 10 parts of silver are added, the whole ground together and mixed with turpentine and

laid on as a "wash" in the ordinary way to form the required design. The whole is fired and thoroughly cleaned and the desired metal deposited electrolytically. The process is applicable to china, glass, porcelain, &c.; and not only is delicate glass strengthened by the metallic deposit, but a base is formed upon which other ornaments may be more or less easily soldered. Drawings of articles thus treated are given in the specification.—S. G. R.

## X.—METALLURGY.

*The System of Amalgamation of Gold Ores in use at the Massachusetts Institute of Technology.* R. H. Richard. Technology Quart. 3, 45—51.

See under XXIII., page 656.

*Physical Properties of Nickel Steel.* J. Hopkinson. Proc. Roy. Soc. 47, 138—139.

FROM experiments on samples of wire drawn from nickel steel containing 25 per cent. of nickel and 74 per cent. iron, by the Steel Company of Scotland, the author finds that nickel steel can exist in two states, magnetic and non-magnetic, over a range of temperature from below freezing to 580° C. The wire becomes non-magnetisable after heating to redness, whether cooled slowly or by being plunged into water, but when cooled by solid carbonic acid it resumes its magnetisable state. The electric resistance is very different in the two states, and the change in resistance effected by cooling with solid carbonic acid is almost as remarkable as the change in magnetic properties.

Of five non-magnetisable samples the highest breaking stress was 50·52 tons per square inch, the lowest 48·75; the greatest extension 33·3 per cent., the lowest 30. Of five magnetisable samples the highest breaking stress was 88·12 tons, lowest 85·76; highest extension 8·33 per cent., lowest 6·70. The broken fragments of both were magnetisable.—J. M. H. M.

*Liquation of Gold and Platinum Alloys.* E. Matthey. Proc. Roy. Soc. 47, 180—186.

IT is a well-known fact that when molten alloys of certain metals are cooled, some of the constituents separate and become concentrated either in the centre or in the external portions of the solidified mass; to this segregation the name of liquation is given. It is especially noticeable in the case of silver-copper alloys, and its importance is now being widely recognised in almost all branches of metallurgy. The author has studied the behaviour of alloys of gold and platinum. There are two methods of parting gold from silver, the one with nitric acid, the other with sulphuric acid. When platinum is present in the gold or silver, and the nitric acid process is employed, the platinum when existing in small proportions is dissolved out with the silver, leaving the gold free; sulphuric acid does not extract the platinum. As it has been found that in an ingot obtained from this residue different parts of it do not possess the same composition, it is evident that the platinum has been re-distributed by liquation. Spheres of gold containing platinum were cast in iron moulds. Parts were taken from different parts of the two hemispheres and assayed. Two alloys were treated in this way; one consisted of 880 of gold to 050 of platinum, and the other of 700 of gold to 120 of platinum. In the one case the maximum difference between the gold percentage is a variation of 032, viz., 887 on the outside against 883·8 at the centre of the alloy; and in the platinum 017·5 on the outside against 052·5 at the centre, an extreme variation of 005 is shown. In the other case the maximum difference between the gold percentage is a variation of 041, viz., 732·4 on the outside, against 694·1

at the centre of the alloy, and in the platinum 122 on the outside against 166 at the centre, an extreme variation of 0.44. The platinum, on cooling, therefore, liquates from the gold, and becomes concentrated towards the centre of the alloy. The following table represents the assays of portions cut from one end of ingots of gold and platinum, and the actual amount of fine gold obtained. The results prove that the percentage of gold in the outer portion of ingots of platinum-gold alloy does not represent the true percentage of gold in the alloy:—

Weight in Troy Ounces.	Platinum by Assay.	Gold by Assay.	Percentage of Gold by the Fine Gold actually obtained.
728.5	.111	.825	.812
355.0	.120	.660	.630
589.5	.120	.800	.780
435.0	.045	.850	.845
480.5	.086	.842	.830
473.0	.110	.830	.821

The results given were observed in platinum-gold alloyed with silver, with copper, and with both silver and copper; but, in order to prove whether or not such alloy had any tendency to carry the platinum to the centre of the mass, 900 parts of fine gold were melted with 100 parts of pure platinum, and, after repeated meltings, cast in the same mould as previously used. The result was as before, the ratio of gold to platinum on the exterior being 900 to 0.98 against 845 and 146 at the centre of the mass.—J. B. C.

*The Laws of Annealing, and its effect on the Mechanical Properties of Metals.* A. Le Chatelier. *Compt. Rend.* 110, 705—708.

THE author has observed that above a certain temperature every metal may be elongated to the point of rupture by the action of a constant load, the rate of extension depending on the magnitude of the load. This extension is produced at all temperatures in all metals, under the influence of a load sufficient to produce a permanent elongation.

From a large series of experiments on the tensile strength of metals at elevated temperatures, of which a few are given below, the following conclusions are drawn: (1) If a metal be kept at a constant temperature, the elastic strength and breaking load constantly decline towards definite limiting values at a gradually diminishing rate; (2) the limiting values are lower according as the temperature is more elevated; (3) the limiting values are higher when the initial values are high, but not to a proportionate extent.

From these generalisations it is further inferred that—

(A.) When the temperature remains constant, a metal is deformed by a given load at a gradually diminishing rate, and tends towards a determinate state of equilibrium. The magnitude of the deformation corresponding to the different states of equilibrium increases more rapidly than the loads which occasion it. The breaking load increases and the extension diminishes with the speed of loading. The deformation occasioned by shocks of the same intensity are inversely proportional to the rapidity of the shock.

(B.) When the temperature rises, without any other change occurring, the deformation increases and the resistance to breaking diminishes.

L = elastic strength in kilos. per millimetre of original section. R = breaking-load.

Copper wire hardened by drawing.

At the ordinary temperature L = 42 kilos. and R = 43 kilos.

After Heating.	At 300°.		At 350°.		At 430°.	
	L.	R.	L.	R.	L.	R.
hrs. min.						
0 12 .....	39.5	41	26.5	33.8	11	27.2
0 15 .....	33	37	11	27.2	..	26.6
1 0 .....	26	33.6	..	26.9	..	26.5
5 0 .....	12	27.4	..	26.9	..	26.5
10 0 .....	..	27.1	..	26.9	..	26.5

Breaking-load in kilos. after prolonged heating.

Before heating.....	27.5	32	43
After prolonged heating to 350°.	25.7	26.3	27

Loads (in kilos.) which metals can support without breaking for definite intervals at elevated temperatures.

	30 Secs.	1 Min.	5 Mins.	15 Mins.	60 Mins.	Indefinite.
Cast iron at 15° .....	39.7	38.8	37.6	37	36	..
Aluminium, at 15° .....	14.9	14.6	14.4	14.2	13.4	..
Zinc, at 15° .....	..	24	21	16.5	11.5	5
Copper, at 15° .....	27.8	27.1	26.3	25.8	25.1	23.8
Copper, at 200° .....	20.7	20.2	19.7	19.1	17.7	15
Copper, at 300° .....	17.7	..	..	15	13	9.5
Copper, at 400° .....	14	13	11.6	9.7	7.5	3

—S. B. A. A.

*The Annealing of Metals.* Faurie. *Compt. Rend.* 110, 738.

A TEMPERATURE (A) exists for every metal beyond which the resistance continues to diminish without any further increase in malleability; another temperature (B) exists at which the malleability commences to diminish. The difference between these temperatures appears to be a function of the residual effects of previous annealings.—S. B. A. A.

*On the Use of Carbon and Coke Bricks in Blast-Furnace Hearths.* F. Burgers. *Stahl und Eisen*, 10, 112; *Proc. Inst. Civil Eng.* 1890, 56.

THE maintenance of the hearth and bottom in the blast furnace is to most smelters a troublesome and anxious task. Even in a new furnace, with the corrosive slags required by modern methods of working, the tendency is, according to the author, for the refractory brickwork of the hearth to melt and flow like butter in the summer sun; hearths with 3 or 4 feet thickness of walls, being reduced in a short time to a few inches, and necessitating the use of continuous cooling with water, which, by rendering the bricks brittle, is often attended with grave dangers from escape of molten metal through the weakened structure. This corrosive action may be well studied on the cinder-fall of the furnace, the best fire-bricks being eaten out into a gutter in an hour or two if the current of molten cinder is allowed to flow over them. These circumstances led the author to seek for a more resisting material, and finding that the lining of a furnace when blown out was often found to be protected in the hearth by a mixture of coke-dust and graphite, cemented by lime and cinder; and also that practice showed that the best lining for the cinder-channel was formed of coke-dust and ashes, he was induced to experiment with bricks containing coal- or coke-dust, graphite, and other substances in

admixture with fire-clay. The first trials were made in 1882, and the bricks made by Dr. Otto and Co., although partly defective owing to the carbon being considerably diminished in the burning, stood remarkably well against slag-corrosion. A patent was, however, refused in Germany, owing to the fact that mixtures of coke-dust and clay had been in use for a long time in the Harz lead-furnaces. In 1885, on the publication of Mr. Poncelet's paper on the manufacture of ferro-manganese (Proc. Inst. C.E., Vol. LXXXI. 408), stating that at La Voulte and Tamaris the hearths and bottoms of the furnaces were built of bricks made of gas-retort carbon, containing 1 to 2 per cent. of ash, mixed with tar and burnt, the author tried finely-ground coke, with a low proportion of ash instead of retort-carbon, and obtained equally good results. The hearth of the No. 2 furnace at Gelsenkirchen was in that year rebuilt with these bricks, and has stood the wear of a large daily make up to the present time. They have also been applied in the new furnaces at the Rhenish steel works at Rombach, and in the lead-smelting furnaces at Mechernich. The principal advantages are the suppression of water-cooling, the maintenance of the original shape of the hearth from the beginning to the end of the period of blowing, and the prevention of furnace bears on the bottom. The hearth may also be completely isolated from the foundation, as has been done by Mr. Lürman at Ruhrort and Rombach. Other advantages are to be found in the circumstance that carbon, even at the highest temperatures, expands but slightly, if at all, and that it is an exceedingly bad conductor of heat. The cost of these bricks is about 5*l.* per ton. The manufacture, which is accompanied with the production of a great quantity of soot and smoke—the first experiments, according to the author, having been comparable to lump-black making—has been carried out at the Mechernich works, which, being surrounded by barren moors, is not liable to interference from the sanitary authorities for nuisance. Mr. F. W. Hupertz, the manager of the works, has, however, devised means for rendering the soot harmless to the works and the neighbourhood, and under his management the bricks for the furnaces mentioned above, and some others, have been produced.

#### PATENTS.

*Improvements in Hydro-Metallurgical Processes.* J. H. Selwyn, London. Eng. Pat. 843, January 16, 1889. 6*d.*

THE object of the patent is to cheapen wet metallurgical processes chiefly by dispensing with the roasting of the ore generally necessary. The finely crushed ore is added to a solution consisting of nearly saturated brine, to which has been added 5 per cent. of hydrochloric acid, 10–15 per cent. of sodium or potassium nitrate or nitrite, and 1–30 per cent. of sulphur chloride, according to the nature of the ore. The mixture is kept hot and extraction is complete in from 1–8 hours, after which the liberated sulphur is collected, and the liquors containing dissolved metals syphoned or filtered off, and the gold, silver, copper, &c. which they may contain successively precipitated by known methods, electrolytical or chemical. The process may also be used for obtaining sulphur from pyrites. The chlorine evolved is collected and used to make chloride of sulphur afresh.

—B. B.

*Improvements in the Treatment of Ores for the Recovery of Metals and in Apparatus therefor.* E. B. Parnell, London. Eng. Pat. 2931, February 19, 1889. 8*d.*

THIS is a combination of appliances for raising ores to a high temperature and then quenching them in water, thereby rendering their disintegration easier. The ore is caused to pass through a chamber by means of a helix or screw-conveyor, the shaft of which is hollow and conveys air heated to 1,600 F. The ore when sufficiently heated falls into a tank from which it is removed by means of an elevator, and passes through a second chamber where it is dried by means of the waste heat from the first chamber. It then passes to rollers or stamps.—H. K. T.

*Improvements in the Wet Method of Extracting Gold.* J. H. Pollok, Glasgow. Eng. Pat. 5578, April 2, 1889. 8*d.*

THESE improvements relate to a process described in the Eng. Pat. 17,495 of 1887 (this Journal, 1889, 444) for the treatment of auriferous material by means of chlorination under hydraulic pressure. The crushed and suitably prepared ore, together with the necessary reagents, are placed in a cylinder capable of being rotated on a horizontal axis. The air in the cylinder is removed and replaced by water. The cylinder is then closed and made to rotate, hydraulic pressure being applied by means of a pipe passing through the centre of rotation at one end of the cylinder. After a sufficient time the contents of the cylinder are run out and treated in the usual way.—H. K. T.

*Improvements in the Method of and Means for Treating or Washing Crushed Ores, Coal, and Similar Materials.* O. Bilharz, Freiberg, Germany. Eng. Pat. 7500, May 4, 1889. 1*s.* 1*d.*

CRUSHED ore is sorted by means of sieves, and is then fed into one end of a channel or trough provided with funnels which collect the ore as it settles. The funnels are grouped into three sets. The first set communicate with a series of settling apparatus, each of which consists of a funnel-shaped box containing a jigger-sieve charged with granulated ore of a given grade. The boxes are connected with receptacles for receiving the classified ores. The second set of funnels of the trough are connected with similar settling apparatus from which the graded ore passes to washing tables. The aprons of the latter are provided at their edges with studs which engage in notches or recesses in the rollers. A stream of water passes to their under side to prevent adhesion with the table. The ore from the third set of funnels is treated similarly to that from the second set. Finally, the ore passing from the channel or trough is caught on a band or apron, from which it is discharged by a screw-conveyor and fed to sorting and washing apparatus. —H. K. T.

*Improvements in the Manufacture of Steel or Steel-like Compounds.* J. Riley, Glasgow. Eng. Pat. 8492, May 22, 1889. 6*d.*

THE improvements consist in the use of nickel and aluminium in the manufacture of steel. When crucible steel is being made, nickel in the form of bars, cakes, or grains is melted with iron or steel scrap. A suitable proportion of ferro-manganese is then added and finally aluminium or an alloy thereof. When a converter is used the metal is deoxidised with ferro-manganese in the usual way and then nickel is added. Finally, more ferro-manganese is added and immediately afterwards aluminium. In the open-hearth process pig iron with a rather large proportion of scrap is decarbonised in the usual way, ferro-manganese is added, next nickel in the form of bars, then more ferro-manganese, and finally aluminium or its alloy. Instead of nickel alone a compound of nickel, copper, or iron is sometimes used, preferably the matte produced in the smelting of ores from Sudbury, Ontario, America. The usual proportions in 1,000 parts of steel are: nickel 5 to 500 parts; the above matte (containing 10 per cent. of nickel and 25 per cent. copper) 25 to 250 parts; and aluminium 1 part. —H. K. T.

*Improvements in Effecting the Re-carburisation of Steel or Ingot Iron.* T. Twynam, London. Eng. Pat. 8543, May 22, 1889. 6*d.*

THIS process has for its object the re-carburisation of low carbon steel or ingot iron produced in the open hearth or Bessemer process. For this purpose the charge of metal in the furnace having been sufficiently purified is tapped and allowed to flow over sticks of wood charcoal arranged in a chute leading to the ladle. In order to prevent the charcoal from being carried forward by the metal some of the bricks in the sides of the chute are made to project. Where a high percentage of carbon is required it is advisable to employ some of the charcoal in the form of small pieces placed in the forepart of the chute, so as not to unduly



arrest the flow of the metal, and it is preferred to have the chute curved at a considerable angle or arranged in two sections so that the second section is at right angles to the first and can be readily detached from it.—H. K. T.

*Improvements in the Distillation of Sodium and Potassium and their Alloy.* W. White, Cheshunt. Eng. Pat. 9784, June 14, 1889. 6d.

SODIUM vapour as ordinarily evolved from the retort is liable to considerable oxidation during condensation, and the patentee proposes as an improvement to collect it by downward distillation. The exit tube passes through the bottom of the crucible or downwards from the end of the retort, according as one or the other is used, into a vertical receiver provided at its upper part with an opening for the escape of the accompanying carbon monoxide.—B. B.

*Improvements in the Conversion of Crude Iron into Malleable Iron and Steel, and Apparatus therefor.* H. A. Becker, Blaenavon. Eng. Pat. 9905, June 17, 1889. 8d.

This invention relates to a form of converter which, instead of being vertical and supported on trunnions, is placed with its long axis in a horizontal position, and is capable of being rotated on this axis. By this means the contents can be discharged without it being necessary to raise the mass of metal. The tuyers are placed in a line in the side opposite to the mouth of the converter, and are thus simultaneously submerged on rotating the converter. They are directed towards its closed end. By suitably placing the converter, the pressure of metal to be overcome by the blast can be easily adjusted.—H. K. T.

*An Improvement in Regenerative Gas Furnaces.* R. S. Casson, Brierley Hill. Eng. Pat. 10,050, June 19, 1889. 6d.

This invention relates to regenerative furnaces for open-hearth furnaces in iron and steel making. The improved furnace is placed *above* the melting chamber instead of below. It is claimed that there is not only a saving in cost of erection, but that it will be easier to repair and cheaper to maintain, as from its position the furnace will not be subjected to injury from slag, melted brickwork, or fluid metal.—D. A. S.

*Improvements in obtaining Precious Metals from their Ores, and in Means or Apparatus employed therein.* D. Dennis and T. K. Rose, Denver, Colorado, U.S.A. Eng. Pat. 11,788, July 24, 1889. 8d.

AN apparatus for the extraction of precious metals by the wet chlorination or bromination processes. It consists of two vessels placed one above the other, the lower ends of each being provided with filter beds of canvas and sand or other porous material, which can be lowered at will to enable the residues to be removed. Material from a mixing vessel is charged into the upper vessel, from which, after the lapse of a suitable period, the clearer portion is run into the lower vessel. Fluid pressure is now applied, the liquid in the upper vessel passing into the lower one, and thence through the lower filter bed into a suitably disposed collecting vessel. The liquid, if not sufficiently clear, can be returned into the upper vessel. The clear filtrate is now treated in the usual way.—H. K. T.

*Improvements in the Manufacture of Steel.* M. F. Coombes and A. W. Hyde, Louisville, Kentucky, U.S.A. Eng. Pat. 2981, February 25, 1890. 4d.

MALLEABLE cast iron or low-carbon steel is heated to whiteness and plunged into a solution of a carbohydrate (sugar, molasses, or honey) or of an acid resulting from the oxidation of a glycol (lactic, tartaric, or oxalic acid) with or without the addition of acids or alkalis.—H. K. T.

## XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

*Electrolytic Separations.* E. F. Smith and L. K. Frankel. J. Frank. Inst. 1890, 129, 236—237.

See under XXIII., page 656.

### PATENTS.

*Improvements relating to Electric Accumulators or Secondary Batteries, and to the Manufacture of Plates or Electrodes therefor.* C. Pollak, Paris, France. Eng. Pat. 7428, May 3, 1889. 8d.

THE lead plates are passed between rollers made up of steel discs, suitably grooved or notched at their peripheries in order to indent the surface of the lead in a suitable way for the reception of the active material. Spongy lead is preferably made by coating the indented plate with a paste of sulphate of lead and sodium or potassium chloride, immersing the whole when dry in a solution of the same chloride, and then passing an electric current through it. A special box-like form of plate is described as suitable for railway work.—B. T.

*A Process and Apparatus for Extracting Precious Metals from Ores or Quartz.* R. L. Cousens, Johannesburg, South Africa. Eng. Pat. 8608, May 23, 1889. 8d.

THE ore or quartz is pulverised and passed over magnetised rollers, whereby iron is removed. It is then placed in a rotating barrel provided with metallic ends, together with a quantity of lime, salt, water, and sometimes a little acid, and an electric current is passed between the terminals. After a sufficient time mercury is introduced, and the rotation continued. The mixture is next run into an agitator and thence through an electrolysis vessel. The latter contains a metallic plate embedded in sand saturated with an acid or alkali. On this is placed a porous diaphragm, and on this, mercury. The latter is connected with the negative terminal of a battery, the metallic plate being connected with the positive terminal. The ore and water flow over the mercury, contact being ensured by a vertical partition extending nearly to the surface of the mercury. From the electrolyser the ore passes over an amalgamated copper plate, kept bright by felt-covered metallic pads continually passing over it, the pads and plate being connected with opposite terminals. The ore passes through several electrolysis vessels similar to the above, and finally between two metallic plates connected with the opposite terminals of a dynamo or battery, the lower plate being covered with a blanket.—H. K. T.

*Improvements in Secondary Batteries.* V. H. Ernst, Jersey, U.S.A. Eng. Pat. 13,818, September 2, 1889. (Internat. Conv. February 12, 1889.) 8d.

THE plates are such as might be made by placing a number of lead tubes horizontally and side by side, and burning the sides of contiguous tubes together. At regular intervals the tubes are cut into from the faces of the plate so as to expose the interior. The active material is packed into the tubes, a core of some loose and preferably absorbent material occupying the centre of each tube. This core allows the electrolyte to penetrate into the active material, and at the same time allows contraction and expansion of the latter to go on with safety. The plates may be arched, if desired, for greater strength, and are perforated at frequent intervals to allow of the escape of gas. (See Eng. Pat. 1281 of 1890.)—E. T.

*Process of Manufacturing Metallic Electrodes for Galvanic Batteries.* F. Marx, Berlin, Germany. Eng. Pat. 20,217, December 16, 1889. 4d.

THE inventor states that if electrodes be used, made from precipitated or electro-deposited metal, instead of from smelted metal, the voltage is increased, and a cell of the same size is able to give a much larger current. For this purpose the loose porous metal obtained by precipitation or by electrolysis is washed carefully, and then rolled or hammered into one concrete mass, from which the electrodes are formed. With zinc, a little mercury, or suitable mercury salt, is added before the mechanical treatment.

—E. T.

*Improvements in Secondary Batteries.* V. H. Ernst, Jersey City, U.S.A. Eng. Pat. 1281, January 24, 1890. (Internat. Conv. September 28, 1889.) 6d.

THE frames of the plates are cast with a number of parallel longitudinal holes through them, and other holes are made opening into these longitudinal ones in the face of the plates. Cores of suitable size are inserted in the longitudinal holes, and in the annular space round them is pressed the active material. When this material is dry the cores are removed and the plate is ready for use. These centre holes expose a much greater surface of active material to the electrolyte, and thus give greater capacity to the cell. (See Eng. Pat. 13,818 of 1889.)—B. T.

*Improvements in Portable Galvanic Batteries, and in Glow Lamps connected therewith.* A. L. Davis, Twickenham. Eng. Pat. 2364, February 13, 1890. 6d.

THE battery is made up as a drum with polygonal ends. This drum is partially filled with a suitable electrolyte, and has electrodes of semicircular shape, parallel to the ends of the drum. The drum always rests with its axis horizontal, the sides of its polygonal-shaped ends forming secure bases. In one position the plates are out of the liquid, but by rolling the drum to other positions, the whole, or part of the plates can be immersed. The drum can be divided by transverse divisions into two or more independent cells. An incandescence lamp is so attached to the centre of one end of the drum, that it can be made to throw its light in any desired direction.—E. T.

*An Improved Electrolytic Solution for Galvanic Batteries.* C. Lütke, jun., Berlin, Germany. Eng. Pat. 2819, February 21, 1890. 4d.

IN order to prevent polarisation a certain quantity of nitrate of mercury is added to the exciting fluid, consisting of a saturated solution of one or several chlorides such as those of iron, zinc, copper, &c., whereby the zinc is kept amalgamated and the electrolyte is to a certain extent regenerated.

—B. T.

## XII.—FATS, OILS, AND SOAP MANUFACTURE.

*On Commercial Turpentine Oil.* A. Wilson. Chem. Trade J. 6, 316.

See under XXIII., page 657.

*Schmidt's Process for converting Oleic Acid into Solid Fatty Acids.* R. Benedikt. Monatsh. 1890, 11, 71—83.

See under XXIII., pages 658—659.

*The Alkaloids of Cod-liver Oil.* A. Gautier and L. Mourgues. Bull. Soc. Chim. 1889, 2, 213—238.

See under XX., pages 647—648.

## XIII.—PAINTS, PIGMENTS, VARNISHES, AND RESINS.

*Experiments on the Preparation of Boiled Linseed Oil.* F. H. Thorp. Technology Quart. 3, 9—17.

THE particular changes which take place in the so-called "boiling" of linseed oil have received little attention. The drying of this oil is due to its oxidation, and the oxidation is greatly hastened by the "boiling." The dryer or substance added to the raw oil partially oxidises the oil during the boiling, and when a thin layer of the boiled oil is exposed to the air it rapidly hardens owing to further oxidation. The dryers most commonly used are litharge, pyrolusite, borate of manganese, and zinc white. Recently the use of oxalate of manganese has been proposed for this purpose. According to Mulder, some of the linoleic acid is converted into linoleic anhydride during the boiling. This anhydride is a tough elastic body. At the same time, some of the metal of the dryer forms a salt with the remainder of the acid, and this salt gives hardness to the varnish. Hazura and Bauer find that the rate of oxidation, and consequent hardening, of the oil, depends on the ratio of linoleic and linolenic acids present. When linseed oil is oxidised it undergoes very marked changes in its physical properties, the colour becomes darker, the oil becomes more viscous, and there is an increase in weight sometimes equalling 8 per cent. Some carbon and hydrogen are also given off. These changes occur also during the process of boiling. The present paper contains a series of experiments by the author on the effect of temperature and of the substances added in producing the essential properties of an oil varnish. The oil used was a very light yellow, cold-pressed, raw, Calcutta linseed oil. 50 cc. were used in each experiment (45·7 grms.). The measured volume was heated in a glass beaker of 150 cc. capacity or a sand bath, several beakers being heated on the same sand bath. The sand was raised round the beakers to about one-half the depth of the oil, and the oil was frequently stirred during the heating. The substances added were dried previously, and the best results were obtained with temperatures between 230° and 275°. After heating the character of the film produced was determined by coating small plates of glass with the oil and leaving them in a light room to dry. The films were considered to be dry when they could be handled without leaving finger-marks upon them. The chief points looked for were the colour and hardness of the film. The following table gives the results of these experiments:—

Dryer.	Quantity of Dryer used.	Time of Boiling.	Temperature.	Time required for Drying.	Character of the Film.
	Grams.	Hours.	°	Hours.	
Litharge.....	1.0	2½	220	6	Almost colourless.
Litharge.....	0.2	2½	250	10	Almost colourless.
Litharge.....	0.8	1½	Above 250	10	
Lead peroxide .....	1.072	1½	220	Several days.	Deeply coloured.
Lead chloride.....	1.247	2½	250—300	24	Somewhat coloured.
Red lead.....	1.024	2½	220—285	24	Deeply coloured.
Lead oxalate.....	1.323	2½	over 300	Did not dry.	
Lead tartrate .....	1.6	2½	270	24	Deeply coloured.
Lead acetate .....	1.46	2½	..	12	Somewhat coloured.
Lead borate .....	1.105	1½	220 and 300	20	Slightly coloured.
Lead carbonate.....	1.197	2	225	10	Slightly coloured.
Zinc oxide .....	0.5	2½	250	45	Nearly colourless.
Zinc sulphate.....	1.987	2½	285	15	Nearly colourless.
Zinc sulphate .....	1.5	2	Below 230	45	Yellow in colour.
Zinc acetate .....	1.0	2½	235—280	10	Colourless.
Zinc borate .....	1.0	2	240	40	Nearly colourless.
Zinc borate.....	0.5	1½	210	46	Nearly colourless.
Zinc borate.....	0.5	1½	240	46	Nearly colourless.
Zinc citrate.....	1.5	2½	230	36	Nearly colourless.
Manganese acetate.....	1.0	2½	225—250	20	Nearly colourless.
Manganese borate .....	1.625	2½	220	20	Colourless and hard.
Manganese sulphate .....	1.72	2	240	40	Colourless.
Manganese oxalate.....	1.64	2	230	40	Colourless.
Manganese acetate.....	0.5	2	225—250	20	Deeply coloured.
Manganese borate.....	0.5	1	230	20	Colourless.
Manganese acetate.....	.5	1½	225—250	20	Colourless.
Manganese oxalate.....	1.5	2½	230	36	Colourless.
Manganese sulphate .....	1.5	2½	240	36	Colourless.
Manganese oxalate.....	1.0	2½	..	48	Yellow in colour.
Manganese citrate .....	1.5	1½	230	24	Dark coloured.
Manganese tartrate .....	1.0	2½	230	24	Colourless.
Manganese formate .....	1.0	1	200	24	Somewhat coloured.

The oil after boiling was examined. For the determination of the lead a weighed amount of the oil was carbonised in a small evaporating dish and then ignited over a Bunsen lamp until all the carbon was burned off. The residue was digested with hot dilute  $\text{HNO}_3$ , filtered, the lead precipitated as sulphate, and weighed.

very similar results, but the third oil dried in half the time required by the fourth, although the latter contains more lead.

The following results were obtained in the case of the manganese salts:—

Boiled Oil.	Quantity of Dryer added.	Per Cent. of Lead in the Boiled Oil.		
		Calcu- lated.	Found.	
			I.	II.
Lead carbonate.....	1.197	2.03	1.392	1.477
Lead acetate.....	1.466	2.23	1.396	1.338
Litharge.....	0.200	0.406	0.244	..
Lead borate.....	1.105	2.03	0.982	0.998

Boiled Oil.	Quantity of Dryer added.	Per Cent. of Manganese in the Boiled Oil.		
		Calcu- lated.	Found.	
			I.	II.
Manganese sulphate ....	Grams. 1.72	1.37	0.045	0.038
Manganese borate.....	1.62	2.06	0.208	0.190
Manganese acetate.....	0.5	0.317	0.248	0.256
Manganese oxalate.....	1.5	1.034	..	0.030
Manganese tartrate.....	1.5	0.670	0.049	0.043

From the drying tests it will be seen that the first two oils in the above table dried in nearly the same time, and gave

The conclusions at which the author arrives are as follows:—

Lead dryers always give the oil a deep colour, which shows more or less in the film. Zinc dryers do not appear to act on the oil to any degree, as oils thus prepared dry slowly and do not give very hard films. Manganese dryers give the best results in all respects. Litharge gives the best results of the lead dryers, the oil being quick drying and the film hard, and if not overheated the oil is but moderately coloured. Of the zinc salts, the acetate seemed to give the best result, although the borate and citrate were nearly as good. The borate and acetate of manganese gave the best results. The acetate requires careful use, for, if heated much above 230°, it gives a deep colour to the oil, owing apparently to the formation of tar. The borate undoubtedly gives the best boiled oil for all purposes. The chlorides, nitrates, and sulphates do not make good dryers. The first two have too violent an action on the oil, while the last are very difficult to decompose, requiring a high temperature. There appears to be no advantage in the use of formates, citrates, or tartrates.—J. B. C.

#### PATENTS.

*Improvements in the Manufacture of White Lead, and in the Apparatus employed therefor.* W. Thompson, London. Eng. Pat. 4056, October 6, 1880. (Second Edition.) 11d.

THE inventor converts metallic or "blue" lead into white lead by exposure in a closed chamber to the combined action of acetic acid vapour and atmospheric air at a temperature of from 80°—120° F., for one-sixth to one-fourth of the time required for completing the conversion. Carbonic acid gas and air are subsequently supplied to the closed chamber, in addition to the acetic acid vapour.

—E. G. C.

*Improvements in the Production of White Lead or Carbonate of Lead.* R. W. E. MacIvor and F. A. Darlington, London; G. Paul, Edinburgh; and J. Allan, Harringay. Eng. Pat. 6815, April 23, 1889. 8d.

HYDRATE of lead is formed by the action of ammonium acetate on oxide of lead, and the hydrate is converted into carbonate by the injection of carbonic acid.—E. E. B.

*Improvements in and relating to the Manufacture of India-rubber or Waterproof Textile Fibres and Fabrics.* C. Dreyfus and J. Robinson, Manchester. Eng. Pat. 7090, April 29, 1889. 6d.

THE patentees ascribe the disagreeable smell of waterproofed fabrics to the use of solvent naphtha containing such impurities as naphthalene. (Ordinary solvent naphtha becomes dark brown or even nearly black when shaken with a quarter its volume of sulphuric acid at 168°—170° Tw., at the same time diminishing in bulk, proving that considerable quantities of impurities are present.) The use of pure hydrocarbons of the aromatic series, or of purified solvent naphtha, is claimed to obviate this objection.—B. L.

*Method of Preparing Iron Oxides from Ferrous Solutions.* A. J. Ramage, Widnes. Eng. Pat. 10,412, June 21, 1889. 4d.

THE ferrous solution is partially oxidised by a current of air, and lime added gradually. The ferrous-ferrie hydrate, consisting of about one-third of Fe<sub>2</sub>O<sub>3</sub> and two-thirds FeO, is filter-pressed and well washed, and then heated in a reverberatory furnace in a reducing flame. At the end of two hours it is drawn out, quenched in water, and ground. This method produces a scarlet oxide; for an Indian red, furnacing must be continued for about four hours.—F. H. L.

*Improvements in Lacquers and Varnishes.* I. M. Lamb and D. Boyde, Manchester. Eng. Pat. 10,428, June 27, 1889. 6d.

SUITABLE resins are dissolved in a solvent consisting of from 20 to 80 per cent. of fusel oil, mixed with a volatile hydrocarbon, preferably benzene or its homologues. The proportion of fusel oil is governed by the rapidity of drying desired; the smaller its proportion the faster the lacquer will dry. As an example of good lacquer the following proportions are given:—Gum damar, 1 lb.; gum benzoin, 6 oz.; solvent (as above) 6 lb.—F. H. L.

*Improvements in Preparing the Surface of Zinc Plates for Lithographic Purposes.* C. G. B. Brocklehurst, Bowdon. Eng. Pat. 12,130, July 1, 1889. 4d.

ZINC plates are faced with lead by treatment with a solution consisting of 1 oz. of sugar of lead,  $\frac{3}{4}$  oz. of nitric acid, and  $\frac{1}{2}$  pint of water, and can then be drawn or written upon by lithographic ink, and used for ordinary lithographic printing.—B. B.

*Improvements in Ink for Printing and like Purposes.* N. Browne, London. From I. Prowattain, Philadelphia, U.S.A. Eng. Pat. 15,839, October 9, 1889. 6d.

THE object of this invention is to replace the expensive oils usually employed in the manufacture of printing ink by the natural product "maltha" or semi-fluid bitumen. When applied to paper by means of type, &c., maltha has a rich dark golden colour, which imparts to bright-coloured inks a peculiar brilliancy and warmth of tone. The proportion of pigment employed varies with the shade of ink required, but with 20 per cent. lamp black a jet black is produced.

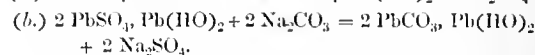
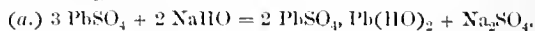
If the maltha be too thin the more volatile constituents may be removed by heat, by adding less fluid bitumen, or by the addition of powdered magnesite or resins.

For "news" inks, the maltha should have a specific gravity of from 30° to 36° B., with 20 per cent. pigment.

For "book" ink the gravity should be from 22° to 28° B., and for lithographic ink from 15° to 20° B., with 25 per cent. pigment. Only about one-half the amount of pigment used with ordinary linseed oils is necessary for mixing with the bitumen. Owing to the homogeneous character of maltha, less grinding than usual is required, and inks made therewith are practically indelible, no solvent being able to remove the impression entirely. The drying qualities are equal to the best commercial inks, and one grade of ink is equally suitable for different classes of work.—F. H. L.

*Improvements in the Manufacture of White Lead.* P. Brommer, Stuttgart, Germany. Eng. Pat. 16,706, October 22, 1889. 6d.

THIS method of manufacturing white lead is based upon the discovery made by the inventor that lead sulphate can be "very readily decomposed by heating it together with a certain quantity of soda-lye, if this quantity be determined so that the product will be, not hydrated oxide of lead, but a basic salt." If the compound formed be 2 PbSO<sub>4</sub>, Pb(HO)<sub>2</sub>, it can be at once transformed into excellent white lead, 2 PbCO<sub>3</sub>, Pb(HO)<sub>2</sub>, by heating with a suitable quantity of sodium carbonate. The by-product obtained is glauber salt. The equations representing the above changes are:—



By the improved process a very beautiful and extremely fine product is said to be obtained, having excellent covering power, while grinding and pounding are unnecessary. The cost of the process is also stated to compare favourably with that of the so-called stack or chamber process.—E. G. C.

*Improvements in Paints.* A. J. Boulton, London. From N. A. Bibikov, Albuquerque, U.S.A. Eng. Pat. 1684, January 31, 1890. 4d.

The object of this invention is to supersede the use of mineral or vegetable oils, glue, &c., in paints, by a material which is fire, water, and acid proof, and which is a non-conductor of electricity. Silicate of potash or soda is dissolved in six or eight parts of water and 25 per cent. of ground mica added. After the surplus water has evaporated a combination similar to oligoclase and andesite is formed, which may be used either alone or with various pigments.

For certain purposes 3—8 per cent. of lime may be added. —F. H. L.

*Process for the Production of Resin-acid Esters, and the Manufacture of Lacquers and Varnishes therefrom.* H. Beck, London. From E. Schaal, Germany. Eng. Pat. 2308, February 12, 1890. 4d.

ALL acid-resins may be converted into esters in the manner described in Eng. Pats. 12,807 of 1884 and 9027 of 1886 (this Journal, 1886, 523), or in the following manner.

When alcohols of high boiling point, phenols or hydrocarbons are employed, they may be allowed to flow into the melted resin-acids, the aqueous vapours being removed by means of a partial vacuum. For the conversion of the abietin acid glycerin-ester the following method is suitable. 100 kilos. rosin are heated in a copper still to from 180° to 210° C., and from 10 to 11 kilos. glycerin added in a thin stream with constant stirring, a vacuum of about 10 centimetres being maintained. When all the glycerin is added, the temperature is raised to 280° C. and the mass distilled, under as low a pressure as possible, for the purpose of separating the product into softer and harder portions.

To purify the crude esters they may be distilled in a current of benzene vapour, or in an atmosphere of carbonic acid or nitrogen. For the production of rapidly drying lacquers, from  $\frac{1}{2}$  to 2 per cent. of the ordinary lead or manganese dryers may be mixed with the esters at the end of the distillation and the mixture allowed to cool in thin layers. Suitable proportions for the preparation of such lacquers are: resin-acid ester 12 parts, turpentine oil 12 parts, linseed oil varnish 8 parts, the ester being dissolved at a temperature of 120° C.—F. H. L.

*Improvements in Apparatus for Vulcanising Dental Plates and other Rubber Articles.* C. A. Davis, Rochester, New York, U.S.A. Eng. Pat. 3399, March 4, 1890. 8d.

THE improvements consist in modifications of the boiler in which vulcanising flasks are heated. The boiler is carried by a flange resting on a ledge near the top of the stout casing or support which surrounds it, and is closed by a cover dependent from a yoke-piece by a screw passing vertically through the latter. A ball-and-socket joint at the point where the screw is attached to the cover allows it to bed fairly on to the faced top of the boiler. The yoke-piece swings in a horizontal plane, permitting the removal of the lid bodily from the boiler, making it easy to get at its contents or replace it altogether. The rubber in the flasks is subjected to an even pressure throughout the process of vulcanisation by a spiral spring confined between them and the lid of the boiler.—B. B.

#### XIV.—TANNING, LEATHER, GLUE, AND SIZE.

*Liquid Kino.* J. H. Maiden (Curator of the Technological Museum, Sydney). Proc. Roy. Soc. Victoria, 1890, 82—83.

*Angophora intermedia* (D.C.), the narrow-leaved apple-tree, is a tallish tree, which extends from Victoria to Queensland, and is the only species of the genus which is found in the southern colony. In the following respect it is perhaps unique amongst Australian trees. Frequently, when an

incision is made into the bark, and more particularly when the knobby excrescences sometimes found on this tree are cut, there exudes a watery liquid which occasionally is almost as clear and as colourless as water, and at other times of an orange-brown or reddish-brown colour, and of the consistency of a thin extract, or even as thick as treacle. This is doubtless the substance which was sent from New South Wales to the Paris Exhibition of 1867, labelled "apple-tree juice," with the statement that it is used as a varnish; but this is not correct, as the liquid is aqueous. It is used by fishermen for tanning their nets. Mr. Kirton informed Baron von Mueller that a single tree will yield as much as two gallons of liquid, which is generally called "liquid kino." This is a modest computation, for the tree which yielded the Bangle Creek sample (*infra*) yielded from 8 to 10 gallons. The quantity is in any case by no means small, and is dependent on a variety of circumstances.

Two samples of this "liquid kino" having recently been forwarded to the Technological Museum, the author has had an opportunity of examining it.

1. From Bangle Creek, Cambewarra, N.S.W., of a clear reddish-brown colour, and in order to give precision to the tint, it is very like raw linseed oil, Strasburg turpentine, or dark balsam of Copaiba, but redder than any of them. It has a specific gravity of 1·008 at 60° F., and an acridulous smell (owing to the presence of acetic acid), accompanied by an odour not so pleasant, and reminding one somewhat of spent tan liquors. It deposits a quantity of sediment of a buff colour, consisting almost entirely of catechol. It contains tannic acid 77·2 per cent., "non-tannin" 50·8 per cent. (Löwenthal's process). The water amounts to no less than 98·3 per cent. The catechol was not estimated in this sample.

2. This was obtained from Cambewarra, but from a different locality. It is darker in colour than the preceding sample, being of a rich ruby colour. Like No. 1, it deposits a small quantity of sediment (catechol). This liquid kino had a specific gravity of 1·022 at 60° F., when received in April 1888.

The following results were obtained in December to January 1889:—Tannic acid, 3·048 per cent. (of the liquid kino, without evaporating), "non-tannin" 1·27 per cent. (a portion of liquid kino, kept in agitation so as to obtain a fair proportion of sediment, was added to water to make up the strength of one grain of liquid kino to the litre), water 96·7 per cent. (after filtration from deposited catechol). The catechol and a little phlobaphene filtered off, were found to be in the proportion of 4·95 per cent. of the original liquid kino. Ether agitated with the filtrate took up 15 per cent., of which one-third was estimated to be catechol and the rest resin.

Mr. Kirton has recorded liquid kino from the Illawarra district of New South Wales, but since there appears to be no reason why it should be found in one colony more than another, it will doubtless also be obtainable in Victoria, most likely on application to fishermen.

#### XV.—AGRICULTURE, MANURES, Etc.

*Nitrogen: Its Uses and Sources in Agriculture.* C. M. Aikman. Reprint of Lecture before the "British Chemical Manure Manufacturers' Association," London.

1. *The Relation of Nitrogen in its Different Forms to Plant Life.*—Boussingault showed that free nitrogen was not used by plants, but he was directly controverted by the experiments of Ville. Lawes, Gilbert, and Pugh, however, confirmed Boussingault's results, and the question was considered as decided till recently, when it was again brought to the front by the experiments of Hellriegel and Wilfarth. From these later experiments, the author feels justified in concluding as follows: (a.) That leguminous plants can draw their nitrogen supplies from the free nitrogen of the air in a way not possible to other plants. (b.) That this

absorption of free nitrogen is not effected directly by the plant, but is the result, so to speak, of the joint action of certain micro-organisms present in certain soils and the plant itself. (c.) That this fixation is connected with the formation of minute tubercles on the roots of the plants of the leguminous class, and that these tubercles may be the home of the fixing organism. (d.) That these fixing micro-organisms are not present in all soils.

In 1876 Berthelot discovered that free nitrogen was being continually fixed by minute organisms in certain soils under the influence of silent electrical discharges. As much as 25 lb. per acre per annum may be so fixed. This fixation does not occur during the winter (compare Appendix).

Although plants, under favourable circumstances, may absorb ammonia and simple forms of organic nitrogen (e.g., urea), nitric acid must undoubtedly be considered as practically the final form of nitrogen for the plant, and into this all other nitrogen must be converted before it becomes available as plant food. Nitrification is, therefore, of the utmost importance. The nitrifying organism will only work in the upper layers of soil, where oxygen is sufficient. Warmth, moisture, organic matter (compare Appendix), lime, and certain other mineral matters are also necessary for its growth. Much organic matter, ferrous oxide, and sulphides, by appropriating oxygen, are fatal to nitrification.

2. *Natural Sources of Gain of Nitrogen.*—These are:—(a.) In the air; free nitrogen (available only in some cases), ammonium nitrate and nitrite, and ammonia; about 4 lb. of nitrogen per acre per annum, in the form of these compounds, are washed down in the rain, according to Warrington. (b.) In the soil; organic compounds containing nitrogen, ammonia, and nitrates; the organic nitrogen is rarely much less than 0.1 per cent. (= 3,500 lb. per acre to 1 ft. deep), and in rich soils may be more than double this amount; the ammonia rarely exceeds 0.0006 per cent.; the nitrates vary, as much as 55 lb. per acre have been found in the first 20 in. of cultivated soil left fallow.

3. *Natural Sources of Loss of Nitrogen.*—Drainage is the chief of these. Nitrates are incapable of being retained by the soil, and are rapidly washed out. The amount of loss may be imagined by the amount of nitrates removed by the rivers. According to Storer (Agricultural Chemistry, 1, 318) the river Rhine discharges daily 220 tons of saltpetre, the Seine 270 tons, and the Nile 1,100 tons (compare Appendix).

Loss of free nitrogen is constantly occurring where large quantities of organic matter decay and oxidation is incomplete, but it amounts to very little.

Retgression of the nitrogen of nitrates to an organic form by the agency of a denitrifying organism may take place; this would probably explain the action of nitrate of soda as a manure in the second year, an action which has been noticed in some cases.

4. *Artificial Sources of Supply and Loss of Nitrogen.*—(a.) *Losses.*—The following table shows, approximately, the amount of nitrogen removed from the soil by the total produce of wheat, barley, and oats in Great Britain according to the last agricultural returns:—

		Containing Nitrogen.	Equal to Sulphate of Ammonia.	Equal to Nitrate of Soda.
Wheat.....	Bushels. 76,224,940	Tons. 37,432	Tons. 176,465	Tons. 227,296
Barley.....	69,948,266	27,324	128,813	165,896
Oats.....	150,789,416	56,835	267,936	341,010
Total.....	296,962,622	121,591	573,214	734,172

It is impossible to estimate how much of this nitrogen is lost to the land. The straw, which is always returned to the land, contains less than one half the nitrogen in the grain. This loss is almost entirely due to our present system of disposal of sewage; taking the amount of nitrogen in the excreta of each individual as  $\frac{1}{2}$  oz. per diem (according to Heiden 1.7 lb. in the solid excreta and 9.7 lb. in the liquid excreta = 11.4 lb. per person per annum), 365,000,000 lb.

of nitrogen per annum will be voided by the entire population (in 1878) of the British Isles, "91,000,000" of which will be lost in London alone. The amount of nitrogen returned to the land as seaweed and fish is very small. Much free nitrogen has been shown to escape from sewage.

The nitrogen of explosive compounds is entirely lost, as it is ultimately converted into free nitrogen; it amounts to some 10,000,000 lb. per annum.

Loss of nitrogen in badly-kept farmyard manure undoubtedly occurs. In milk, sold off the farm, there is another loss of nitrogen. In the milk of a cow giving 2,000 quarts (American quarts, each of which = 57.75 cub. in.) per annum there are 22 lb. of nitrogen, the 2,450,444 cows in milk in Great Britain in 1888 would thus remove 54,000,000 lb. of nitrogen.

(b.) *Supplies.*—Our total consumption of bones is estimated at not less than 100,000 tons per annum; if the percentage of nitrogen in them be taken at 3—4, there will be 3,000—4,000 lb. of nitrogen applied in this form to the soil per annum. 60,000 out of the 100,000 tons are produced at home, and of this some 22,000 are obtained in and around London.

The following table gives the importation of guano into this country; the earlier shipments contained 13—14 per cent. nitrogen, but now they contain not more than 6 per cent. :—

PERUVIAN GUANO IMPORTED INTO UNITED KINGDOM.

Year.	Tons.	Year.	Tons.
1865	213,024	1880	58,631
1870	247,028	1881	33,333
1871	144,735	1882	27,382
1872	74,964	1883	36,713
1873	135,895	1884	15,802
1874	91,346	1885	..
1875	86,042	1886	28,733
1876	158,674	1887	5,784
1877	111,835	1888	16,446
1878	127,813	1889	17,000
1879	45,475		

*Nitrate of soda* is formed in beds at a depth of 10 in. to 16 ft. in layers 6 in. to 3 ft., often quite near the surface, but generally covered with a layer of "costra," a conglomerate rock. The crude stuff, known as "caliché," contains 20—60 per cent. of NaCl and other impurities. No beds occur nearer than 15 miles to the sea; the most distant are about 90 miles; the districts are rainless and without vegetation. They probably result from the decay of vast quantities of seaweed. Legrange estimates the quantity in the beds as some 100,000,000 tons. Nitrate was first exported in 1830, when only about 800 tons were sent out.

IMPORTS OF NITRATE OF SODA INTO THE UNITED KINGDOM, 1873—1889.

Year.	Tons.	Year.	Tons.
1873	124,000	1882	96,000
1874	108,200	1883	103,700
1875	161,900	1884	103,700
1876	160,800	1885	109,400
1877	69,600	1886	75,100
1878	104,400	1887	83,100
1879	55,500	1888	103,100
1880	48,300	1889	120,000
1881	54,800		

The above table represents the total imports; the quantity used last year in agriculture may be roughly estimated at 100,000 tons, which is less than some 15 years ago, the nitrate containing 15—16 per cent. of nitrogen.

*Sulphate of ammonia* has been rapidly increasing in production:—

SULPHATE OF AMMONIA PRODUCED IN THE UNITED KINGDOM FROM 1870 TO 1889.

Year.	Tons.	Year.	Tons.
1870	10,000	1880	60,000
1871	41,000	1881	65,000
1872	42,000	1882	72,000
1873	43,000	1883	75,000
1874	45,000	1884	87,000
1875	46,000	1885	97,000
1876	48,000	1886	106,500
1877	52,000	1887	113,700
1878	55,000	1888	122,800
1879	57,000	1889	132,000

80,000—90,000 tons are exported and 30,000—40,000 are consumed at home. The sources and respective quantities produced for the last four years are given below. (Compare L. Mond, this Journal, 1889, 506.)

	1886.	1887.	1888.	1889.
Gas works .....	82,500	85,000	93,000	100,000
Iron works .....	4,000	5,000	5,300	6,000
Shale works .....	18,000	21,000	22,000	23,000
Coke and carboursing works ..	2,000	2,700	2,500	3,000

*Fish guano*.—Some 5,000 tons containing about 9 per cent. nitrogen are probably annually imported, and about 10,000 tons containing about 8 per cent. nitrogen are produced at home.

*Meat meal and dried blood* are imported to the extent of about 5,000 tons, at 7—8 per cent. nitrogen, and about 10,000 tons at 12 per cent. nitrogen, are produced at home per annum. (Compare Appendix.)

The production of *shoddy* in this country is—

1,200 tons containing 8—12 per cent. nitrogen.
6,000 " " 6—8 " "
5,000 " " 5—6 " "

*Scutch*, the waste products of glue and leather factories, contains about 7 per cent. nitrogen.

*Horns, hair, bristles, and hoofs*, all of which contain about 14 per cent. of nitrogen, and *leather* are probably but little used.

*Oil-seeds and oil-cakes* for cattle feeding of course supply much nitrogen, the former containing about 3—4 per cent., and the latter 3—6 per cent. In 1889, 256,296 tons of oil-cake, about 370,000 tons of linseed, 80,000 tons of rapeseed, and 289,413 tons of cotton-seed were imported.

There were also used for cattle feeding in 1887: wheat, about 2,800,000 tons; maize, 1,560,000 tons; peas and beans, 870,000 tons; oats, 720,000 tons.

The *straw* imported in 1887 amounted to 52,393 tons; of *peat moss litter* there are no statistics.

In discussion the President (Mr. Voss) remarked on the decrease in the consumption of nitrogen in artificial manure during the last 15 years. In 1873 the total nitrogen used in the various manures mentioned in the paper amounted

to 34,000 tons; at present, however, only 28,000 tons are used. The great fluctuations in the prices of manures, and the increased use of cakes for cattle, explains this to a great extent. Dr. Sibson thought that the chief supply of ammonia in vegetation comes from the subsoil, where it is held absorbed by the clay, &c., having been collected and stored by the rain for thousands of years. Mr. Brown had tried experiments on the growth of leguminous plants and found that they grew well in the absence of any source of nitrogen except the atmosphere. Mr. Bernard Dyer pointed out that if the moderate dressings of  $\frac{3}{4}$  cwt. and 2 cwt. per acre of nitrate of soda were applied to the whole area of wheat and mangel crops respectively in the United Kingdom, about 350,000 tons of that manure would be used. He found it difficult to understand why the use of nitrogenous manures had fallen off; much land was undoubtedly under-farmed, owing to lack of instruction to the farmers.

An appendix contains letters which the author has received from several agricultural chemists. Sir J. B. Lawes estimates the loss of nitrogen on our soils at from 15 to 20 lb. per acre per annum. He is of opinion that the fact may be accepted that leguminous plants do, under certain conditions, obtain nitrogen from the atmosphere through the agency of germs; on the other hand, it is certain that these plants do take enormous quantities of nitrogen from the soil, so that the agricultural question becomes more complicated than it was before. There is no reason to think that the soil of the permanent wheat field at Rothamsted has obtained any appreciable amount of nitrogen from the atmosphere; but soils covered with vegetation undoubtedly do gain nitrogen slowly. Mr. Warington urges that Berthelot has not yet proved that a naked soil can gain nitrogen under the influence of electricity, but has simply shown that certain vegetable substances can do so, and has argued that soils should do the same. That free nitrogen is absorbed by an uncovered soil can hardly be concluded at present, for Schloesing has repeated Berthelot's experiments and found no evidence of such absorption. It is doubtful whether nitrification requires organic matter, and certain that it takes place in solutions to which no organic matter has been added. The loss of nitrogen by drainage in England is somewhere about 8 lb. per acre per annum. Dr. Stutzer thinks the estimate of nitrogen lost as nitrates in rivers, given in the paper, excessive. Messrs. R. D. Steele and Co. fix the imports of meat meal and dried blood at 2,500 tons per annum, and the home production at 7,000 tons. Ground hoof is a good deal used now. Fish guano should be estimated at 7,000—8,000 tons per annum. —A. G. B.

*New Experiments on the Question of the Fixation of Free Nitrogen.* Sir J. B. Lawes and Dr. J. H. Gilbert. Proc. Roy. Soc. 47, 85—118.

EXPERIMENTS similar to the well-known ones of Hellriegel, which were commenced in 1883, have been made by the authors at Rothamsted in 1888 and 1889. The results fully confirm Hellriegel's statements, and show large gains of nitrogen over that contained in seed and manure in many cases of leguminous plants grown in prepared sand or soil containing known percentages of nitrogen. The cases showing this luxuriant growth and increase in nitrogen were those in which root tubercles were well developed, and this was brought about by adding a little aqueous extract of the crushed tubercles to the prepared pots, or by watering them with the washings of soil in which similar leguminous crops, provided with root tubercles, had grown. The authors therefore are now prepared to endorse the conclusion drawn from Hellriegel's experiments "that although chlorophyllous plants may not directly utilise the free nitrogen of the air, some of them at any rate may acquire nitrogen brought into combination under the influence of lower organisms, the development of which is apparently, in some cases, a coincident of the growth of the higher plant whose nutrition they are to serve."—J. M. H. M.



*The Assimilation of Carbon by Green Plants from Certain Organic Compounds.* E. H. Acton. *Proc. Roy. Soc.* 47, 150—175.

THE observation of A. Meyer that starch is formed in the dark when solutions of glucose, glycerin, saccharon, or inulin are supplied to growing shoots, holds true when these solutions are supplied to roots or direct to the leaves. Starch is also formed when "soluble starch" is supplied direct to the leaves, but not when it is supplied to the roots, and when "humus extract" is supplied to roots, but not when supplied direct to leaves. It is not formed at all from acrolein or its compounds, allyl alcohol, dextrin, glycogen, aldehyde or its compounds, levulinic acid, or artificial humus made by action of alkalis on sugar. Glucose is more readily taken up from a 0.5 per cent. solution than saccharon. All the glucose can be withdrawn from a 1 per cent. solution if the roots are left in sufficiently long.

—J. M. H. M.

*Catalytic Formation of Ammonia from Nitrates.* O. Loew. *Ber.* 23, 673—680.

THE reduction of nitrates to ammonia is performed readily by moulds, and, as the author has proved, if ethyl alcohol (0.2 per cent.) be added, and the access of air cut off by means of a mercury joint, the reduction is carried on at the expense of the alcohol. The author submits that this result can only be explained if it is accepted that the reduction is caused by an atomic motion of the living protoplasm, that the dormant power of the living cell is converted into active, the atomic motion being transferred to the potassium nitrate as also to the alcohol.

In order to prove the correctness of this assumption, the author conducted a series of experiments with platinum black in place of the moulds. He holds that the oxygen of

the platinum black is not merely condensed on the surface of the platinum, but that the peculiar power it possesses is due rather to an atomic motion. In confirmation of this may be cited the fact that if oxygen be merely compressed it will not spontaneously oxidise alcohol and hydrogen.

Pure crystallised dextrose (5 grms.) and potassium nitrate (0.5 grm.) dissolved in water (100 cc.) were warmed to 60°—65° for six hours with 50 grms. of very active platinum black. The resulting acid liquid was filtered, concentrated, and treated with slight excess of milk of lime, which caused an immediate evolution of ammonia. Another, and at the same time quantitative experiment not only confirmed this result, but also showed that 45.6 per cent. of the nitrogen of the potassium nitrate was thus reduced to ammonia.

Experiments were also made to show that the reduction was not caused by any aldehydes, possibly formed from the dextrose; and these gave a negative result. Indeed, the reducing power of formic aldehyde was tried on nitrates and proved itself to be without any such power.

Other experiments, in which the platinum black was omitted, showed that it is undoubtedly the agent by means of which this reduction is brought about, and the author concludes that although the acids, chiefly gluconic and saccharic, are formed by the oxidation of the dextrose by the oxygen of the platinum black, yet another part of this causes a certain atomic motion in the nitrate and dextrose molecules resulting in the reduction of the nitrate.—J. W. L.

*Truffles.* A. Chatin. *Compt. Rend.* 110, 376—382; and 435—440.

THE author gives analyses of truffles from several localities, together with that of the soils in which they were found. In the following table the most complete analyses of the truffles are given. The localities are shown in the headings:—

	Saygnac les Eglises (Dordogne).	Souillac (Lot).	Dégagnac (Lot).	Chamont (Haute-Marne).	Dijon.	Tullins (Isère).
	Per Cent. 77.00	Per Cent. ..	Per Cent. 79.16	Per Cent. 75.74	Per Cent. 75	Per Cent. 75
Water .....	23.00	..	20.84	24.26	25	25
Dry organic matter .....						

#### COMPOSITION OF THE DRY MATTER.

Nitrogen .....	3.98	4.98	5.06	..	8.32	16.0
Organic matter less nitrogen .....	90.49	87.19	85.00	..	81.68	72.8
Ash .....	5.62	7.83	0.88	..	10.00	11.2

#### COMPOSITION OF THE ASH.

Phosphoric acid .....	21.65	30.25	21.17	18.45	18.90	23.15
Sulphuric acid .....	3.10	4.65	4.74	3.94	2.40	2.15
Chlorine and iodine .....	0.20	0.20	0.20	0.35	0.39	0.36
Lime .....	6.00	9.40	6.20	7.25	7.50	6.50
Magnesia .....	1.20	0.20	1.32	0.83	0.85	3.10
Potash .....	17.40	25.15	27.26	24.00	23.77	24.40
Soda .....	1.00	1.10	2.10	1.00	0.60	1.20
Forrie oxide, traces of alumina .....	3.80	3.20	4.40	4.00	7.50	8.40
Manganese oxide .....	0.05	Trace	Trace	Trace	Trace	Trace
Residue insoluble in acid (silica) .....	35.25	10.00	24.80	30.25	28.05	23.24
Carbonic acid and loss .....	10.35	8.85	8.01	9.83	10.04	7.50
	100.00	100.00	100.00	100.00	100.00	100.00

The trace of iodine is appreciable by comparison with titrated and coloured liquids at about  $\frac{1}{20}$  mgrm. (per 100 grms.?).—J. M. H. M.

*Absorption of Ammonia from the Air by Soil.* T. Schloesing. Compt. Rend. **110**, 429—434; 499—504.

FROM 1886—90 the author has made a number of experiments proving the absorption of ammonia from the air by soils of very different character, in quantities representing a gain of nitrogen to the soil of from 15 to 50 kilos. per hectare of surface per annum. The experiments were made by exposing known weights of soil in glass dishes in a wooden flue opening into a chimney through which a current of air was maintained by gas burners. The air was filtered before entering the flue through a sheet of perforated metal, to prevent access of birds, insects, &c.; and the amount of nitrogen carried in by fine dust was estimated by placing an empty jar of the same size as the others in front of them, and determining the nitrogen contained in the dust deposited in this jar at the termination of the experiment. The increase of ammonia absorbed, owing to the constant current maintained, over that which would have been observed with air subject only to the ordinary disturbance, was allowed for by exposing two vessels of dilute acid, with known surface, one inside the flue and subject to the current of air, and the other outside the apparatus altogether. Thus the nitrogen contained in the dust amounted to 1.9 kilos. per hectare per annum, and the extra ammonia due to agitation of the air amounted to from 0.534 to 0.637 of the net gains of the soils after deducting the 1.9 due to dust. After making allowance for both of these sources, the various soils gained, as stated above, nitrogen to the extent of 15—50 kilos. per hectare per annum. That this gain was not from free nitrogen brought into combination was shown by the fact that some of the soils experimented with were the very same samples with which the author has obtained so many negative results as to the fixation of free nitrogen, but still more conclusively by a check sample of soil, placed amongst the others, but fitted with a cover to prevent access of the current of air, the crown being pierced with a little hole so that ordinary diffusion could take place. This sample gained in 495 days only 0.5 mgrm. of nitrogen per 100 grms. of soil, whilst the other uncovered samples exposed with it gained from 3.6 to 12.9 mgrms. of nitrogen per 100 grms. The nitric nitrogen, ammoniacal nitrogen, and total nitrogen were determined in each sample of soil before and after the exposure. The absorption of ammonia is greatest when the soils are frequently moistened, because then the ammonia is nitrified as fast as it is absorbed, but it takes place even when the soils are kept so dry that no nitrification occurs. The absorption is shown by subsoils as well as surface soils, by soils poor and rich in nitrogen, and by those containing as much as 37 per cent. of calcium carbonate as well as by those nearly free from lime.

—J. M. H. M.

*A Coleopterous Insect attacking the Tunisian Vines.*

A. Laboulbène. Compt. Rend. **110**, 539—540.

AN addition to the 130 species of insect enemies of the vine figured in V. Mayet's "*Les Insectes de la Vigne*" (Paris, 1890). It proves to be *Ligniperda fransisca* (Fabricius), and attacks the pith of the stems after the harvest, in October, leaving hollow stems of bark. The shoots attacked should be cut off and burnt before the insects escape.

—J. M. H. M.

*Presence of Boric Acid in Plants.* E. Bechi.

Bull. Soc. Chim. 1890, **3**, 122.

WITH regard to the communications of Lippmann and of Crampton (this Journal, 1889, 569) relating to the presence of boron in plants, the author points out that he discovered this fact long ago in plants grown on Boroniferous

soils, and showed that the ash of beeches from Vallombrosa, Tuscany, contained as much as  $\frac{1}{100000}$ th of boric acid. He intends publishing his results.—D. A. L.

*Manurial Experiments with Beans.* H. Patensen. Hildesheimer landw. und forstw. Vereinsblatt, **29**, 85—89, and 95—98.

BEANS were grown on a heavy undrained loam, given to forming weather-pan, and in a level, moist situation; during the preceding years it had grown sugar beet with artificial manures, and the immediately preceding crop was oats with ammonia superphosphate and Chili saltpetre, which had yielded a very good harvest. These experiments were conducted to test the value of farmyard manure for beans. The farmyard manure employed contained per cent. N, 0.5;  $P_2O_5$ , 0.26;  $K_2SO_4$ , 0.63; CaO, 0.7, and the manuring and results were as follows:—

Manuring.	Average Yields from Two Sets of Experiments in Centners per Morgen.	
	Seed.	Straw, &c.
Unmanured .....	5.3	11.3
8 fuder (160 centners) farmyard manure..	11.5	18.0
3 ctrs. Thomas' powder and 2 ctrs. kainite	6.0	12.7
1 ctr. Chili saltpetre .....	5.1	11.1

The farmyard manure only, yielded a profit, which demonstrates its efficiency for the purpose. The partial manuring was not successful. The plants developed about the same on all the plots, but on the farmyard manure plots the beans were longer and the leaves were nearer together, although they were neither larger nor more numerous. The number of blossoms was about the same on all plots, but on the unmanured and mineral manured plots the blossoms to a large extent fell off without fertilising. Again, the mineral manured plots fell victims to the attacks of parasites (*Erysipe Martii*, *Peronospora viciae*, and *Uromyces fabae*), but the farmyard manure plots were free. The author points out that perhaps in many instances where failure of crops is attributed to these pests, it may be to some extent due to want of proper manure. The plants on all the plots had the healthy blue green colour, and showed no signs of want of nitrogen.—D. A. L.

*Hildesheim Manurial Experiments with Beet in 1889.*

K. Müller. Hildesheimer landw. w. forstw. Vereinsblatt, 1890, 2—5.

SINCE 1885, experiments have been conducted annually on various farms to ascertain the suitability of various manures for the Hildesheim soils. The reliable experiments last year gave the following results per morgen, taking the beet at a value of 1.25 marks, and Chili saltpetre at 10 marks per centner:—

Manuring.			Nett Profit Marks per Morgen.	Percentage of Sugar.
Centners Chili Saltpetre.	Pfund, $P_2O_5$ .			
	Soluble.	Thomas Slag.		
0	0	0	..	15.8
2	0	0	16.87	15.7
2	15	0	28.35	15.9
2	30	0	25.08	16.1
2	0	30	31.90	16.0
3	30	0	..	15.8

The results agree with those of the previous year in showing that, under the prevailing conditions, twice as much nitrogen as phosphoric acid produces a better result than applying twice as much phosphoric acid as nitrogen. In one case, Chili saltpetre alone produced no result, but yielded a great increase with 15 pfd. of phosphoric acid, and even in this case the use of 30 pfd. of phosphoric acid did not increase the profit. The Thomas slag acted well, although sown in the spring; better results would be obtained by sowing in the autumn. It is pointed out that since the commencement of the experiments Chili saltpetre has acted always in dry and in wet seasons, while the phosphoric acid was more active in wet than in dry seasons. The use of

potash in addition to the nitrogen and phosphoric acid gives a further increase in yield, although the nett profit is not so large, owing to the cost of potash.

Other experiments are in progress.—D. A. L.

*Results of Manurial Experiments on Mangels, Maize, and Meadow Grass.* J. Samek. Tiroler landw. Blatter, 1890, 42—43.

THE following are the results obtained with "mammoth" mangel on a tenacious deep soil derived from the weathering of clayey limestones and poor in potash; the second year after a liberal manuring with dung.

Manuring in Kilos. per Hectare.				Yield per Hectare, M.-Ctr.		Profit from Manuring.	
18 per cent. Superphosphate.	Ammonium Sulphate.	Chili Saltpetre.	48 per cent. Potassium Sulphate.	Roots.	Leaves.	Fl.	Kr.
0	0	0	0	320	54	..	..
400	0	0	0	428.8	44	38	5
400	115	0	0	512	58	63	7
400	0	150	0	563	65	92	72
400	115	0	75	537	71	70	9
400	0	150	75	572	72	89	24
400		220 blood meal		468	60	42	47

The nitrate was applied in two lots. The potash is necessary even if not profitable.

The results of growing maize on a shallow sandy medium soil rich in potash, consisting of Adige river alluvium, are given in the following table. The experiments were

made the second season after farmyard manure; the phosphates and potash were sown in the furrows before the seed, the nitrate half before the seed, half when banking up, as in the case of the mangels.

Manuring, kilos. per Hectare.				Yields.		Profit.			
Superphosphate.	Ammonium Sulphate.	Sodium Nitrate.	Potassium Sulphate.	Maize Grain per Hectare.	Hay M.-Ctr. per Hectare.	Maize.		Hay.	
						Fl.	Kr.	Fl.	Kr.
0	0	0	0	27.5	53.5	..	..	..	..
300	115	0	0	36.1	..	6	10	..	..
300	0	150	0	43.2	65.0	46	70	47	..
300	60	60	0	40	..	30	30	..	..
300	0	0	0	..	55.1	..	..	34	50
300	0	150	100	..	81.9	..	..	81	75

The meadow experiments were conducted on a shallow calcareous loam poor in potash, which in the previous year had been well manured with farmyard manure, and for which the present wet season was favourable. The herbage on the manured plots was chiefly leguminous, on the unmanured gramineous. Both the potash and phosphate acted well. Both on the roots and on the maize the nitrate proved better than the ammonium salts.—D. A. L.

*The Nitrifying Process and its Specific Ferment.* Percy F. Frankland and Grace C. Frankland. Proc. Roy. Soc. 47, (289) 296—298.

THE process of nitrification has been practically studied for centuries, but it was first in the year 1878 that it was shown by Schloesing and Müntz to be dependent upon the presence of certain minute forms of life, or micro-organisms, or in other words to be a fermentation change.

The authors have been engaged during the last three years in endeavouring to isolate the nitrifying organism,

and the present memoir gives in detail an account of the numerous experiments which were made in this direction.

Nitrification, having been in the first instance induced in a particular ammoniacal solution by means of a small quantity of garden soil, was carried on through 24 generations, a minute quantity on the point of a sterilised needle being introduced from one nitrifying solution to the other. From several of these generations, gelatin-plates were sown and the resulting colonies inoculated into identical ammoniacal solutions, to see if nitrification would ensue; but, although these experiments were repeated many times, on no occasion were they successful.

It appeared, therefore, that the nitrifying organism either refused to grow in gelatin, or that the authors had failed to find it, or that, growing in gelatin, it refused to nitrify after being passed through this medium.

Experiments were, therefore, commenced to endeavour to isolate the organism by the dilution method. For this purpose a number of series of dilutions were made by the addition to sterilised distilled water of a very small quantity



*The Reduction of Acids of the Sugar Group.* E. Fischer.  
Ber. **23**, 930—938.

THE reduction of oxy-acids rich in oxygen by means of sodium amalgam, which was described in a previous paper (this Journal, 1889, 994) furnishes a ready means for the synthesis of numerous sugars and allied bodies. Only those acids which form lactones are susceptible to this mode of reduction, and the ease with which reduction takes place seems to be proportional to the stability of the lactone. In fact, any body which prevents the formation of the lactone, also hinders the progress of the reduction. In the case of acids which form no lactones, reduction may, in some instances, be effected by employing an ether of the acid. The anides of oxy-acids are not affected by sodium amalgam. The mono-basic acids all yield sugars on reduction, whilst the di-basic acids are converted into aldehyde acids. In conjunction with Kiliani's reaction for the preparation of cyanogen addition compounds, this reduction process renders possible the building-up of sugars richer in carbon from other known sugars. For instance, from the carboxylic acids of mannose, galactose, and glucose, three well characterised sugars,  $C_7H_{14}O_6$ , are obtainable. By the addition of HCN, &c., they yield three acids,  $C_8H_{16}O_7$ , from which, by reduction, three sugars,  $C_8H_{16}O_6$ , may be prepared. The process is also applicable to those saccharoses which contain an aldehyde group. The author proposes to name the new sugars thus obtained, heptose, octose, nonnose, &c., their corresponding alcohols, heptite, octite, nonnite, &c., and so on. In order to indicate their origin, the name of the sugar from which they are derived is prefixed, thus: manno-heptose, manno octose, manno-heptite, gala-heptose, &c.

The following is a brief account of the results actually obtained by the reduction of sugar acids:

The reduction products of the three (*d*, *i*, and *l*) *mannonic acids* have already been described (this Journal, 1890, 527—528).

*Galactonic acid* yields galactose.

*Mannose carboxylic acid* yields, according to Passmore, *manno-heptose*, which is crystallisable and dextro-rotary. Its phenylhydrazone is difficultly soluble in cold water and melts at 188°—190° C. Its osazone forms fine yellow

needles, insoluble in water, melting at 198°—200° C. On further reduction the sugar is converted into an alcohol,  $C_7H_{16}O_7$ , which is identical with *Persëit*, for which Maquenne has established the same formula.

By means of the cyanogen reaction *manno-heptose* is convertible into *manno-octonic acid*, which forms a crystalline lactone melting at 167°—170° C., and possessing the optical activity,  $[\alpha]_D = -43.58^\circ$ .

Manno-octose, prepared from the acid, is dextro-rotary and forms a difficultly soluble phenylhydrazone melting at 212°—213° C.

*Glucose-heptose*, prepared from the corresponding acid, crystallises from water in beautiful plates, which melt at 190° C. with decomposition. Its phenylhydrazone is easily soluble in water, and its osazone forms golden yellow needles melting at 197° C. The sugar readily combines with HCN, and the *gluco-octonic acid* thus obtained yields a crystalline barium salt.

*Gala-heptose*, from *galactonic acid*, yields a difficultly soluble hydrazone, melting at 199° C. with decomposition. The osazone melts at 220° C. The sugar combines readily with HCN.

According to Piloty, *rhamnose carboxylic acid* yields on reduction *methyl-herose*,  $CH_3(CHOH)_5CHO$ , which melts at 180°—181° C. Its hydrazone is easily soluble. With HCN it forms *methyl-heptonic acid*, the lactone of which is crystalline. *Methyl-heptose* forms a difficultly soluble hydrazone.

*Levulose carboxylic acid* gives two peculiar sugars which are being further examined.

According to Reinbrecht, milk sugar yields with HCN an acid which has one carbon atom more than *lacto-bionic acid*. On reduction the acid is converted into a sugar probably possessing the formula  $C_{13}H_{24}O_{12}$ .

*Saccharic acid* by careful reduction is converted into an aldehyde acid, which has not been obtained in a crystalline state. It reduces Fehling's solution and is very similar in all respects to *glucuronic acid*.

*Mucic acid* is not itself reducible, but its ethyl ether is converted by sodium amalgam into an aldehyde acid, which forms a pale yellow syrup, reduces Fehling's solution, and on oxidation is converted into mucic acid.—H. T. P.

TABLE SHOWING THE EVAPORATION (PERCENTAGES BY WEIGHT) NECESSARY TO REDUCE A SUGAR SOLUTION FROM A LOWER TO A HIGHER DENSITY.—(G. L. Spencer.) *Spencer's Handbook for Sugar Manufacturers* (pp. 67—71.)

Initial Density.		35° Brix.	37° Brix.	39° Brix.	41° Brix.	43° Brix.	45° Brix.	47° Brix.	49° Brix.	51° Brix.	53° Brix.	55° Brix.	57° Brix.	59° Brix.
		19°6' Baumé.	20°7' Baumé.	21°8' Baumé.	22°46' Baumé.	23°45' Baumé.	25° Baumé.	26°1° Baumé.	27°2° Baumé.	28°2° Baumé.	29°3° Baumé.	30°4° Baumé.	31°4° Baumé.	32°5° Baumé.
Degrees Brix.	Degrees Baumé.	Per Cent.												
9.0	5.1	71.28	75.67	76.92	78.05	79.07	80.00	80.85	81.63	82.35	83.02	83.63	84.21	84.74
9.2	5.2	73.71	75.13	76.41	77.56	78.60	79.55	80.42	81.22	81.96	82.64	83.27	83.86	84.40
9.4	5.3	73.14	74.59	75.80	77.07	78.14	79.11	80.00	80.82	81.57	82.26	82.91	83.51	84.07
9.6	5.4	72.57	74.05	75.38	76.58	77.67	78.77	79.58	80.41	81.17	81.88	82.54	83.16	83.73
9.8	5.55	72.00	73.51	74.87	76.09	77.21	78.22	79.15	80.00	80.78	81.51	82.18	82.81	83.39
10.0	5.7	71.43	73.00	74.36	75.61	76.74	77.78	78.72	79.59	80.39	81.13	81.82	82.65	83.05
10.2	5.8	70.86	72.43	73.84	75.12	76.28	77.33	78.29	79.18	80.00	80.75	81.45	82.10	82.71
10.4	5.9	70.29	71.89	73.33	74.63	75.81	76.88	77.87	79.77	79.61	80.38	81.09	81.75	82.37
10.6	6.0	69.71	71.35	72.82	74.14	75.35	76.44	77.44	78.37	79.21	80.00	80.72	81.40	82.03
10.8	6.1	69.14	70.81	72.31	73.66	74.88	76.00	77.02	77.96	78.82	79.63	80.36	81.05	81.69
11.0	6.2	68.57	70.27	71.79	73.17	74.42	75.55	76.59	77.55	78.43	79.26	80.00	80.70	81.35
11.2	6.3	68.00	69.73	71.28	72.68	73.96	75.11	76.17	77.14	78.04	78.87	79.63	80.35	81.01
11.4	6.5	67.43	69.19	70.77	72.19	73.49	74.66	75.71	76.74	77.65	78.49	79.27	80.00	80.68
11.6	6.6	66.86	68.65	70.25	71.70	73.02	74.22	75.32	76.33	77.25	78.11	78.91	79.65	80.34

TABLE SHOWING THE EVAPORATION (PERCENTAGES BY WEIGHT) NECESSARY TO REDUCE A SUGAR SOLUTION FROM A LOWER TO A HIGHER DENSITY—*continued*.

Initial Density.		35° Brix.	37° Brix.	39° Brix.	41° Brix.	43° Brix.	45° Brix.	47° Brix.	49° Brix.	51° Brix.	53° Brix.	55° Brix.	57° Brix.	59° Brix.
		19°6' Baumé.	20°7' Baumé.	21°8' Baumé.	22°9' Baumé.	23°95' Baumé.	25° Baumé.	26°1' Baumé.	27°2' Baumé.	28°2' Baumé.	29°33' Baumé.	30°4' Baumé.	31°43' Baumé.	32°5' Baumé.
Degrees Brix.	Degrees Baumé.	Per Cent.												
11°8	6°7	66°29	68°11	69°71	71°22	72°56	73°77	74°89	75°92	76°86	77°73	78°55	79°29	80°00
<b>12°0</b>	6°8	65°71	67°57	69°23	70°73	72°09	73°33	74°47	75°51	76°47	77°36	78°18	78°95	79°66
12°2	6°9	65°14	67°03	68°72	70°24	71°63	72°88	74°04	75°10	76°08	76°97	77°82	78°59	79°32
12°4	7°0	64°57	66°59	68°20	69°75	71°16	72°44	73°61	74°69	75°69	76°60	77°45	78°24	78°98
12°6	7°1	64°00	66°05	67°49	69°26	70°69	72°00	73°19	74°29	75°29	76°22	77°09	77°89	78°64
12°8	7°2	63°43	65°51	67°18	68°78	70°23	71°55	72°76	73°85	74°90	75°85	76°73	77°54	78°30
<b>13°0</b>	7°4	62°86	64°86	66°06	68°29	69°76	71°11	72°34	73°47	74°51	75°46	76°36	77°19	77°97
13°2	7°5	62°29	64°32	66°15	67°80	69°30	70°66	71°91	73°06	74°12	75°09	76°00	76°84	77°63
13°4	7°6	61°71	63°88	65°04	67°31	68°83	70°22	71°49	72°65	73°73	74°71	75°64	76°49	77°29
13°6	7°7	61°14	63°34	65°13	66°83	68°37	69°78	71°06	72°25	73°33	74°34	75°27	76°14	76°95
13°8	7°8	60°57	62°80	64°61	66°34	67°99	69°33	70°64	71°84	72°94	73°96	74°91	75°79	76°61
<b>14°0</b>	7°9	60°00	62°16	64°10	65°85	67°44	68°88	70°21	71°43	72°55	73°58	74°54	75°43	76°27
14°2	8°0	59°43	61°62	63°59	65°36	66°97	68°44	69°78	71°02	72°15	73°20	74°18	75°08	75°93
14°4	8°1	58°86	61°08	63°08	64°88	66°51	68°00	69°36	70°61	71°76	72°83	73°82	74°73	75°59
14°6	8°3	58°29	60°54	62°56	64°39	66°05	67°55	68°93	70°20	71°37	72°45	73°45	74°38	75°25
14°8	8°4	57°71	60°00	62°05	63°90	65°58	67°11	68°51	69°80	70°98	72°07	73°09	74°03	74°91
<b>15°0</b>	8°5	57°14	59°46	61°54	63°41	65°12	66°66	68°09	69°39	70°59	71°61	72°72	73°68	74°58
15°2	8°55	56°57	58°92	61°02	62°93	64°65	66°44	67°66	68°78	70°19	71°32	72°36	73°33	74°24
15°4	8°7	56°00	58°38	60°51	62°44	64°18	65°77	67°23	68°57	69°89	70°94	72°00	72°98	73°90
15°6	8°8	55°43	57°84	60°00	61°95	63°72	65°33	66°80	68°16	69°41	70°56	71°64	72°63	73°56
15°8	8°9	54°86	57°30	59°49	61°46	63°25	64°88	66°38	67°75	69°01	70°19	71°27	72°28	73°22
<b>16°0</b>	8°0	54°29	56°75	58°97	60°97	62°79	64°44	65°96	67°35	68°62	69°81	70°91	71°93	72°88
16°2	9°2	53°71	56°21	58°46	60°48	62°32	64°00	65°53	66°94	68°23	69°43	70°54	71°58	72°54
16°4	9°3	53°14	55°77	57°95	60°00	61°86	63°55	65°17	66°53	67°81	69°06	70°18	71°23	72°20
16°6	9°4	52°57	55°17	57°44	59°51	61°39	63°11	64°68	66°12	67°45	68°68	69°82	70°88	71°86
16°8	9°5	52°00	54°59	56°93	59°02	60°93	62°66	64°25	65°71	67°06	68°30	69°45	70°52	71°52
<b>17°0</b>	9°6	51°43	54°05	56°41	58°53	60°46	62°22	63°83	65°30	66°66	67°92	69°09	70°17	71°18
17°2	9°7	50°86	53°51	55°90	58°05	60°00	61°77	63°40	64°90	66°27	67°54	68°73	69°82	70°85
17°4	9°8	50°29	52°97	55°39	57°56	59°53	61°33	62°98	64°49	65°88	67°17	68°36	69°47	70°51
17°6	9°9	49°71	52°43	54°88	57°07	59°07	60°88	62°51	64°08	65°49	66°79	68°00	69°12	70°17
17°8	10°0	49°14	51°89	54°36	56°58	58°60	60°44	62°13	63°67	65°10	66°41	67°63	68°77	69°83
<b>18°0</b>	10°1	48°57	51°35	53°81	56°09	58°14	60°00	61°70	63°26	64°70	66°03	67°27	68°42	69°49
18°2	10°3	48°00	50°81	53°33	55°61	57°67	59°55	61°27	62°86	64°31	65°66	66°91	68°07	69°15
18°4	10°4	47°43	50°27	52°82	55°12	57°21	59°11	60°85	62°45	63°92	65°28	66°55	67°72	68°81
18°6	10°5	46°86	49°73	52°31	54°63	56°74	58°66	60°42	62°04	63°53	64°99	66°18	67°37	68°47
18°8	10°6	46°29	49°19	51°80	54°14	56°28	58°22	60°00	61°63	63°13	64°53	65°82	67°01	68°13
<b>19°0</b>	10°7	45°71	48°65	51°28	53°66	55°81	57°88	59°57	61°22	62°75	64°15	65°45	66°66	67°80
19°2	10°8	45°14	48°11	50°77	53°17	55°35	57°33	59°15	60°81	62°35	63°77	65°09	66°31	67°46
19°4	10°9	44°57	47°57	50°26	52°68	54°88	56°88	58°72	60°40	61°96	63°39	64°72	65°96	67°12
19°6	11°1	44°00	47°03	49°74	52°29	54°42	56°44	58°29	60°00	61°57	63°02	64°36	65°61	66°78
19°8	11°2	43°43	46°49	49°23	51°70	53°95	56°00	57°87	59°56	61°17	62°64	64°00	65°26	66°44
<b>20°0</b>	11°3	42°86	45°95	48°72	51°22	53°48	55°55	57°44	59°18	60°78	62°25	63°63	64°91	66°10

Formula for calculating the above table:  
Degree Brix of Syrup = Initial Degree Brix + Degree Brix of Syrup  $\times 100$  = percent. water evaporated in terms of the weight of the original solution.

TABLE SHOWING THE EVAPORATION (PERCENTAGES BY VOLUME) NECESSARY TO REDUCE A SUGAR SOLUTION FROM A LOWER TO A HIGHER DENSITY.—(G. L. Spencer.)

(This table was originally constructed for the old Baumé scale, hence the odd numbers.)

Initial Density.			36°4° Brix. 20°4 Baumé. 1°1611 sp. gr.	38°3° Brix. 21°4 Baumé. 1°1707 sp. gr.	40°1° Brix. 22°4° Baumé. 1°1790 sp. gr.	42° Brix. 23°4 Baumé. 1°1898 sp. gr.	44° Brix. 24°5° Baumé. 1°2003 sp. gr.	45°8° Brix. 25°5° Baumé. 1°2039 sp. gr.	47°7° Brix. 26°5° Baumé. 1°2201 sp. gr.	49°6° Brix. 27°5° Baumé. 1°2305 sp. gr.	51°5° Brix. 28°5° Baumé. 1°2411 sp. gr.	53°5° Brix. 29°6° Baumé. 1°2523 sp. gr.	55°4° Brix. 30°6° Baumé. 1°2631 sp. gr.	57°3° Brix. 31°6° Baumé. 1°2740 sp. gr.
Degrees Brix.	Degrees Baumé.	Specific Gravity.	Per Cent.											
5.1	9.0	1.0359	77.94	79.21	80.30	81.34	82.34	83.17	83.97	84.72	85.41	86.08	86.62	87.22
5.3	9.3	1.0372	77.18	78.49	79.62	80.69	81.73	82.59	83.42	84.19	84.90	85.60	86.21	86.78
5.5	9.7	1.0388	76.16	77.53	78.71	79.84	80.93	81.82	82.69	83.41	84.24	84.96	85.60	86.20
5.7	10.1	1.0405	75.13	76.56	77.80	78.98	80.12	81.05	81.95	82.79	83.68	84.32	84.99	85.61
5.9	10.4	1.0418	74.12	75.84	77.11	78.32	79.49	80.45	81.39	82.25	83.05	83.83	84.52	85.16
6.1	10.8	1.0434	73.34	74.87	76.19	77.46	78.68	79.77	80.65	81.54	82.38	83.19	83.91	84.57
6.3	11.1	1.0447	72.56	74.14	75.50	76.80	78.05	79.07	80.08	81.00	81.86	82.70	83.43	84.12
6.5	11.5	1.0464	71.53	73.16	74.57	75.92	77.22	78.29	79.33	80.28	81.17	82.04	82.80	83.52
6.7	11.9	1.0481	70.49	72.19	73.65	75.04	76.38	77.49	78.56	79.56	80.50	81.38	82.17	82.91
6.9	12.2	1.0493	69.71	71.45	72.95	74.38	75.76	76.89	78.00	79.02	79.97	80.89	81.70	82.46
7.1	12.6	1.0510	68.67	70.47	72.02	73.50	74.92	76.10	77.24	78.30	79.28	80.23	81.07	81.85
7.3	12.9	1.0523	67.88	69.73	71.32	72.84	74.30	75.51	76.68	77.76	78.77	79.74	80.61	81.41
7.5	13.3	1.0540	66.83	68.74	70.38	71.96	73.47	74.71	75.92	77.04	78.08	79.09	79.98	80.81
7.75	13.7	1.0557	65.78	67.73	69.44	70.97	72.54	73.82	75.08	76.24	77.31	78.35	79.27	80.13
7.9	14.0	1.0570	64.99	67.00	68.73	70.40	71.98	73.30	74.58	75.76	76.86	77.92	78.86	79.74
8.1	14.4	1.0587	63.93	66.00	67.78	69.50	71.14	72.49	73.81	75.02	76.16	77.25	78.22	79.12
8.4	14.8	1.0604	62.86	65.09	66.84	68.60	70.29	71.68	73.04	74.29	75.46	76.58	77.58	78.51
8.5	15.1	1.0617	62.07	64.25	66.12	67.91	69.64	71.06	72.45	73.73	74.91	76.07	77.09	78.04
8.8	15.5	1.0634	61.00	63.24	65.17	67.01	68.79	70.25	71.67	72.99	74.21	75.40	76.44	77.42
8.9	15.8	1.0647	60.19	62.49	64.49	66.34	68.14	69.64	71.09	72.43	73.68	74.89	75.96	76.96
9.2	16.2	1.0665	59.12	61.47	63.49	65.44	67.30	68.83	70.32	71.70	72.97	74.22	75.32	76.38
9.4	16.6	1.0682	58.04	60.45	62.52	64.60	66.42	68.00	69.53	70.90	72.26	73.53	74.66	75.71
9.6	17.0	1.0700	56.96	59.44	61.55	63.59	65.56	67.17	68.74	70.20	71.54	72.85	74.01	75.08
9.8	17.4	1.0717	55.88	58.42	60.60	62.70	64.70	66.36	67.97	69.46	70.84	72.18	73.36	74.47
10.0	17.7	1.0730	55.06	57.64	59.85	61.91	64.06	65.72	67.36	68.88	70.29	71.65	72.86	73.98
10.1	18.0	1.0744	54.24	56.87	59.14	61.32	63.40	65.11	66.77	68.29	69.76	71.15	72.37	73.52

Formula for calculating the above table:  
 (Initial specific gravity × initial degree Brix) = (specific gravity of the syrup × degree Brix of the syrup) × 100 = per cent. syrup obtained.  
 100 - per cent. syrup = per cent. water evaporated.

## ERRATUM.

In the May number of this Journal, page 528, line 34 from top, for "König" read "Hönig."



## XVII.—BREWING, WINES, SPIRITS, Etc.

*Diminution of the Fermenting Power of Saccharomyces Ellipsoideus by Copper Salts.* A. Rommier. Compt. Rend. 110, 536—538.

THREE to four mgrms. of copper, added as sulphate to 40 cc. of sterilised grape juice seeded with pure cultures of *S. ellipsoideus* greatly retarded the development of the fermentation. As it is this ferment which communicates certain qualities to high-class vintages, the spraying of vines with copper preparations to prevent mildew may thus have an injurious influence.—J. M. H. M.

## XVIII.—CHEMISTRY OF FOODS, SANITARY CHEMISTRY, AND DISINFECTANTS.

### (A.)—CHEMISTRY OF FOOD.

#### PATENTS.

*Composition for Coating Coffee.* F. W. Moore, Wilmington, Delaware, U.S.A. Eng. Pat. 5033, March 22, 1889. (Internat. Conv. August 25, 1888.) 4d.

THE mixture consists of the following ingredients:—Milk or condensed milk, ground or powdered glue, "liquid glycerin," and refined lard, and in some cases bicarbonate of soda, fine table salt, and vinegar are also added.—F. W. T. K.

*Improvements in the Method of Preserving Milk.* A. Vászrhelyi and J. Jámhor, Budapest, Hungary. Eng. Pat. 6984, April 26, 1889. 8d.

FRESH and sound milk not later than one hour after milking is placed in jars made of a suitable material.

The jars are made in three parts. The bottom part, in which eventually the milk is preserved, is first filled; the other two parts together form what is called the mediator. This is screwed into the bottom can and acts as a filler. For this purpose, even after the bottom can is filled, the top filler is kept three parts full.

When a number of cans and mediators have been fixed in a tray they are filled and then the whole is lowered into water in a suitably arranged boiler. The milk is heated up to 76°—77° K. As soon as the mediator and can are full, by the milk expanding, a tap at the top of the mediator is turned and the whole is thus hermetically closed. The water in the boiler is then raised "to an intense heat, and this will keep the milk for another 50 minutes at a somewhat high temperature." The jars are next deposited upside down in a cooler, and left in this position for 60—80 minutes. This ensures the mixing of the milk, as it is in the nature of the milk that its fatty particles, and therefore the butter milk, will rise to the surface. The cans are finally placed in an upright position. The vacuum above the milk in the mediator is caused by the contraction of the milk. The air-tight stopper between the can or mediator is now turned, and as the ordinary temperature is rather higher, the can will be full, and there is little chance of the fat coagulating should the cans be shaken. The mediator is opened, and the milk in it having been run off, it is taken off and the process is complete. The milk is said not to lose its freshness, pureness, and sweetness even after 18 months or two years in hot countries, and when opened tastes like new milk, fresh and sweet.—F. W. T. K.

*Improved Apparatus for and Method of Purifying and Preserving Milk.* J. T. Appleberg, Knoxville, Tennessee, U.S.A. Eng. Pat. 1328, January 25, 1890. 8d.

THE apparatus consists of a box with hinged cover or lid and a hinged side. These can be closed air-tight. There is a central aperture in the lid which admits of the insertion

of a thermometer. The closed bottom of the box is provided with a grating or support on which rests a steam coil. As many of these boxes as may be found convenient may be connected with each other, either side by side, in tiers, or otherwise. The fresh milk, after straining, is poured into cans of the usual shape and construction. The lid of a can having been removed it is placed on the coil in a box and the lid and door of the box closed. The thermometer or indicator is then inserted through the aperture in the lid with its bulb projecting into the milk. Steam is then admitted into the coil and the temperature raised to 160° F. This initial heating is carried out rapidly so as to prevent separation of the milk. The temperature is then raised slowly to 185° or 190° F. (never to the boiling point). After the cans with their contents have been subjected to this treatment for a sufficient length of time, the steam is turned off, and the cans withdrawn and closed, after which they are ready (when cooled off sufficiently) for shipment and sale. The temperature of 185°—190° is sufficiently high to sterilise the milk, cans, and lids.—F. W. T. K.

*Improvements in the Manufacture of Butter and in Apparatus therefor.* F. R. C. Strüver, Pine Creek, Queensland. Eng. Pat. 1428, January 27, 1890. 8d.

THE cream from which the butter is to be made is enclosed in a bag made of a sheet or sheets of moleskin or other material which will allow of the escape of the butter milk but not of the cream. The mouth of the bag so formed is held tightly closed by means of a metal clasp or ring, specially devised for the purpose. When a number of these bags is ready, each is placed between suitably perforated plates (one above the other) enclosed in a perforated cylinder, and subjected to pressure for from 15 to 30 hours, according to the ripeness of the cream, by placing a heavy weight on the top of the whole.

The butter obtained is finally worked up by means of a butter-worker, the main portions of which are a cylinder, having inwardly projecting bars, and a central hollow spindle having radiating bars which can be made to revolve between the former. The butter-worker or the perforated cylinder which holds the bag is introduced into a vessel or cylinder which is covered with damask or other woollen material. This is kept damp and by the evaporation of the moisture the temperature of the whole apparatus is kept comparatively low. A low temperature is essential.

—F. W. T. K.

*Improvements in Apparatus for making Butter.* H. H. Lake, London. From C. H. F. Wilhelm Ove Ramel, Veile, Denmark. Eng. Pat. 2561, February 17, 1890. 6d.

TWO square frames, the corners of each of which are connected with each other by diagonal arms, serve as supports for eight vertical axes which are provided with beaters and which are set in motion by a tooth gearing, worked from a vertical central shaft, the central part of each frame forming a bearing for this shaft. Four vertical partitions in the apparatus serve to break the current of the milk. The whole is made of Britannia metal or some other suitable material, and is enclosed in a receptacle of sheet-metal provided a short distance from its bottom with a cock through which the butter milk is let out. With this apparatus the cream can be separated from the milk in about half a minute, and butter can be obtained in three minutes more.—F. W. T. K.

### (B.)—SANITARY CHEMISTRY.

*Recent Experiments on the Removal of Bacteria from Drinking Water by Continuous Filtration through Sand.* W. T. Sedgwick. Technology Quart. 3, 69—75.

THE present paper deals with the results of Dr. Fraenkel's investigations published in the *Deutsche Med. Wochenschr.* Dec. 12, 1889. The experimental filter beds consisted of barrel-shaped wooden tanks two metres high and 0.75 m. in average diameter, having at the bottom a sieve-like perforated support. This was filled precisely like the tanks

in actual use, *viz.*, with 150 mm. of stones of the size of the fist, 80 mm. of coarse gravel, 100 mm. of fine gravel and 600 mm. of finer sand. The surface was thus 1 m. below the top of the tank and was provided with an overflow. Special means were provided for avoiding sudden changes in pressure, so that the flow was perfectly regular and under control. Experiments were first begun with *Bacillus violaceus*, a harmless bacterium. It was cultivated in a very weak solution (1 part of ordinary bouillon in 20 parts of distilled water). Small doses of this fluid were added to the unfiltered water from time to time and usually at regular intervals of six hours. The supernatant water was then stirred in order to secure an even distribution of the bacillus all over the top of the tank. Samples of the water standing on the surface of the sand as well as of the effluent were examined bacteriologically every day from June 1st to the end of July. The results established the fact that, contrary to the prevailing opinion, the Berlin continuous sand filters are by no means germ-tight. The least efficient periods are those in which the filters are "young," *i.e.*, not yet covered on the top with a layer of organic material accumulated from the water previously passed through, or on the other hand "old," *i.e.*, so covered with organic matter that water passes through only under the compulsion of high pressure. It was observed that after prolonged running the number passed out increased somewhat even under no marked increase of pressure, and Fraenkel suggests that possibly some other cause than mere pressure is at work in such cases; such, for example, as the effect of the steady accumulation of food material within the tank, giving support finally to an undue number of bacteria in the lower layers. It appears that the number of germs in the effluent is directly dependent on the number in the unfiltered water. Moreover, the rate of flow seemed to be of the utmost significance. The filter which was run at the higher speed always showed the more bacteria, and, indeed, so uniformly that Fraenkel declares the results proportional. He lays down the rule that the qualitative efficiency of a continuous filter is inversely proportional to its quantitative efficiency. From the end of July to the middle of October Dr. Fraenkel experimented with the same filters as before, now using pathogenic germs. The results showed that the germ of cholera and typhoid may also pass through the sand filters. He points out, however, that the reduction in the number of germs by an effectively working and intelligently operated sand filter is astonishing; but any such system as is necessary for a large city requires the most skilful and intelligent supervision if the results are to be of the best. The rate of filtration is also, obviously, extremely important, and requires constant attention. If it be allowed that the germs may multiply in the filter, or that they are hardy—as increasing evidence seems to be indicating in the case of typhoid fever—the way is at once open for filters to become breeding places as well as passages for germs. For sewage disposal, continuous filtration fails utterly, since there is no free oxygen in sewage, and hence, no nitrification or other purification except the purely mechanical effect of the straining out of suspended matters, which lead to speedy stoppage of the filter. In drinking-waters there is an abundance of free oxygen, so that continuous filtration might, theoretically, allow the complete nitrification of the suspended matter mechanically held back. That this does not take place is probably due partly to the fact that the arrival of fresh material for oxidation takes place more rapidly than its removal by nitrification, and partly because nitrification appears to require a more or less prolonged contact between bacteria and the substances to be oxidised. It appears, therefore, that in the case of drinking water rich in oxygen, as well as of sewage destitute of oxygen, the process of continuous filtration is effective chiefly in the removal of suspended matters, and of these the finest particles (bacteria) are imperfectly separated. Intermittent filtration, on the other hand, readily effects the nitrification of the organic matters in sewage, by allowing the entrance of oxygen into the sand between the flooding periods and the purification of drinking-water by similar oxidation of the matters mechanically held back.—J. B. C.

*Experiments on the Efficacy of Filtration through Sand.*  
C. Fränkel and C. Piefke. *Zeits. für Hygiene*, 8, 1890, 1; and *Proc. Inst. Civil Eng.* 1890, 37—40.

IN the early months of 1889, Berlin was visited by an epidemic of typhoid fever which extended over a wide area, and which, from the middle of January to the middle of April, caused some seven hundred cases to come under the notice of the authorities. By reference to a map it is shown that this outbreak must have been wholly unconnected with the levels of the sub-soil water, as in lieu of extending over a district parallel with the course of the river, which would follow the lowest levels of the sub-soil water, it took a direction exactly the opposite, and attacked the western portions of the town, leaving the east end of Berlin untouched. For this reason the water-supply became suspected, and Virchow, at the meeting of the Berlin Medical Society on the 19th June, pointed to this as a possible cause of the epidemic. The authors mention several matters which may have contributed to the evil. The waterworks near the Stralau Gate possesses eight open and three covered filtering-beds. During the long-continued frosts of the winter months the open basins become frozen over, and in time become useless, so that the whole of the effective filtration has to be carried out in the three covered basins, which in consequence are worked at far too great a pressure. Thus, in February last, a rise in working speed took place of from 130 millimetres to 160 millimetres per hour, and on the 12th of March the maximum rate of filtration of 224 millimetres per hour was attained. The formation of a layer of bacteria on the surface of the sand is found to greatly retard the rate of filtration, and when the head of water is increased, it tends to drive the micro-organisms through the pores of the filtering material, and ultimately into the filtered water. This state of things is, of course, attained all the more quickly when the water to be filtered is very impure. By reference to a graphic diagram, the numbers of germs present per cubic centimetre of filtered water during the period in question are indicated. These rose rapidly from about one hundred in the middle of January to over ten times the amount in February, and attained, during March, the enormous total of upwards of four thousand per cubic centimetre. At the beginning of April, when the whole of the filter-beds were again in operation, the numbers of germs at once sank to the normal level. During these months the waters of the Spree were highly charged with bacteria, the numbers of germs, from January to March, often exceeding one hundred thousand per cubic centimetre. Though the presence of typhoid germs has not been demonstrated therein, the water supply of Berlin was manifestly extremely impure.

Much suspicion was expressed respecting this state of things, and the authors were commissioned by Dr. Koeh to investigate whether it might be possible to spread the germs of infectious diseases through the mains of the water company. For this purpose they undertook a series of experiments, all the necessary assistance being given to them by the company. It has hitherto been assumed by experts that the sand filter, carefully and properly managed, yields a germ-free, hygienically perfect filtrate. If this theory were thoroughly sound, investigations of the character of those here recorded would have been useless, but it soon became evident that complete experiments touching the fate of pathogenic micro-organisms passing through the sand filter had not hitherto been attempted. It was decided to employ small sections of the actual filter-beds, the apparatus being explained by reference to a diagram. Two tubs were used, each 2.1 metres (6.7 ft.) high and 0.75 metre (2.5 ft.) in mean diameter, at the base of which was a sieve-like collecting channel. The filtering material was disposed in these tubs in the same thicknesses as in the actual sections of the beds; *viz.*, 100 millimetres (3.9 in.) of pebbles of the size of hazel-nuts, 80 millimetres (3.1 in.) of coarse gravel, 100 millimetres (3.9 in.) of fine gravel, and 600 millimetres (2 ft. nearly) of fine sand. The surface of this sand was about 1.2 metre (4 ft.) below the top of the vessel, which was provided with an overflow-pipe.

The outlet-pipe from the filters was curved upwards to above the level of the sand, and the inlet-pipe was adjusted

to supply the water at any given rate. It was thus possible to work the filters at the required speed, and to avoid all disturbance of the contents. Having completed all these preparations, and formed two distinct filters, the authors were enabled to commence their observations, and they deemed it advisable to study, in the first instance, the behaviour of a bacillus of a non-dangerous character and of strongly marked features, and for this purpose the *bacillus violaceus* was selected. It was cultivated in very dilute nutritive bouillon, so as to render it impossible for the bacteria, when they were exposed to the still further dilution in the filter, to find the requisite supplies of food-stuff. This liquid, which had a pale bluish tint, was added every six hours to the contents of the unfiltered-water reservoir, in quantities of about 100 cubic centimetres at a time. Samples were taken daily for bacteriological examination, both of the unfiltered and of the filtered liquids, and they were cultivated on gelatin plates in the usual way. The filters were worked, the one at the rate of 300 millimetres (11·8 in.), the other at the rate of 100 millimetres per hour. The results are set forth in tabulated form, and prove that, throughout the whole period of observation, the bacteria passed through the filter, the numbers being in general accordance with the rate of filtration. About three times as many colonies were formed in the former filtrate as there were in the latter. When the speed of filtration was reduced in the second filter to 50 millimetres per hour, the relative abundance of the colonies of blue bacteria was about as 6 to 1. The proportion of the blue bacteria in the unfiltered water to those in the filtrate was, roughly speaking, 1,000 to 1. The slower rate of filtration produced, unquestionably, the better results. Similar experiments with cholera and typhoid fever bacteria gave corresponding results, which are set forth in tables, and the authors conclude that the sand filter is not a germ-proof apparatus, and is incapable of retaining the spores of the ordinary water-bacteria, as also those of a pathogenic character. The numbers of germs passing through the filter are in proportion to those in the unfiltered liquid, and in accordance with the speed of filtration. Both at the beginning and at the end of each periodic use of the filter, there is an interval of danger when the germs pass through the beds in greater abundance (see this Journal, 1889, 998).

*On the Passage of the Spores of Fungi and of Bacteria through Cloth used for Air-Filters.* K. Möller. Zeits. für Hygiene, 7, 1889, 379; Proc. Inst. Civil Eng. 1890, 40.

The author combats the conclusions of Dr. Petri on the above subject, and, as a member of the firm engaged in the production of air-filters, and as the first who undertook to filter air upon a commercial scale for industrial purposes, he points out certain mis-statements and erroneous deductions.

Air-filters are of two kinds; and for "germ-proof filters" he employs an ante-filter and ten layers of a specially thick, closely woven, and very much roughened material; for "dust-proof filters," only one thickness of a much less closely-woven fabric is used. Filters of the latter kind, though capable of excluding very large proportions of germs, are obviously not germ-proof. By the author's arrangement of the filter in the form of a series of pockets, the cloth moreover is used in a direction very oblique to the air-current, forming an angle with it of from 3° to 5°, whereas Dr. Petri tested the cloth at right angles to the air-current. By reference to enlarged sections of the fabric, the vast difference in the degree of permeability due to this change in the direction of the movement of germs is explained. It is shown also that Dr. Petri considerably strained the filter-cloth by subjecting it to the passage of abnormal volumes of air. Moreover, the author asserts that a woolly surface is one capable of entangling large numbers of germs, and that Dr. Petri actually neglected the obvious precaution of sterilising his cloth before the commencement of his experiments. He thus, doubtless, was led to regard a vast number of germs as having passed through the cloth, which, as a matter of fact, had been simply blown off its surface

by the air-current. The experiments of Dr. Petri are discussed in detail, and certain of them, for various reasons, are shown to be untrustworthy.

*On the Disinfection of Latrines by means of Lime.* E. Pfuhl. Zeits. für Hygiene, 7, 1889, 363; Proc. Inst. Civil Eng. 1890, 41.

HAVING by previous experiments demonstrated that the excreta of typhoid and cholera patients could be disinfected by means of relatively small quantities of milk of lime, the author resolved to investigate further whether the experience gained in the laboratory could be used in actual practice for the disinfection of fecal matters in tubs, cesspools, and night-stools. Lime can be procured on a commercial scale in such a pure state, as only to contain 1 per cent. of foreign matters, as has been found in the case of Wiesbaden, where from 2,000 to 3,000 kilogrammes of lime are used every 24 hours for the treatment of the sewage-water. The precautions to be taken in slacking and preparing the milk of lime are described. It is advisable to use double the quantity of water theoretically needed, viz., 60 parts of water to 100 parts of quicklime. It may be assumed with sufficient accuracy that 1 litre of slaked lime weighs  $\frac{1}{2}$  kilogramme; and in preparing a mixture of lime for use as a disinfectant, 1 litre of slaked lime is to be mixed with 4 litres of water. To test the action of the lime on the fecal matters, the author advocates the use of red litmus paper, coloured blue to various shades by solutions of lime of different known strengths. For cesspool-matters, as the result of a series of experiments on a cesspit of large size, details of which are given, as also full particulars of trials carried out on tubs used daily by soldiers in a barracks, the author recommends the use of from 1 to 1½ litre of slaked lime to each 100 litres of excreta contributed daily. The larger quantity being that advocated for tubs or pails. As the results of actual measurement, the daily increment of cesspool contents was found to be 400 cubic centimetres per head. The disinfection can best be effected when carried out daily. No reliance can be placed on any hand-mixing of the excreta with the lime. There is, therefore, no other course than to depend upon automatic admixture, or to arrange for some system of mechanical stirring of the cesspool contents, such as is accomplished by the apparatus of Thiriart, which is described.

(C)—DISINFECTANTS.

*The Action of Hydrogen Peroxide upon Infusoria.* J. Paneth. Chem. Centr. 1890, 174; and Proc. Inst. Civil Eng. 1890, 43.

THE infusoria with which these experiments were made were produced by inoculating a hay infusion with stagnant water and mud. The liquid thus obtained contained at least six different varieties of infusoria. The hydrogen peroxide was used in the form of a carefully neutralised solution of known strength. When the solution contained one part of hydrogen peroxide in 10,000 parts of water all ciliate infusoria were invariably killed in one quarter to half an hour. With a dilution of one in 20,000, parts of the organisms were destroyed, but some survived. The hydrogen peroxide was decomposed, either through the substances dissolved in the infusion or more probably by a catalytic action of the organisms. Hydrogen peroxide is therefore a violent poison for some forms of animal protoplasm. The author concludes from his experiments that the oxidising effect of such infusoria cannot be due to the presence of hydrogen peroxide in their cells, and from other experiments he is of opinion that they do not contain active oxygen to any considerable amount.

*The Disinfecting Action of Chloride of Lime.* F. Nissen. Zeits. für Hygiene, 8, 1890, 62; and Proc. Inst. Civil Eng. 1890, 42.

THE first bacteriological tests of the action of chloride of lime were given in Koch's work "On Disinfection," and it was shown therein that anthrax spores, dried on to silk

threads, were destroyed by a five days' exposure to the action of a 5 per cent. solution of the salt. Subsequently Sternberg and Jaeger have published the results of observations which differ in certain indicated particulars from those of Koch, and it therefore appeared advisable to undertake a fresh inquiry, which the author has carried out under the guidance of Dr. Koch. Samples of chloride of lime were procured from various dealers, and in all cases the material used was taken from the bottom of the jar or cask. The disinfecting action was tested by exposing the bacilli of typhoid fever, Asiatic cholera, anthrax, and two other species, immersed in alkaline bouillon, containing 1 per cent. of peptone and 0.5 per cent. of common salt, to equal volumes of the solution of chloride of lime. The liquids were well shaken, and samples, withdrawn after the lapse of 1, 5, 10, 15, and 20 minutes, and at longer intervals, were submitted to cultivation. The results of the observations are set forth in a series of tables, as also those of a further set of experiments directed to test the deadening powers, as respects the development of micro-organisms, of the chloride solution. In this latter respect, the influence of calcium chloride is insignificant. A third series of experiments was carried out with polluted liquids and human excreta, and from these it was ascertained that a 5 per cent. solution was capable of destroying the germs present in diarrhetic faeces within less than 10 minutes. From the first set of tests it was shown that the bacilli of cholera, typhoid fever, and anthrax\* were destroyed by a solution containing not less than 0.12 per cent. of chloride of lime in one minute. The germs of *staphylococcus* and *streptococcus* were rendered incapable of further development by exposure for one minute to a 0.2 per cent. solution of the chloride in bouillon.

#### PATENTS.

*Improvements in the Manufacture or Preparation of Disinfectants or Antiseptics.* W. Black, Newcastle, and W. L. Rennoldson, Jarrow. Eng. Pat. 8527, May 22, 1889. 4d.

In a previous patent (Eng. Pat. 1969, of 1888; this Journal, 1889, 131) the manufacture of antiseptics is described, wherein perchloride of mercury is incorporated with glauber salt or equivalent absorbing salt. The efficiency of the disinfectant or antiseptic is now further enhanced by the further incorporation of free sulphuric or hydrochloric acid, which makes the action of the perchloride more energetic, as well as of eucalyptus oil or thymol or some similar disinfecting oil or essence. The proportions preferred are: Perchloride of mercury four parts by weight, sulphuric or hydrochloric acid four parts, eucalyptus oil (or its equivalent) one part, indigo (or its equivalent) one part, and glauber's salt (sulphate of soda), either anhydrous or crystallised, or both together, 90 parts, by weight respectively. The colouring matter is merely added for the sake of convenience, as it is an advantage in practice to have the material or its solution of a distinct colour. The disinfectant is manufactured in the form of a liquid or solution or in a dry state, and made up, if so desired, in impervious packets containing any proper quantities for use.

—F. W. T. K.

*A New Method of Producing and Liberating Aërial or Gaseous Disinfectants.* G. T. Moody, F. W. Streetfield, London. Eng. Pat. 19,309, December 2, 1889. 4d.

This invention consists in the production of free haloids for disinfection, inhalation, and similar purposes by incorporating a certain quantity of the haloid compound with a convenient medium such as paraffin, wax, fats, oils, and other combustible substances, and making candles, tapers, night lights,

matches, or pastilles out of the mixture or using it in lamps. Iodoform, bromonaphthalene, and chloronaphthalene are stable and give very satisfactory results, but the patentees do not restrict themselves to the use of these halogen compounds. On combustion a considerable portion of the halogen is liberated in the free state. For general hygienic purposes combustible material containing a quantity of haloid compound equal to 1 or 2 per cent. of the free haloid is employed. The amount of halogen evolved can be regulated with great exactness by dissolving a known weight of the halogen compound in the combustible and by burning the candle, &c. for a definite time.—F. W. T. K.

*Improvements in Toilet or Sanitary Paper.* R. Hannan, Glasgow. Eng. Pat. 19,638, December 6, 1889. 4d.

To ensure the sanitary treatment of drains, closets, &c., the toilet paper is impregnated with antiseptic substances which have no injurious effect on the human body. Any ordinary variety of soluble antiseptic matter may be employed, such as carbolate of soda, sulphate of zinc, and the like. A mixture of boracic acid with borax to which a small proportion (say one-fortieth of its weight) of salicylate of sodium or other soluble salicylate has been added, is preferred. The liquid or dissolved antiseptic is either added to the pulp, or the finished tissue paper is run through a trough containing the solution, or is passed through a spray of the antiseptic and then dried.—F. W. T. K.

## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

*Synthesis of Alcohol Acids of the Pyridine Series.*

A. Einhorn. Ber. 23, 219—224.

SINCE cocaine has been found to be a derivative of  $\alpha$ -pyridyl- $\beta$ -lactic acid, the author has paid particular attention to the condensation product prepared from  $\alpha$ -picoline and chloral, which was discovered by him in conjunction with A. Liebrecht (Ber. 20, 1592).

The following bodies were obtained from  $\omega$ -trichlor- $\alpha$ -hydroxypropylpyridine as raw material. By treatment with phosphorus pentachloride the corresponding hydrocarbon pyridyl- $\omega$ -trichlorpropylene,  $C_5H_4CH:CH.CCl_3$ , was produced with elimination of water, forming prismatic needles, melting at 97°. By means of a boiling solution of caustic soda the raw material was converted into pyridyl- $\alpha$ -lactic acid,  $C_5H_4N.CH_2.CHOH.CO_2H$ , and thereby converting the group  $CCl_3$  into  $COOH$ . A particularly characteristic copper salt of the acid was prepared. The free pyridyl- $\alpha$ -lactic acid, which represents the carbon acid of  $\alpha$ -picolyl-alkine of Ladenburg (Ber. 22, 2584; this Journal, 1890, 100), and crystallises from alcohol in transparent prisms, melting between 202°—204° C. Methyl and ethyl ethers, as well as platinum and gold double salts of the were acid, obtained.

Pyridylacrylic acid (m.p. 202°—203° C.) resulted from the treatment with alcoholic potash, which does not only eliminate the chlorine from the  $\omega$ -trichlor- $\alpha$ -hydroxypropylpyridine, but at the same time splits off water and also forms some pyridyl- $\alpha$ -lactic acid. A number of ethers and double compounds of this acid were also prepared.

Pyridyl- $\beta$ -lactic acid,  $C_5H_4N.CH_2.CHOH.CO_2H$ . This acid is obtained along with pyridine-ethylene,  $C_5H_4N$ , (Ladenburg's vinylpyridine, Ber. 22, 2583; this Journal, 1890, 99), and undecomposed pyridyl-acrylic acid, by heating the latter compound with anhydrous acetic acid in a sealed tube to 100° C. The hydrobromide of pyridyl- $\beta$ -bromopropionic acid, which thus results, is carefully

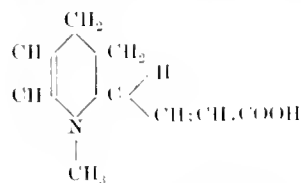
\* In a note appended by the author he states that some anthrax spores recently received, with such high powers of endurance as to be capable of resisting a four hours' exposure to a 0.1 per cent. solution of corrosive sublimate had succumbed in 4½ hours to a 5 per cent. solution of chloride of lime.

decomposed by soda, yielding above bodies. The free pyridyl- $\beta$ -lactic acid is extremely soluble in water, and forms a beautiful copper salt, the hydrochloride crystallises from alcohol in white prisms, melting at  $145^{\circ}$ — $146^{\circ}$  C.

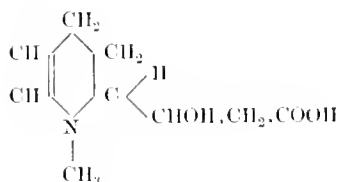
Pyridylglyceric acid,  $C_5H_5N \cdot CHOH \cdot COOH$ , is formed by oxidation of the sodium salt of the acrylic compound

with potassium permanganate; the hydrochloride, which contains one molecule of water of crystallisation, melts at  $189^{\circ}$ — $190^{\circ}$  C.

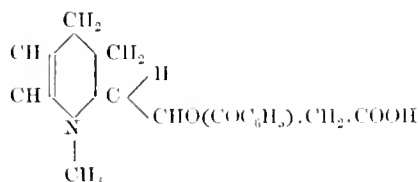
The importance of the relation which exists between cocaine and its compounds and the bodies prepared by the author may be seen from the following table:—



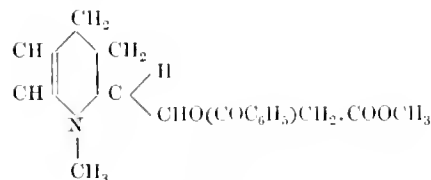
Anhydro-cocaine.



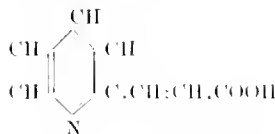
Ecgonine.



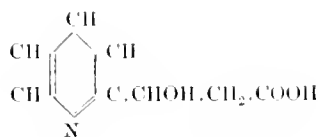
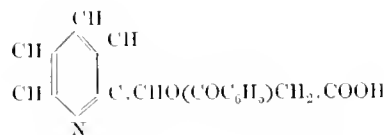
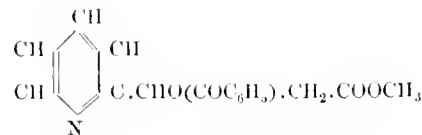
Benzoyl-ecgonine.



Cocaine.



Pyridylacrylic acid.

Pyridyl- $\beta$ -lactic acid.Benzoyl-pyridyl- $\beta$ -lactic acid.Benzoyl-pyridyl- $\beta$ -lactic methylate.

—O. J. S.

On *Apiol*. J. Ginsberg. Ber. 23, 323—325.

REFERRING to previous investigations on the subject (Ber. 21, 2516; this Journal, 1888, 399 and 583; 1889, 1007), author gives further particulars of an acid obtained by the action of sulphuric acid and manganese dioxide on iso-apiol, to which he gave the name *aponic acid*.

The yield of the acid being a very bad one, author, after trying various methods, found the following to be the best. A mixture of 240 grms. of water, 60 grms. of sulphuric acid, and 6 grms. of iso-apiol are brought to the boil, and 60 grms. of commercial manganese dioxide gradually added, and boiling continued for four hours. On extraction with ether, evaporation of the latter, conversion into ammonium salt and decomposition with hydrochloric acid, the free acid is obtained from water in small colourless needles, melting at  $252^{\circ}$  without decomposition. Apiol treated in the same manner does not yield an oxidation product. From the formula obtained,  $C_{14}H_{10}O_3$ , aponic acid might be identical with one of the oxybenzoic acids, which however is not the case, the formula, therefore, may also be  $C_{14}O_{12}O_6$  or  $C_{14}H_{10}O_6$ .

Iso-apiol tested after Zeisel's test proved to contain two  $(OCH_3)$  groups, whereas aponic acid does not contain any. Aponic acid is an extremely stable compound, which is not acted upon by hydrochloric or bromic acid under pressure,

and is not even attacked by a mixture of nitric and sulphuric acid. Phenylhydrazine nor hydroxylamine do form a condensation product with the acid. The calcium, silver, and barium salts were also prepared, the latter for instance having the formula  $C_{14}H_{10}O_6Ba$  or  $C_{14}H_8O_6Ba$ . The silver salt and ethyl iodide yielded an ethyl ether.—O. J. S.

Some Derivatives of *Cantharidine*. F. Anderlini.

Ber. 23, 485—486.

THE author, after mentioning work which had been done by Piccard and Homolka in order to throw some light upon the constitution of cantharidine, describes some investigations made by him in that direction.

His attempts to oxidise cantharidine by means of fuming nitric acid, were not successful, as he could not obtain the product free from undecomposed cantharidine. According to experiments made by Homolka (Ber. 19, 1082) it was to be foreseen that cantharidine would enter into a combination with phenylhydrazine, which was confirmed by author. On heating 1 part of cantharidine with 4 parts of phenylhydrazine and 2 parts of acetic acid (50 per cent.) to  $130^{\circ}$ — $140^{\circ}$  C. two bodies were obtained, which were separated by crystallisation from benzene. The more

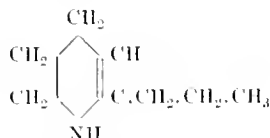
soluble part which was purified by recrystallisation from alcohol melts at  $237^{\circ}$ — $238^{\circ}$  C. and is cantharidphenylhydrazine ( $C_{16}H_{12}O_4 \cdot C_6H_5N_2H$ ). The second compound melts at  $130^{\circ}$ — $131^{\circ}$  C. and has the formula  $C_{16}H_{21}N_4O_2$ . On heating cantharidine with 10 parts of strong alcoholic solution of ammonia in a sealed tube for eight hours small prisms were obtained which gave figures corresponding to the formula  $C_{10}H_{12}(NH)O_2$ , melting at  $200^{\circ}$ — $201^{\circ}$  C. In this compound one of the oxygen atoms of cantharidine is substituted by an imido group.—O. J. S.

*Catalytic Formation of Ammonia from Nitrates.* O. Loew. Ber. **23**, 675—680.

See under XV., page 634.

*$\gamma$ -Coniceine, Conyrrine, and Inactive Coniine.* E. Lellmann and W. C. Müller. Ber. **23**, 680—684.

THE authors have confirmed the identity of the substance coniceine obtained by the action of alcoholic potash on coniine chlorinated at the nitrogen atom (this Journal, 1889, 636) with the base obtained by Hofmann by the action of bromine and sodium hydrate on coniine, and called  $\gamma$ -coniceine. Pure coniceine is optically inactive. The constitutional formula for this body previously proposed by the authors, viz. :—



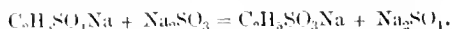
Coniceine.

is confirmed, for by distillation of its zinc double chloride with zinc dust it is completely converted into  $\alpha$ -propylpyridine, the identity of which was established by an analysis of its platinum double chloride. This salt was found to melt at  $172^{\circ}$ , whilst Ladenburg gives  $159^{\circ}$ — $160^{\circ}$  as its melting point. The crystalline form of the two preparations agreed, and by distilling coniine hydrochloride with zinc dust and converting the propyl-pyridine obtained into the double platinum chloride, this was also found to melt at  $172^{\circ}$ . It appears therefore that two distinct modifications of this salt exist. Some  $\gamma$ -coniceine seems to be formed together with conyrrine when coniine hydrochloride is distilled with zinc dust. Tin and hydrochloric acid reduce coniceine to coniine—a further confirmation of the above formula. The product obtained, like Ladenburg's synthetically-prepared coniine, was inactive.—C. A. K.

*New Method for Preparing Ethylsulphonic Acid.*

F. Mayer. Ber. **23**, 908—912.

THE author has found that on heating concentrated solutions containing molecular proportions of sodium sulphite and sodium ethylsulphate from  $110^{\circ}$ — $120^{\circ}$  in a closed vessel, sodium ethylsulphonate is obtained according to the equation :—



For the purpose of identifying the acid obtained in this manner the following salts were prepared and examined :—

The sodium salt  $C_2H_5SO_3Na + H_2O$  forms small lamellar crystals, which lose water on heating. The double salt  $4 C_2H_5SO_3Na \cdot Na_2H_2O$  was obtained by dissolving two parts of anhydrous sodium ethylsulphonate and one part of sodium iodide in hot alcohol, 96° Tr. On cooling the mass crystallises in fine silky needles. Although this salt contains water it does not lose weight when heated to  $100^{\circ}$ , but decomposes at a higher temperature. The barium salt  $(C_2H_5SO_3)_2Ba + 2H_2O$  was prepared by decomposing the sodium salt with sulphuric acid, extracting the resulting ethylsulphonic acid with alcohol, saturating with barium carbonate, filtering, evaporating the filtrate to a small bulk,

and adding alcohol to the evaporated mass. On standing the barium salt crystallises out in the form of colourless lustrous needles. The chloride  $C_2H_5SO_3Cl$  was obtained by mixing the anhydrous sodium salt with phosphoric pentachloride, adding water and subjecting the resulting oily product to fractional distillation. It boils at  $171^{\circ}$ — $173^{\circ}$ .

—D. B.

*A By-product in the Synthetical Method of Preparation of Cocaine.* C. Liebermann and F. Giesel. Ber. **23**, 926—929.

IN a previous communication (this Journal, 1890, 543) the authors described a by-product obtained in the preparation of cocaine, which appeared closely allied to the dextrorotatory cocaine obtained by Einhorn and Marquardt (this Journal, 1890, 513). There were, however, some points of difference in the two products and their derivatives, and in order to establish the identity or otherwise of the two bodies the authors have prepared dextro-rotatory cocaine according to the directions of Einhorn and Marquardt, and compared its properties with the base obtained by themselves. The two bodies appear identical in every respect; both the bases were obtained in a crystalline condition, and the slight solubility of their nitrate in water, which had previously appeared to distinguish the two products, is characteristic of them both. The by-product is therefore isomeric with cocaine.—C. A. K.

*Preparation and Properties of Aricine.* H. Moissan and E. Landrin. Compt. Rend. **110**, 469—471.

THE authors have extracted 2 kilos. of the alkaloid aricine, first discovered by Pelletier and Coriol in the bark known as *Cusco cinchona*, or *arica* bark. The bark used by the authors contained neither quinine nor cinchonine, but from 3 to 3.5 per cent. of aricine, a much larger proportion than that recognised by previous experimenters. To extract it the powdered bark is mixed with 10 per cent. of lime and the same quantity of strong caustic soda, and the mixture nearly dried on a water-bath. The residue is repeatedly extracted with ether, and the ethereal solution precipitated by addition of sulphuric acid, when a cheesy yellow sulphate of aricine separates. This is dissolved in boiling water, and the crude alkaloid precipitated by ammonia as a resinous substance. This is powdered, and crystallised two or three times from boiling alcohol. Analysis leads to the formula  $(C_{23}H_{26}N_2O_4)$  which is that already adopted by Gerhardt. The pure alkaloid is insoluble in water, soluble in 90 per cent. alcohol (which dissolves 10 grms. per litre at  $15^{\circ}$  and 90 grms. at the boiling temperature), and soluble in ether (30 grms. per litre at  $15^{\circ}$ ); it melts at  $188^{\circ}$ — $189^{\circ}$ . The rotatory power in alcoholic solution is  $[\alpha]_D = -58^{\circ} 18'$  and in ethereal solution  $[\alpha]_D = -92^{\circ} 30'$ . The hydrochloride in alcoholic solution has a rotatory power of  $[\alpha]_D = +14^{\circ} 30'$ . The melting point and optical properties thus differentiate the alkaloid from its isomer *cusconine*.—J. M. H. M.

*Fluoroform.* Meslans. Compt. Rend. **110**, 717—719.

IODOFORM (2 parts), silver fluoride (2 parts), and chloroform (1 part) are mixed in a flask kept well cooled by iced water and connected with a lead worm (cooled down to  $-23^{\circ}$ ) leading to a U-tube containing dry silver fluoride heated to  $100^{\circ}$ . When the temperature of the water around the flask is slightly raised a colourless gas is steadily evolved and may be collected in glass jars over mercury. It is freed from chloroform by prolonged contact with dry caoutchouc, and from a trace of carbon monoxide by cuprous chloride. Fluoroform has an agreeable odour resembling chloroform; it burns with some difficulty, forming hydrogen fluoride, and imparting a blue colour to the flame. It is sparingly soluble in water, chloroform, and benzene, but dissolves in about one-fifth its volume of alcohol.

It may be liquefied by a pressure of 40 atmospheres at  $20^{\circ}$ , and if more strongly compressed and released, it solidifies. When heated in a sealed tube with alcoholic potash, it is decomposed into potassium fluoride and formate.



Sodium burns with brilliancy when heated in this gas, carbon, sodium fluoride, and methane being formed.

The action of dry silver fluoride on iodoform containing no admixture of chloroform gives rise to a gaseous fluoriodoform which is now under investigation.—S. B. A. A.

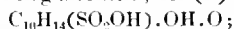
#### *Sulpho-conjugated Phenols derived from Camphor.*

P. Cazenave. *Compt. Rend.* **110**, 719—722.

THE action of concentrated sulphuric acid on camphor is known to result in the formation of camphorone, but the simultaneous formation of sulphonic derivatives has hitherto been overlooked. When camphor is heated with concentrated sulphuric acid, the camphorone removed, and the largely diluted acid liquid saturated with barium carbonate and filtered, the solution is coloured faintly violet by ferric chloride, and yields on evaporation small quantities of a difficultly crystallisable barium salt of a sulphonic acid.

By heating camphor monochloride (100 grms.) with concentrated sulphuric acid (500 grms.) to 30°, a solid (A) and three liquid syrupy compounds (B, C, D) may be isolated, all of which dissolve in water, are inactive to light, give the phenol reactions with ferric salts and acetic anhydride, and contain sulphur in the sulphonic state, only removable by fusion with potash.

The formula found for (A) is  $C_9H_{12} \cdot SO_2 \cdot (OH)_2 \cdot O$ , both of the hydroxyls being alcoholic; for (B)—



for (C),  $C_{10}H_{14}(SO_2OH)_3 \cdot OH \cdot O_5$ . No formula has yet been determined for (D).

The author points out that the formation of these compounds is a proof that the terebene series contains the benzene nucleus.—S. B. A. A.

#### *Some Compounds of Chlorides of Mercury with Ammonia.*

G. André. *Bull. Soc. Chim.* 1889, **2**, 145—153. (See also this Journal, 1889, 915 and 916.)

WHEN the compound  $NH_2HgCl \cdot HgCl_2$  is heated to 130° in a vacuum, nitrogen is slowly evolved, and between 160° and 230° it is accompanied by ammonia. Beyond this temperature nitrogen is mainly given off. Mitscherlich succeeded at one stage in isolating the compound  $2 NH_2Cl$ .

The action of potash and dilute sulphuric on the compound  $NH_2HgCl \cdot HgCl_2$  decomposes it, and in both cases the same amount of nitrogen, in form of ammonia (4.3 per cent.), evolved. If treated with sulphuretted hydrogen all the nitrogen is evolved as ammonia. Potassium chloride dissolves the compound, and this solution may be readily analysed. It is treated with sulphuretted hydrogen, the mercury separated as sulphide, and the filtrate boiled with caustic alkali, and the ammonia collected and titrated. The chlorine may be determined by heating with lime to redness, or by dissolving the compound in ammonium sulphate, adding acetic acid, precipitating the mercury with sulphuretted hydrogen, and, after getting rid of the gas, titrating the chlorine.

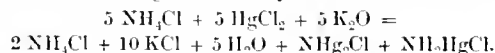
Ammonium chloride dissolves the compound, forming an addition product  $NH_2HgCl + NH_4Cl = (NH_3)_2HgCl_2$ .

The chloramide is decomposed by boiling water, giving a compound  $NH_2(HgOHg)Cl$ , which dissolves in cold ammonium chloride, but not in a hot solution.

If the equivalent of potash is first added to mercuric chloride before the addition of ammonium chloride, the chloride of tetramerenammonium,  $NH_2Cl$ , forms part of the precipitate. On the other hand, ammonia has a tendency to react with mercuric chloride and to form the chloramide, which again is decomposed by water, forming  $NH_2(HgOHg)Cl$ . The precipitate may consist, therefore, of the three compounds,  $NH_2Cl$ ,  $NH_2HgCl$ , and  $NH_2(HgOHg)Cl$ . This result appears from the analyses, which do not correspond to any simple formula.

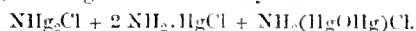
On adding ammonium chloride solution to freshly precipitated mercuric oxide, formed by adding caustic potash to mercuric chloride, the yellow tint of the oxide becomes paler, and, after two hours' shaking, the precipitate is perfectly colourless. The precipitate, when washed and

dried, contains three-fifths of the nitrogen of the ammonia salt. The following reaction may occur—

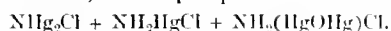


Whenever ammonium chloride is present with tetramerenammonium chloride, the former disappears according to the reaction  $NH_2Cl + NH_4Cl = 2 NH_3HgCl$ .

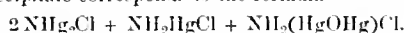
This being the case, and the chloramide undergoing decomposition with water forming dimerenammonium chloride, the formula of the precipitate may be represented as follows, which agrees with the analyses—



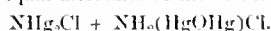
But if the ammonium chloride is first added, and subsequently the potash, then the light yellow precipitate remains unaltered, and the precipitate has the formula—



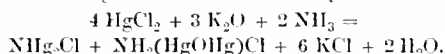
When less ammonium chloride is employed in the first reaction, it requires longer for the precipitate to become white, and only one quarter of the nitrogen is taken up. The precipitate corresponds to the formula—



If the reaction occurs at the boiling point of water, the compound  $NH_2Cl$ , being stable, and the chloramide being converted into dimerenammonium chloride, the product will consist of equal molecules of the bodies—

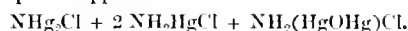


When ammonia is allowed to act upon a mixture of freshly precipitated mercuric oxide and chloride, the filtrate contains no nitrogen. The reaction takes place as follows:—



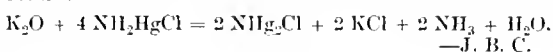
This compound of equal molecules of the two salts is very stable.

If a mixture of mercuric chloride, ammonia, and potash be made, the two former being in excess, the precipitate, which changes, on shaking, from yellow to white, takes up two-thirds of the nitrogen in the form of ammonium chloride. The compound appears to have the formula—



If the ammonia alone is in excess, the amount of mercury in the precipitate increases and the chloramide disappears. The formula here is  $NH_2HgCl + 2 NH_2(HgOHg)Cl$ .

When potash is added to the chloramide there is no change of colour in the precipitate. From the quantity of ammonia found in the filtrate, the following reaction probably occurs:—



—J. B. C.

#### *The Alkaloids of Cod Liver Oil.* A. Gautier and L. Mourgues. *Bull. Soc. Chim.* 1889, **2**, 213—238.

IN pursuance of the work of one of the authors on the prevalence of leucomaines in the animal economy, a research was begun on the question of the presence of alkaloidal bodies in cod-liver oil, and their therapeutic value. The oils to which most attention was paid were the light brown samples which are generally admitted to be the most active. Such oils are not obtained by extraction from the perfectly fresh livers, but are produced after a certain fermentative change, distinct from putrefaction, has taken place which causes them to contain biliary products. The sample worked upon came from Bergen, and its purity was assumed from the evidence afforded by its direct origin, its specific gravity (0.928 at 15° C.), its rise of temperature with sulphuric acid (89.3° C.), and by its remaining fluid at 0° C. After many experiments and the qualitative proof of the presence of alkaloids, the following plan was adopted for their extraction: 100 kilos. of the oil were extracted with an equal volume of 35 per cent. alcohol containing 3 grms. of oxalic acid per litre, by shaking in 20 bottles of a capacity of 15 litres each. Oxidation was prevented by replacing the air by carbon dioxide. The extract was



nearly neutralised with milk of lime, filtered, and distilled *in vacuo* at 40° C. When the bulk of the solution had been reduced to one-twentieth, lime was added to complete neutrality and the evaporation finished *in vacuo*. The residue was treated with 83 per cent. alcohol, the solution filtered, distilled *in vacuo*, potash added to the residue and the bases thus freed shaken out with ether. From their ethereal solution they were precipitated by powdered oxalic acid, the oxalates washed with ether and dried. By this means 53 grms. of oxalates were obtained, and subsequent examination of the extracted oil showed it to be almost free from basic constituents. It appears, therefore, that the light brown oil contains as much as 0.5 gm. of mixed alkaloids per kilo., equivalent to about 6.5 mgrms. in a spoonful weighing 13.5 grms., whereas the almost colourless specimens gave no ponderable quantity when as much as 10 kilos. were treated. From 52 grms. of the crude oxalates 26.5 grms. of free dried alkaloids were obtained. By fractional distillation and crystallisation of the chloroplatinates, there were separated and identified:—

(1.) Butylamine, consisting chiefly of normal butylamine, but having its boiling point raised by the presence of a little amylamine. Its physiological action was tried on a young guinea-pig, 0.025 gm. being injected, producing stupor and increasing the secretion of urine.

(2.) Amylamine, found to be chiefly iso-amylamine, corresponding to fermentation amylalcohol. It was extremely poisonous, 4 mgrms. of the hydrochloride subcutaneously injected into a greenfinch causing death in three minutes; smaller doses increase the flow of urine.

(3.) Hexylamine, having a physiological action similar to, but weaker than that of amylamine.

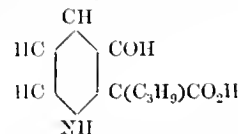
(4.) *Dihydrolutidine*, a new base forming about one-ninth of the total quantity of alkaloids extracted from the oil, being obtained from the fraction boiling between 198° and 200° at 770 mm. Analysis gave the figures C = 77.31, H = 10.47, N = 12.52 parts per cent. respectively, instead of the theoretical values C = 77.07, H = 10.09, N = 12.84, which correspond to the formula  $C_7H_{11}N$ , lutidine being  $C_7H_9N$ . Its vapour density determined by Victor Meyer's method, using a bath of diphenylamine vapour, was 3.3 instead of 3.8. It is a colourless somewhat oily liquid, alkaline and caustic, with a pungent but not disagreeable odour. It absorbs carbon dioxide from the air and is slightly soluble in water. Its boiling point is 199° C. (corr.). The hydrochloride crystallises in ill-defined needles or plates, has a bitter taste, is very soluble in water though not deliquescent, and in aqueous solution is partially dissociated at 100° C. The nitrate reduces silver nitrate just as the dihydropyridines do (Hofmann). The sulphate is somewhat bitter, odorous, and deliquescent, and crystallises in needles arranged in stellate groups. The chloroplatinate is a canary-yellow salt crystallising in needles or lozenge-shaped plates, soluble in hot water, but sparingly in the cold; it loses hydrochloric acid when boiled with water. The double salt formed with gold chloride forms thin lozenge-shaped plates, or in needles disposed fan-wise. When dihydrolutidine is mixed with a slight excess of methyl iodide in the cold, a confused mass of crystals of methyl dihydrolutidine iodide separates out, from which weak potash liberates methyl dihydrolutidine, which is one of the dihydrocollidines already known. Unlike the products of the action of methyl iodide on most of the pyridine bases, methyl dihydrolutidine does not give a red colouration with strong hot caustic potash. From its behaviour on oxidation with potassium permanganate, and the nature of the products, the authors are of opinion that dihydrolutidine contains two side chains, and corresponds to the formula  $C_5H_3(11)(CH_2)_2NH$ , and may be regarded as a dimethyl dihydropyridine. Dihydrolutidine is fairly toxic; a dose of 0.07 gm. per kilo. first caused trembling and excitement, followed by depression and partial paralysis, when administered to a guinea-pig. Death commonly follows.

(5.) *Aselline*,  $C_{25}H_{22}N_4$ .—After the separation of the above-mentioned bases by distillation, a new fixed base, yielding a sparingly soluble chloroplatinate, is obtained as a greyish amorphous non-hygroscopic mass, becoming yellowish when exposed to air and light. It has a density

of 1.05, and fuses to a brown liquid with an odour recalling that of certain ptomaines. It is only slightly soluble in water (yielding an alkaline, slightly bitter solution, from which it is precipitated by potash), but is easily soluble in ether and alcohol. Concentrated sulphuric acid gives a faint red colouration, soon becoming brown. Its salts are partially dissociated by water, the base being precipitated. The hydrochloride crystallises in small confused crosses, and is rather bitter. It forms a double salt with gold chloride, sparingly soluble, and depositing metallic gold on heating its solution, a white one with mercuric chloride, and a yellow easily alterable compound with platinum chloride. Aselline constitutes about one-fifteenth of the total bases; with regard to its physiological action, a small dose produces stertorous breathing and stupor; a large one, convulsions and death.

(6.) *Morrhaine*,  $C_{19}H_{27}N_3$ .—The chloroplatinate of this new base is more soluble than that of aselline and is obtained after the removal of the latter. The base is a yellowish oily liquid with an odour of *Seringa flowers*. It is slightly soluble in water, freely soluble in alcohol and ether, strongly alkaline, and absorbs carbon dioxide from the air. It precipitates cupric salts but does not redissolve the precipitate. Its hydrochloride crystallises in needles forming stellate groupings and is very deliquescent; the double salt with gold chloride is easily reduced on heating its solution; that with mercuric chloride is soluble, while the chloroplatinate crystallises in feathery needles sometimes agglomerated with rounded masses. Its physiological action is of especial interest, inasmuch as it forms rather more than a third of the total bases, and thus any activity that it may possess would largely account for the therapeutic value of the oil. Its toxicity is slight but it is a powerful diuretic and sudorific. It greatly aids assimilation and increases the appetite. 29 mgrms. injected as hydrochloride into a guinea-pig weighing 235 grms. caused micturition no fewer than five times in 2 hours 12 minutes, the animal losing 12.5 grms. This corresponds to the passage of 3,872 grms. in the case of a man, the normal maximum being 1,400 grms. in two hours. Morrhaine then is the characteristic active constituent of cod-liver oil.

Besides these alkaloids there are found in the alcoholic extract from cod-liver oil several complex *lecithins* and other organic bodies containing phosphorus, which doubtless add to the medicinal properties of the oil by presenting that element in an easily assimilable form. In the original extraction of the oil by alcohol, certain acids are removed from it as well as the bases, and remain as potassium salts after the latter have been shaken out by ether in the scheme of extraction detailed above. They can be obtained by acidulation by sulphuric acid, and comprise formic and butyric acids, phosphoric and glycerin-phosphoric acids, and *morrhaine acid*, the most important in therapeutic effect. *Morrhaine acid*,  $C_7H_{13}NO_5$ , is probably contained in the oil in the form of lecithins or similar bodies, from which it separates on evaporating the acidulated alcoholic extract. When purified it crystallises in dirty yellow prisms or plates. It is slightly soluble in water, freely soluble in alcohol, and behaves both as an acid and base, combining with both alkalis and acids. It forms double salts with platinum and gold chlorides. From its behaviour on distillation with alkalis and oxidation with permanganate the authors believe it to contain the pyridine nucleus, and one carboxyl group united with that nucleus *indirectly*, and assign to it the formula—

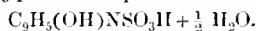


The *Gadaine* of De Jongh seems to be identical with morrhaine acid, although stated by him to contain no nitrogen. The physiological action of morrhaine acid was found to be to increase the appetite and renal secretion. The latter property is particularly striking when a guinea-pig is used for experiment, as normally the urine of that animal is turbid and scanty, becoming clear and abundant after the administration of the dose.

Cod-liver oil may, therefore, be regarded as a vehicle and storehouse for quite a number of active principles tending to increase excretion and appetite, while presenting a certain quantity of food substances in a highly assimilable form.—B. B.

*On p-Hydroxyquinolinesulphonic Acid.* A. Claus and M. Posselt. J. prakt. Chem. **41**, 158—160. (See Monatsh. **10**, 804, and J. prakt. Chem. **41**, 41.)

THE authors describe briefly the above-named acid and its salts—*p*-hydroxyquinolinesulphonic acid—



In yellow needles decomposing at  $270^\circ$ , with difficulty soluble in water, soluble in alcohol, dilute hydrochloric acid and glacial acetic acid. Insoluble however, in ether, benzene, &c. The acid forms no dyes with diazo-compounds. It forms basic and normal salts of the alkalis. The normal sodium (1 mol.  $\text{H}_2\text{O}$ ) and potassium (1 mol.  $\text{H}_2\text{O}$ ) salts are described. A *mono-* and *di-bromo derivative* were obtained with 1 and 2 mols. respectively of bromine, both occur in needle-shaped crystals, the latter being insoluble in water but soluble in alcohol.—D. A. S.

*Action of Ammonium Formate on Ketones.* R. Leuckart. J. prakt. Chem. 1890, **41**, 330—340.

AROMATIC aldehydes and ketones, on heating with ammonium formate, are first reduced, owing to the liberation of carbon monoxide, and then converted into the corresponding base. Benzophenone, for example, gives benzhydramine,  $(\text{C}_6\text{H}_5)_2\text{CH.NH}_2$ , whilst acetophenone yields phenylethylamine,  $\text{C}_6\text{H}_5.\text{CH}(\text{CH}_3).\text{NH}_2$ . The reaction is in some cases rather complicated; benzaldehyde, for example, gives not only benzylamine, but also formylbenzylamine, dibenzylamine, formyldibenzylamine, and large quantities of tribenzylamine.

Benzil, phenanthraquinone, and benzoin, yield nitrogenous condensation products on heating with ammonium formate, whilst anthraquinone is simply reduced to anthracene.

Three bases are formed when acetone is heated with ammonium formate ( $1\frac{1}{2}$  parts) at  $200^\circ$ — $210^\circ$  for five hours. These compounds can be isolated as follows:—The crude product, which is a yellowish brown oil with a powerful coniine-like smell, is mixed with excess of soda and the bases distilled with steam; the distillate is acidified with hydrochloric acid, concentrated by evaporation, then rendered strongly alkaline with soda, the liberated bases extracted with ether, dried over caustic soda, and fractionated.

The fraction boiling at  $155^\circ$ — $156^\circ$ , which represents about 9 per cent. of the acetone employed, is a colourless strongly basic oil, with a slight alkaloid-like odour; it has the composition of a tetrahydrocollidine,  $\text{C}_{10}\text{H}_{15}\text{N}$ .

The fraction boiling at  $195^\circ$ — $200^\circ$  is a yellow strongly basic oil, with a very powerful disagreeable smell; it has the composition,  $\text{C}_{10}\text{H}_{15}\text{N}$ .

The fraction boiling at  $260^\circ$ — $270^\circ$  is also a yellowish-brown, strongly basic oil with a very powerful disagreeable smell; its composition is  $\text{C}_{10}\text{H}_{15}\text{N}$ .

These three compounds are probably members of a homologous series of bases nearly related to the pyridine bases or their reduction products; this is shown not only by their composition, but also by their strongly basic character, and their peculiar coniine-like smell, which properties they have in common with the coniceines  $\text{C}_8\text{H}_{13}\text{N}$ , para-coniine, triacetone, and other bases of like nature. Their method of formation also shows that the bases described above are probably pyridine derivatives, as such compounds can be obtained by the action of ammonia on acetone. The bases containing oxygen, for example, which are obtained by heating acetone, or a mixture of acetone and an aldehyde with ammonia, are hexahydropyridine derivatives, as has been shown by Heintz and E. Fischer, whilst from acetone and aldehyde ammonia, and from mesityl oxide and ammonia trimethylpyridine is obtained.

The boiling point of the base  $\text{C}_{10}\text{H}_{15}\text{N}$ , described above is also very near to that of the isomeric  $\alpha$ -coniceine, with

which, however, it is not identical, as  $\alpha$ -coniceine solidifies at  $35^\circ$ .

These experiments show that ammonium formate and ammonia act on acetone in a similar manner, but that the former has, at the same time, a reducing action; the further investigation of the compounds described above is delayed owing to the difficulty of preparing them in large quantities.

—F. S. K.

*Pyridine and its Relations to Quinoline, Isoquinoline, and the Alkaloids.* A. Elinger. J. prakt. Chem. 1890, **41**, 341—359.

THE halogen alkyl additive compounds of certain alkaloids are, as is well known, by the action of freshly prepared moist silver oxide, under suitable conditions, converted into quaternary bases, which are soluble in water, combine with carbon dioxide, and form, with acids, salts which are identical with the corresponding halogen alkyl additive product of the alkaloid, are not decomposed by dilute ammonia, and form crystalline, anhydrous, very stable double salts, identical with the corresponding double salts of the halogen additive products. These double salts, when treated with alkalis, yield a base which is quite different to that obtained by means of silver oxide, and which behaves in all respects like a tertiary base; it is almost insoluble in water, does not combine with carbon dioxide, and its halogen salts are not identical with the corresponding alkyl halogen additive compound.

Isoquinoline behaves in this respect like many alkaloids, so that an isoquinoline nucleus is probably present in many of these compounds; quinoline, on the other hand, does not show a like behaviour as the bases, obtained by decomposing its halogen alkyl additive compounds with alkalis, yield with acids, salts which are identical with the original alkyl halogen additive products. These salts do not give quaternary bases with silver oxide, but bases, the halogen salts of which are identical with the halogen alkyl additive compounds of quinoline.

The behaviour of pyridine has been investigated in this direction, and the author finds that pyridine behaves exactly like the alkaloids and isoquinoline. It would seem, then, that the so-called pyridine ring in quinoline is differently constituted to that in pyridine itself.—F. S. K.

*On the Germination of the Seed of the Castor-Oil Plant* (*Ricinus communis*). J. R. Green. Proc. Roy. Soc. **47**, 146—147.

A FERMENT exists as a zymogen in the resting seed, which is readily developed by warmth and weak acids into an active condition, and splits up the fat into glycerin and (chiefly) ricinoleic acid. Further changes, brought about by the protoplasm of the endosperm cells, form from the latter a lower carbon acid, soluble in water, and crystalline. The proteids of the seed, globulin and albumose, are split up by another ferment, with formation of peptone and asparagin. The reserve materials absorbed by the endosperm are a crystalline acid, sugar, asparagin, and, perhaps, some peptone. The mode of absorption is always dialysis. During the germination, a rennet ferment of considerable vigour is liberated in the endosperm.—J. M. H. M.

*Recent Discoveries in Fine Chemicals.* Chem. Ind. **13**, 177—182.

*Monochloral-antipyrine.*—If concentrated solutions of equal weights of chloral hydrate and antipyrine are mixed, an oily layer is formed, which soon crystallises. If for 4.7 parts of chloral hydrate, 5.3 of antipyrine be taken and each be dissolved in 5 parts of water by the aid of a gentle heat and mixed, large crystals may be obtained of this compound. When re-crystallised from alcohol it has the formula  $\text{C}_{13}\text{H}_{13}\text{N}_2\text{Cl}_2\text{O}_2$ . Its m.p. is  $67^\circ$ — $68^\circ \text{C}$ , and it dissolves in water at  $140^\circ \text{C}$ . to the extent of 7.85 grms. in 100 grms.; it gives, with ferric chloride, the blood-red colour

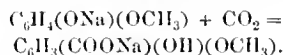
characteristic of antipyrine, and yields chloroform when heated with potash solution. It also reduces Fehling's solution when warm.

**Dehydrated Compound.**—When this compound is kept for a certain time heated to its melting point, crystals are deposited of a *dehydration compound*, first prepared by Reuter. It is insoluble in water, melts at  $186^{\circ}$ – $187^{\circ}$  C., and is easily distinguished from monochloral-antipyrine by not giving the ferric chloride reaction.

**Bichloral-antipyrine.**—This is obtained if instead of taking equal molecular proportions, a concentrated solution of chloral-hydrate be used in excess. An oily layer is formed from which prismatic needles are obtained containing 2 mols. of chloral to 1 of antipyrine. The melting point of bichloral-antipyrine is also  $67^{\circ}$ – $68^{\circ}$  C., but this compound is more soluble than the monochloral-antipyrine, the proportion being 9.98 grms. in 100 of water at  $14^{\circ}$  C. It gives with ferric chloride the red colouration of antipyrine and reduces Fehling's solution when heated. In water it seems to undergo some dissociation. (*Béhal et Choay, Journ. Pharm. Chim. May 15, 1890, 539. Pharm. J. 1890, 977.*)

**Incompatibility of Antipyrine and Carbolic Acid.**—Millard and Stark first pointed this out (*Pharm. J. 1890, 861*). It has been confirmed more recently by Tardy (*Répertoire, May 10, 209*), who shows that a solution of antipyrine to which a solution of carbolic acid was added, separated after some hours into two layers. The upper layer only contained antipyrine, the lower, an oily liquid containing both antipyrine and carbolic acid. The taste was piquant, and the colour a faint rose; it had a slight odour of phenol, and a specific gravity of 1.13. It was neutral, insoluble in water, but soluble in alcohol and in ether.

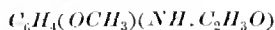
**Guaïacol carboxylic acid** has been found to possess extraordinary antiseptic powers. In its preparation, sodium guaïacolate is saturated with carbon dioxide under pressure in the cold, and the mixture is then heated (without changing the vessels) to  $100^{\circ}$  C. The reaction is—



The product is dissolved in water and decomposed by a mineral acid when the guaïacol carboxylic acid crystallises out in combination with 2 mols. of water. The substance melts in the anhydrous condition at  $148^{\circ}$ – $150^{\circ}$  C., gives in solution a blue colour with ferric chloride, and on being sufficiently heated is decomposed into carbon dioxide and guaïacol (*Pharm. J. 1890, 977*).

**Benzanilide**,  $\text{C}_6\text{H}_5 \cdot \text{NH} \cdot \text{COC}_6\text{H}_5$  has been recommended by Hahn (*Pharm. Zeit. 34, (72)*) as a powerful febrifuge in the use of which untoward accompaniments are not observed.

**Methacetin or Para-acetanisidine**—



(*Pharm. Zeit. 34, (55)*), forms small white, almost tasteless plates, melting at  $127^{\circ}$  C., and far more soluble in water than the analogously constituted phenacetin. Its reactions are very similar to those of phenacetin, and only differ in being less intense than those of that compound. 1 cc. of hydrochloric acid dissolves 0.1 grm. of methacetin very easily, whereas the same quantity of phenacetin would prove almost insoluble. Methacetin is useful in rheumatism in the joints, and completely transcends phenacetin in its efficacy in cases of neuralgia of the most varied kind.

**Formyl-para-phenactidine** ( $\text{C}_2\text{H}_5\text{O} \cdot \text{C}_6\text{H}_4 \cdot \text{NH} \cdot \text{CHO}$ ), though constituted analogously to phenacetin, appears to possess no antipyretic powers, but it serves as an antidote in cases of strychnine poisoning.

**Phenylurethane** ( $\text{C}_2\text{H}_5 \cdot \text{OCO} \cdot \text{NH} \cdot \text{C}_6\text{H}_5$ ) and **Pyrodine** (**Acetylphenylhydrazide**) have, neither of them, succeeded well. The latter is reported by Reubers (*Deutsch. med. Woch. 1889, No. 47*) to be a direct blood poison, the antithermic properties of which are really due to destruction of the red blood corpuscles. Guttman, Zerner, and Oestreich report deleterious intoxicating effects.

**Thalline**, though for similar reasons no longer used as an antipyretic, is still used somewhat considerably in the treatment of gonorrhœa.

**Amylene hydrate** has been proved by the researches of Laves and Jastrowitz (*Berl. klin. Woch. 1888, No. 21*) to be a valuable hypnotic, the employment of which is never dangerous and only seldom occasions unpleasant symptoms. Wildermuth (*Neurol. Centr. 1889, No. 15*) has obtained favourable results with epileptics by its employment.

**Rubidium-ammonium bromide** is, according to Lanfenauer and Rottenbiller (*Ther. Mon. 3, 348*, and *Gyogyosaz, 1889, No. 43*) a good anti-epileptic. Its high price is the only thing against it. Lanfenauer states the interesting conclusion that the anti-epileptic action of the alkaline bromides varies directly with their molecular weights.

**The Relative Antiseptic Powers of Isomeric Organic Compounds.** T. Carnelley and W. Frew. *Proc. Chem. Soc. 1890, 90.*

The authors have determined the relative antiseptic powers, in reference to ordinary aerial micro-organisms, of a number of isomeric organic compounds, more particularly di-derivatives of benzene, with the object of investigating the influence of atomic arrangement on this property.

A table of results is given which indicate that, so far as the compounds which have been tried are concerned (and with the exception of the hydroxybenzoic acids), para-compounds are more antiseptic than the corresponding ortho- and meta-compounds. On the whole, compounds containing the carboxyl-group are comparatively weak, while phenols and nitro-compounds are relatively strong antiseptics; para-nitrophenol being, with the exception of  $\alpha$ -naphthol, the most powerful of any of the compounds tried. These results entirely accord with those of Walcott Gibbs and Hare, who have recently investigated the poisonous action of di-derivatives of benzene on dogs.

**Note on the Preparation of Pyrocatechol.** W. H. Perkin, jun. *Proc. Chem. Soc. 1890, 90.*

THE very high price of pyrocatechol (catechol) renders it desirable to discover improved methods of preparing it; the author has, therefore, studied the action of iodhydric acid on guaïacol, which is easily procured at a moderate cost. He finds that an almost theoretical yield of catechol may be obtained by boiling guaïacol with a fuming solution of hydrogen iodide. Details are given in the paper.

## PATENTS.

**Improvements in Apparatus for Filtering Perfumes, Tinctures, Essences, Cordials, and other Liquids.** F. L. Johnstone, Henley-on-Thames. Eng. Pat. 1888, February 6, 1890, 6d.

THE object of this invention is to filter the insoluble matter held in various liquids, without the loss of strength or aroma by evaporation, by enclosing them whilst filtering in air-tight vessels and pipes. The liquid to be filtered is poured into a holder, made of any suitable material, having a biogged lid which can be closed air-tight. One end of a pipe is attached to this lid, and the other to an air-vessel connected with a vacuum pump. The pump on being set in motion draws air from the filtered-liquid receiver below it, which also has an air-tight lid, and compresses the air in the air-vessel above it. A tap under the air-vessel is then turned off and another at the upper part turned on, when the compressed air passes into the liquid-holder, forces the liquid through the filtering material it contains and through further materials, finally flowing through a spiral tube into the filtered-liquid receiver, from which it can be drawn off by a tap. There are three claims.—E. S.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

### PATENTS.

*Process for Rendering Cardboard and the like Impervious to the Action of Photographic Chemicals, and suitable for Photographic Purposes.* W. F. Greene, London. Eng. Pat. 7747, May 9, 1889. 6d.

THE object of this invention is to prepare ordinary cards in such a manner that permanent photographic prints may be produced directly on them, thus avoiding the processes of mounting and rolling.

The cards are coated one or more times with—

Oxide of zinc.....	1 part.
Turpentine .....	3 parts.
Pale gold size .....	1 part.

With or without a preliminary treatment in dilute sulphuric acid (2:1). When dry the cards are sensitised and printed in the usual manner. There are ten claims.—F. H. L.

*An Improved Method of Deciding the Correct Exposure in Platinotype Printing, and Apparatus therefor.* C. R. Crawford, London. Eng. Pat. 10,504, June 28, 1889. 6d.

THE printing frame containing the negative to be printed on platinotype paper is exposed side by side with an actinometer, made by preparing a negative of a series of numbers or letters, each one of which is covered with one more thickness of tracing paper or other translucent material than the previous letter.

Under this negative is placed a piece of ordinary sensitised paper, and the printing is carried on till the platinotype paper is found to be correctly exposed. The last visible number on the actinometer is then noted, and the operation can be repeated with fresh papers, the exposure being continued till the same number is visible in the other frame.—F. H. L.

*Improvements in or relating to the Manufacture of Photographic Films.* A. J. Boulton, London. From George Eastman, Rochester, U.S.A. Eng. Pat. 19,897, December 10, 1889. 6d.

A SOLUTION is prepared by dissolving 9,000 grains of soluble gun cotton and 5,460 grains of camphor in 112 ounces of methyl-alcohol and filtering; to avoid shrinkage on drying it is preferable to add 28 ounces of "fusil" oil and 7 ounces of amyl acetate. The coating table, which may have either a polished or dead surface, is covered with a weak solution of mineral wax or bees-wax in "benzine" and a thin film of the solution of gun-cotton spread upon it by means of a longitudinal spreader. The thickness of this film should be from 3 to 5 thousandths of an inch.

The film is then washed with a 2 or 3 per cent. solution of sodium or potassium silicate, and when dry is coated with emulsion. When the sensitive film has dried, the whole is cut into pieces of suitable width and stripped from rigid support.—F. H. L.

*Improved Apparatus for Coating Photographic Plates.* F. Bishop and A. Cowan, London. Eng. Pat. 304, January 7, 1890. 8d.

THE trough for holding the emulsion is made of the usual V-shape, one side being bedded on to a hot-water chamber, the other being adjustable by means of screws, so as to enable the thickness of the film being easily regulated, and to ensure its absolute uniformity. The hot-water chamber is pivoted on balanced levers in such a manner that plates of varying thickness may easily pass underneath. For coating large plates, the trough is provided with a rubber lip which rides on the surface of the glass and compensates for its inequalities. The plates are supported in passing under the trough on knife-edged rails at their extreme edges, and guided by means of rollers; gutters are also provided to catch any superfluous emulsion, and prevent its soiling the back. The specification is accompanied by drawings of the trough.—F. H. L.

## XXII.—EXPLOSIVES, MATCHES, Etc.

*The Annual Report (Fourteenth) of H.M. Inspectors of Explosives, for 1889.*

THIS report contains a full account of the working of the Explosives Act (1875) during the year 1889. The only modifications made by the Secretary of State were for the purpose of giving increased liberty in the method of packing the explosives to the manufacturers. The number of factories now licensed is 122, an increase of 10 on the previous year; the total number of magazines is now 357, being an increase of four on last year.

Increased facilities for storage of different explosives in the same magazine are now given. There were 132 accidents during the year, causing 39 deaths and injuring 102 persons. This shows a slight increase in the deaths over 1888, but is below the average for the last 10 years, and the number of deaths from accidents in manufacture was only seven. Dr. Duprè reports that the condition of the various authorised explosives examined during the year was, with two exceptions, highly satisfactory. The year has been characterised by continued activity among inventors, a great number of new explosives having been submitted for examination. A noticeable feature is also the gradual displacement of kieselguhr dynamite by the various gelatinised preparations. The new explosives examined were: Bronolith, 4 varieties; Diripsite, Trench's explosives, 2 varieties; Von Dahmen's dynamite; Fortis, 3 varieties; Fortisine; Stonite; Meganite; Blasting Matagnite; Forcite; Smokeless rifle powder; and Cycene. The last two are still under consideration, and all the others passed the tests satisfactorily except Diripsite, Meganite, 1 variety of Trench's explosive, 2 varieties of Bronolith, and 1 variety of Fortis. Bronolith is a picrate mixture, the samples passed were those rich in nitre; Diripsite is a chlorate mixture; Trench's explosive consisted mainly of gun-cotton and nitre, with the addition in one case of dinitrobenzene and in the other of picric acid, the latter being the one which failed to pass the test; Von Dahmen's dynamite (see this Journal, 1890, 105); Balistite is essentially a blasting gelatin, containing a very high proportion of gun-cotton; Fortis (this Journal, 1888, 401); Fortisine is a new name for fortis Nos. 3 and 4; Black Dynamite is essentially dynamite No. 1, containing, in place of ordinary calcined kieselguhr, kieselguhr only very partially calcined (this Journal, 1889, 818); Meganite is a gelatin dynamite containing sodium nitrate in place of potassium nitrate; Blasting Matagnite is a blasting gelatin containing nitrobenzene; Forcite is substantially a gelatin dynamite.

The importations of explosives during the year show a considerable increase as compared with those of 1888. The inspectors again express their views that "the capricious action of the railway companies in carrying some explosives and refusing to carry others (though sometimes identical in composition or practically so) led to much surreptitious conveyance."

Owing to "the way in which all explosives, whether manufactured in or imported into this country, are carefully watched as to their chemical stability, general safety, and method of packing," the inspectors consider that "no real reason exists why all authorised explosives should not be carried by the railways under suitable regulations," and they emphatically repeat that "all authorised explosives may be carried at least as safely as (and in several cases with considerably greater freedom from risk than) gunpowder."

There have been a few malicious attempts to destroy life and property by explosion, and several infernal machines had to be examined. An account is given of the principal foreign explosions, and the great Antwerp explosion of 6th September 1889, was specially investigated, and from the special report thereon large extracts are published in the present report. Experiments were also made as to the sensitiveness of blasting gelatin and gelatin dynamite when in the frozen and unfrozen condition, and although the results are not altogether concordant or conclusive, it would appear that blasting gelatin is more sensitive to explosion by percussion when frozen than when unfrozen. In the case of gelatin dynamite the sensitiveness of the variety known as "Gélinite" appears not to be appreciably affected by freezing. "Comparing blasting gelatin and gelatin dynamite it would seem that (a) of the unfrozen varieties blasting gelatin is the less sensitive; (b) of the frozen varieties blasting gelatin is very much the more sensitive; (c) unfrozen blasting gelatin is less sensitive than gelatin dynamite, whether in a frozen or unfrozen condition; and (d) frozen blasting gelatin is vastly more sensitive than gelatin dynamite in whatever condition." "It also appears that unfrozen dynamite is distinctly more sensitive to explosion than blasting gelatin or gelatin dynamite, in whatever condition; the sensitiveness of frozen blasting gelatin approaching it the most nearly. Both blasting gelatin and gelatin dynamite appear to furnish less effective results when exploded in a frozen condition than when exploded in a plastic condition."

The more important petroleum accidents, including the petroleum fire which resulted from the explosion of the ammunition factory at Antwerp previously noticed, are also referred to.—W. M.

*Notes on the Erosive Power of Compressed Gases.*  
Dauvrée. Mém. de l'Artillerie de la Marine, 2, 1889, 245; and Proc. Inst. Civil Eng. 1890, 53.

The author attributes the erosive action of powder to three distinct causes. First, there is the physical action, caused by the elevation of the temperature of the metal which is in contact with the superheated gases. It is this elevation of temperature which twists and contorts thin layers of the steel into tortuous forms. Secondly, there is the chemical action due to the combination of these small particles with the elements of which the gases are composed. The most noteworthy result of this combination is the production of iron pyrites. Thirdly, there is the mechanical action due to the impact of the gases against the walls which enclose them.

With dynamite, nitro-glycerin, and gun-cotton, this mechanical action is much the most marked, while the calorific and chemical effects of explosion are not always perceptible. In comparing the effects produced by the explosion of these agents with that of gunpowder, the much greater erosive effect produced by the former the author attributes to the rapid increase of the erosive power of gases as their pressure and temperature rise. He attributes

the pitting and distress in the surface of the metal in contact with the gases, to the fact that these are so highly compressed at the instant of explosion as to act like solid bodies.

*Smokeless Powder (Experiments made at Essen in July and August, 1889).* Revue d'Artillerie, 35, 1890, 486; and Proc. Inst. Civil Eng. 1890, 85—87.

These experiments were made to ascertain the ballistic effect of Nobel's powder, as regards maximum pressure and muzzle velocity in four guns, varying in calibre from 5 to 8½ centimetres, that is to say, from 1·968 to 3·317 in.

The article commences with a description of the powder taken from the specification of Nobel's patent of 31st January 1888. According to this it is a substance somewhat resembling horn in appearance, composed of nitro-glycerol, nitro-cellulose, and camphor, and is therefore a kind of explosive gelatin with camphor added, and with a considerable preponderance of nitro-cellulose. It may be made into grains of any form, and burns sufficiently slowly as to admit of its use in guns in lieu of ordinary powder, and it possesses the advantages of leaving very little residue and little or no smoke. The object of adding the camphor is to assist the dissolution of the nitro-cellulose, and also to moderate the rapidity of combustion. The proportion of camphor indicated is from 10 to 30 per cent. of the weight of the nitro-glycerol. When nitro-glycerol predominates it is recommended to make use of a volatile liquid capable of dissolving the nitro-glycerol and the camphor, such as benzene. When, on the contrary, nitro-cellulose predominates, a liquid such as acetic ether or acetone is used.

Two methods of preparation are described. In the first method 10 parts by weight of camphor are dissolved in 100 parts of nitro-glycerol, to which 200 parts of benzene are added. Into this mixture is introduced 50 parts of dried pulp of soluble gun-cotton. The benzene is then evaporated, and the materials thoroughly mixed by passing them between two hollow cylinders heated by steam to a temperature of 43·3° C. to 60° C. When the mixture is sufficiently homogeneous it is moulded into grains or rolled out into sheets, which are afterwards cut up into grains or pellets ready for use.

The other method is as follows:—Mix together 100 parts of nitro-glycerol, 10 to 25 parts of camphor, and 200 to 400 parts of acetate of amyl. Into this put 200 parts of dry pulp of soluble gun-cotton. This mixture is kneaded until the gun-cotton is completely dissolved. The pasty matter thus obtained is reduced into thin sheets, from which the moisture is expelled by heat, and the dry material is formed into grains or pellets fit for use.

The article then refers to a second patent of Nobel, dated 26th March 1889, in which there is no mention of camphor. The essential elements are as before, nitro-glycerol and nitro-cellulose, with the addition of nitrated starch, or of nitrated dextrine, and a certain quantity of an alkaline nitrate, chlorate or picrate.

The article then proceeds to give an account of experiments made at Essen with the Nobel powder in August 1889. The powder used was composed of a mixture in equal proportions of nitro-glycerol and nitro-cellulose kneaded together and made into cubical grains.

Experiments were made with cubical grains of four dimensions, viz., 0·1181, 0·1378, 0·1575, and 0·1969 in. of a side, as previous to these experiments nothing was known either as to the best weight of charge or the size of grain suitable to the calibre of the guns. The guns used on the experiments were as follows:—

No. 1—calibre, 1·968 in.; length of bore, 40 calibres; weight, 490 lb.

No. 2—calibre, 2·362 in.; length of bore, 40 calibres; weight, 862 lb.

No. 3—calibre, 2·952 in.; length of bore, 28 calibres; weight, 904 lb.

No. 4—calibre, 3·317 in.; length of bore, 40 calibres; weight, 2,310 lb.

The results are given in the following table:—

Description of Gun.	Date.	No. of Round.	Charge.	Size of Grain.	Projectile.	Velocity at 110 ft. from Muzzle.	Maximum Pressure.
			Lb.	In.	Lb.	Ft. per Second.	Tons per Sq. In.
No. 1	13 July 1889.....	1	0·63	0·1378	3·81	1,886	8·161
		2	0·77	0·1378	3·81	2,089	8·955
		3	0·80	0·1378	3·81	2,171	12·23
	17 August 1889.....	1	0·66	0·1181	4·07	1,798	9·713
		2	0·77	0·1181	4·10	2,014	11·33
		3	0·792	0·1181	4·10		13·01
		4	0·792	0·1181	4·10	Mean 2,070	12·28
		5	0·792	0·1181	4·10		13·80
		1	0·66	0·1181	6·84	1,169	3·152
		2	1·10	0·1969	6·84	1,937	6·895
		3	1·21	0·1969	6·84	1,758	8·003
No. 2	5 August 1889.....	4	1·10	0·1575	6·86	1,811	9·522
		5	1·00	0·1181	6·86	1,860	10·180
		6	1·10	0·1181	6·86	2,043	12·18
		1	0·66	0·1181	15·0	1,086	5·762
		2	0·77	0·1181	15·0	1,188	7·078
		3	0·88	0·1181	15·0	1,283	8·593
	12—13 August.....	4	1·00	0·1181	15·0	1,401	10·24
		5	1·10	0·1181	15·0	1,512	12·28
		6	1·17	0·1181	15·0	1,588	13·56
		7*	1·00	0·1181	15·0	1,421	10·14
		8*	1·00	0·1181	15·0	1,424	10·50
		9	1·10	0·1181	15·0	1,522	12·11
		10	1·17	0·1181	15·0	1,591	12·31
		1	2·20	0·1969	15·11	1,097	5·33
No. 4	5 August 1889.....	2	2·64	0·1969	15·50	1,886	8·13
		3	3·03	0·1969	15·50	2,004	9·02
		4	3·30	0·1969	15·50	2,136	9·25
		5	3·52	0·1969	15·50	2,239	11·29
		6	3·74	0·1969	15·50	2,326	11·69
	6 August 1889.....	1	3·30	0·1575	15·50	2,316	13·97
		2	3·54	0·1575	15·60	2,324	13·83
		3	3·30	0·1575	17·81		13·24
		4	3·30	0·1575	17·81	Mean 2,231	13·70
		5	3·30	0·1181	17·81		13·24
		6	2·86	0·1181	17·81	Mean 2,168	11·61
		7	2·86	0·1181	17·81		14·09

\* The powder in rounds 7 and 8 had been previously put into water for 30 minutes and then dried at a temperature of 86° F.

### XXIII.—ANALYTICAL CHEMISTRY.

#### *A new Apparatus for Fractional Distillation in Vacuo.*

H. Gautier. Bull. Soc. Chim. 1889, 2, 675—676.

This apparatus consists of a cylindrical glass tube, to the bottom of which a number of narrower tubes are fused, each of which is connected with a separate receiver. Into the upper portion a second tube is ground, which may be closed

by a cork with two holes. Through one a tube passes connected with the distilling apparatus, while the tube in the other hole is connected with the vacuum pump. By turning the cylinder round the liquid may be led into any of the receivers.—J. B. C.

*Viscosimeter; for Observations of Oils under constant Temperature.* C. Engler and A. Kunkler. *Diagl. Polyt. J.*, 276, 42—47.

THE apparatus represented in Figs. 1, 2, and 3, has been designed to remedy the two defects of Engler's viscosimeter, viz., that in observations at high temperatures, the temperature of the oil varies sensibly during the running off,

Fig. 3.

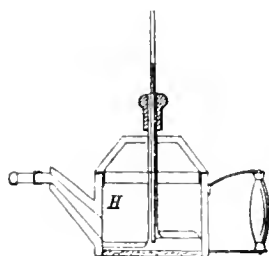


Fig. 1.

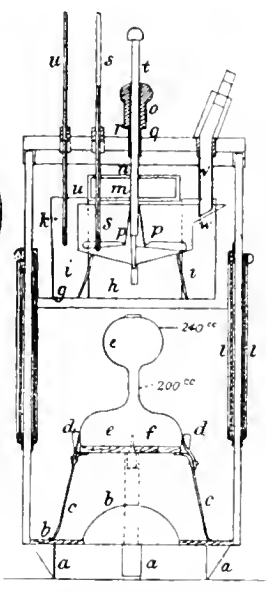
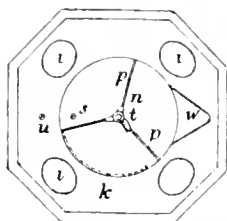


Fig. 2.



and the extremity of the efflux tube undergoes a gradual cooling. It is an octagonal jacketed air bath, 35 cm. high, and 20 cm. broad. The feet *a* stand in the ring of a tripod in such a way that the level of the bath can be adjusted so as to control the level of the liquid in the viscosimeter itself contained in the upper portion of the bath. The heating surface *b* is arched; above this and supported by the stand *c* is the measuring vessel *e*, cut off from direct radiation from *b* by the asbestos plate *f*. Above this is the dividing plate *g* upon which the viscosimeter is supported, the flow of oil passing to *e* through the opening *h*. Circulation of hot air into the upper chamber takes place through *h* as also through the four oval tubes *i* (Fig. 2). Through the cover plate pass the thermometers *u*, *s*, the axis of stirring apparatus with the stop *t* for the efflux tube, and the funnel *r* for introducing the oil, heated to the required temperature in the apparatus II (Fig. 3.) In the sides and cover, windows are let in the bath for illuminating, the former to permit observations of the level in the viscosimeter and the efflux of the oil into *e*. The method of using the instrument for the observations in question need no special description; full details of manipulation are given in the original.

That the instrument fully satisfies the required condition of constant temperature, is stated by the authors in the following terms: Up to 100° the temperature in all parts of the bath—heated with no charge of oil in the viscosimeter—is equal and constant, excepting in the lower stratum of air in the viscosimeter itself, which is of course shielded from the general circulation. This difference disappears with the introduction of the oil. At temperatures higher than 100° the air above the viscosimeter is of somewhat lower temperature, but the difference at 150° did not exceed 4°.

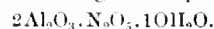
—C. F. C.

*The Action of Nitric Acid on Aluminium.* A. Ditte. *Compt. Rend.* 110, 782—784.

WHEN aluminium is placed in dilute nitric acid, it appears to be unacted upon, but after some time small bubbles of

gas appear on the surface of the metal and it gradually dissolves; the evolution of gas and dissolution of the metal is much hastened by allowing the action to take place in vacuo. This shows that the slowness of the action under ordinary conditions is due to the formation of a protective layer of gas on the surface of the metal. The chief constituent of the evolved gas is nitrogen; nitric oxide is also present, and ammonia is retained in solution. If a little platinum chloride be added at the commencement of the reaction, the aluminium becomes coated with a slightly adherent coat of reduced platinum and dissolves with evolution of a mere trace of nitrogen. Large quantities of ammonia are found in the solution.

The action does not cease when all the free nitric acid is neutralised, for aluminium will dissolve in solutions of its salts forming basic compounds. The action is very slow in the cold, but on boiling aluminium in a solution of its nitrate, it dissolves with evolution of hydrogen, and a white precipitate is formed, having the composition—



These experiments elucidate the action of water on aluminium; in this case a double protective coat of alumina and hydrogen is formed; if the hydrogen be prevented from accumulating by boiling and the alumina dissolved immediately it is formed, the metal will dissolve. This is exactly what takes place when aluminium is boiled in a solution of one of its neutral salts, with the sulphate  $4\text{Al}_2\text{O}_3 \cdot 3\text{SO}_3$  is formed, with the nitrate  $2\text{Al}_2\text{O}_3 \cdot \text{N}_2\text{O}_5 \cdot 10\text{H}_2\text{O}$ , and with the chloride  $\text{Al}_2\text{Cl}_6 \cdot 10\text{Al}_2\text{O}_3$ . The truth of this explanation is borne out by the fact that solution of salts, such as sodium chloride, which do not dissolve alumina, have no such action. (This Journal, 1890, 547 and 549.)

—A. L. S.

*Reagent for Free Chlorine in Hydrochloric Acid.*

G. A. Le Roy. *Boll. Soc. Chim.* 1889, 2, 729.

THE author recommends the use of diphenylamine to detect minute quantities of free chlorine in hydrochloric acid. A few small crystals of diphenylamine in presence of free chlorine produces immediately a blue colouration.—J. B. C.

*Volumetric Estimation of Copper.* A. Etard and P. Lebeau. *Compt. Rend.* 110, 408—410.

A CONCENTRATED solution of cupric bromide, mixed with strong hydrobromic acid, becomes deep violet in colour, like potassium permanganate, and almost opaque. The colour is due to a compound of cupric bromide and hydrobromic acid, which is decomposed by addition of water with formation of the ordinary green, and then blue, of cupric solutions.

If a titrated solution of stannous chloride be run into the violet liquid the colour is discharged as fast as the cupric bromide is reduced to cuprous bromide, and the end reaction is as sharp as in the titration of ferrous salts by permanganate.

In practice the copper compound may be in solution, if concentrated hydrochloric acid and some strong hydrobromic acid be added before titration, thus effecting considerable economy in consumption of the latter reagent. Reducing and oxidising agents must be absent. The test analyses with pure cupric sulphate are satisfactory.

—J. M. H. M.

*Chrome Iron.* S. Meunier. *Compt. Rend.* 110, 424—426.

By the action of a current of hydrogen on a mixture of ferrous and chromic chlorides contained in a red-hot porcelain tube, a silver-white alloy or compound of iron and chromium is formed, magnetic, and not attacked by hydrochloric or nitric acid and very slightly by aqua regia. It contains 35.01 per cent. of iron, and 64.80 per cent. chromium. When this substance is oxidised at a red heat by a current of steam it is completely converted into chromite, having all the properties of the native mineral and giving on analysis, ferrous oxide 32.41 per cent., chromic oxide 63.06 per cent.—J. M. H. M.



*Determination of Free Halogens and of Codides in presence of Chlorine and Bromine.* P. Lebeau. *Compt. Rend.* **110**, 520-522.

*Determination of Iodides.*—To the solution of iodide, contained in a 200 c.c. flask, are added 30–40 c.c. of carbon disulphide and a few drops of sulphindigotie acid. A titrated solution of bromine water is then run in from a stoppered burette until the colour of the indigo is just discharged; the iodine, as fast as it is liberated, dissolves in the carbon disulphide. The bromine water is titrated from time to time against standard potassium iodide.

*Determination of Free Halogens.*—The free halogen is shaken in a stoppered flask with water and zinc powder. The solution of neutral zinc bromide, chloride, or iodide, thus formed is titrated with standard silver nitrate, with potassium chromate as indicator. The bromine solution used in the previous process may be checked against pure silver nitrate in this way.—J. M. H. M.

*Action of Sodium Thiosulphate on Silver Salts.* J. Fogh. *Compt. Rend.* **110**, 709–711.

The reaction—

$2 \text{AgNO}_3 + \text{Na}_2\text{S}_2\text{O}_3 = 2 \text{NaNO}_3 + \text{Ag}_2\text{S} + \text{SO}_3$  takes place quantitatively. In an actual experiment the reaction was accompanied by the liberation of 46 eals. of heat. Two stages are observed in this reaction, a thiosulphate of silver is first momentarily formed and then decomposed into silver sulphide and sulphuric anhydride. It is calculated that 5.2 eals. are liberated in the first stage and 40 eals. in the second.

When an excess of sodium salt is present the silver thiosulphate is dissolved as fast as it is produced with the formation of a double salt and liberation of 34.8 eals.

The action of sodium thiosulphate on the haloid salts of silver is thus explained, since the decomposition of silver chloride, bromide and iodide is accompanied by the absorption of -26, -34.6, and -48.4 eals. respectively. The first two of these numbers being less than the heat of dissolution of the silver thiosulphate formed in sodium thiosulphate, the respective decompositions can readily take place without the aid of external energy.

For this reason solutions of the double thiosulphate of sodium and silver are precipitated by potassium iodide, but not by the chloride or bromide.—S. B. A. A.

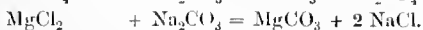
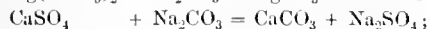
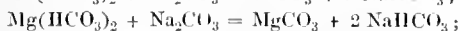
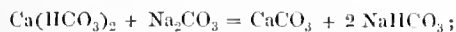
*Some Compounds of Mercury Chloride with Ammonia.*

G. Andre. *Bull. Soc. Chim.*, 1889, **2**, 145–153.

See under XX., page 647.

*Method of Analysis applicable to Water for feeding Boilers, to determine the amount of Anti-incrustation Agent necessary.* L. Vignon. *Bull. Soc. Chim.* 1889, **2**, 596–603. (See following Abstract.)

ROSSEL, in his report for 1888 to the Swiss Steam-Users Association (Zürich, Zürcher and Furrer, 1889), states that, theoretically, the best water for boiler use would be pure distilled water, rendered slightly alkaline. To render the water ordinarily available as suitable as possible, he recommends that sodium carbonate, and this substance alone, should be added to soften the water. The reactions which take place are—



The sodium bicarbonate formed is again decomposed at the temperature of the boiler and regenerates the carbonate. After the reaction the chlorides and sulphate of sodium remain in solution, while the carbonates of calcium and magnesium form a pulverulent and non-adherent precipitate.

An excess of sodium carbonate is dangerous as it would finally attack joints and taps.

We must therefore determine—

I. The quantity of carbonate necessary to combine with free carbon dioxide and to precipitate the bicarbonates of calcium and magnesium (this quantity being continually regenerated).

II. The quantity of carbonate necessary to precipitate magnesium chloride and calcium sulphate.

For this purpose a special method of analysis of the feed-water has been devised. The reagents required are—

(1.) A clear solution, containing 1.80 per thousand  $\text{Ca}(\text{OH})_2$  (saturated at 15° and decanted).

(2.) A solution containing 5 grms. of phenolphthalein in 100 cc. of 93 per cent. alcohol.

(3.) A neutral solution of calcium chloride containing about 5 per cent.

(4.) A solution of sodium carbonate containing 1 per thousand. The soda crystals to be used finally for softening the water may be employed instead of pure substance in making up this solution.

(1.) *Estimation of carbonic acid present in the free state, or combined with carbonates to form bicarbonates.*—50 cc. of distilled water and 10 drops of phenolphthalein solution are poured into a 100 cc. graduated cylinder provided with a glass stopper; 50 cc. of the feed-water, 10 drops of phenolphthalein and 5 cc. of the calcium chloride solution (to convert any magnesium bicarbonate into the calcium salt) are poured into a second cylinder, similar to the first.

The distilled water is then just coloured by adding 0.2 to 0.5 cc. of the lime-water from a burette, and lime-water is added gradually to the feed-water. The temporary colouration produced by each addition disappears quickly at first, but the reaction gradually slackens, and it is best to shake the solution well and to wait some little time between successive additions of the lime-water. When the colour of the feed-water has become constant (this takes place in less than an hour) and approximately equal in tint to that of the standard, the two are made exactly equal in volume and of the same colour by additions of water and lime-water.

The difference between the lime-water added to the standard and that added to the feed-water gives immediately the amount of free carbon or that combined with a carbonate to form a bicarbonate. If the analysis has been carried out precisely according to the above directions, and  $n$  be the number of cc. of the lime-water solution containing 1.80 per thousand  $\text{Ca}(\text{OH})_2$  required by the feed-water, the amount of pure  $\text{Na}_2\text{CO}_3$  per cubic metre required for purpose I. is theoretically 514  $n$  grms.; but the author recommends that this amount be augmented by 20 per cent. and increased to 616  $n$  grms., in order to allow for impurities in the commercial product and to make the water slightly alkaline. If  $V$  be the mean volume of water contained in the boiler while working, expressed in cubic metres, the weight of  $\text{Na}_2\text{CO}_3$  to be added once for all, after the boiler has been cleaned, is therefore 616  $nV$  grms. for purpose I.

(2.) *Determination of  $\text{Na}_2\text{CO}_3$  required to precipitate the metals present as chlorides and sulphates.*—100 cc. of the feed-water is poured into a 250 cc. graduated cylinder. The amount of lime-water necessary to neutralise exactly is added, then 5 cc. of the phenolphthalein solution. The liquid should remain colourless after being shaken up. It is then filtered and allowed to settle, and half the total volume (corresponding to 50 cc. of the feed-water) is introduced into a 500 cc. graduated cylinder. A titrated solution of sodium carbonate is then added till the colour becomes distinctly red; it is useful to compare with a standard made by adding the same quantity of phenolphthalein and 0.3 to 0.5 cc. of the  $\text{Na}_2\text{CO}_3$  solution to an equal volume of distilled water. As a rule, the results obtained are very definite; but when the water contains much magnesium chloride and magnesium sulphate (which rarely occurs) the end reaction is not well marked.

If  $V'$  be the average consumption in cubic metres per 24 hours, and  $x$  the number of grms. of sodium carbonate ( $\text{Na}_2\text{CO}_3$ ) required per cubic metre under head  $H$ , then each day  $Vx$  grms. of sodium carbonate must be introduced into the boiler before getting up steam.—P. J. H.

*On a new Method of Analysis applicable to Water for Manufacturing Purposes, and for feeding Boilers.*  
L. Vignon. Bull. Soc. Chim. [3], 3, 2—4. (See preceding Abstract.)

THREE notes have previously been published on this subject (Bull. Soc. Chim. [3], 1, 903; this Journal, 1889, 138, and above). Two improvements in the details of the analysis are now suggested.

(1.) Ordinary distilled water may contain as much as 5 cc. of  $\text{CO}_2$  per litre; it is advisable, therefore, to prepare the two standard-tint solutions with distilled water which has just been boiled gently for 15 minutes in a platinum or porcelain evaporating dish. The use of glass would vitiate the results by introducing alkali.

(2.) In operation (2) above, it is more convenient and expeditious to get rid of the excess of  $\text{CO}_2$  by boiling in the way just described, than to precipitate with lime water.

—P. J. H.

*Electrolytic Separations.* E. F. Smith and L. K. Frankel.  
J. Franklin Inst. 1890, 129, 236—247.

(1.) *Cadmium from Cobalt and Nickel.*—Cadmium may be completely separated from cobalt. On passing a current yielding 0.4 cc. of  $\text{O}_2$  gas per minute for 14 hours into solutions of these metals (0.1690 gm. of each in 200 cc. water) containing 4.5 grms. of potassium cyanide, the whole of the cadmium is deposited without any admixture of cobalt. The separation of cadmium from nickel was not found possible with any approach to accuracy.

(2.) *Mercury from Zinc, Nickel and Cobalt.*—Mercury may be satisfactorily separated from zinc; solutions of these metals (0.2440 gm. each in 200 cc. water) containing 3—4.5 grms. of potassium cyanide, deposit the whole of the mercury ( $\pm 0.2$  per cent.) when treated for 16 hours with a current yielding 0.5 cc.  $\text{O}_2$  gas per minute. The separation of mercury from nickel is only approximate; solutions of the same strength as before containing 4.5 grms. of potassium cyanide and treated for 16 hours with a current of 0.4 cc. gave results from 0.2 to 0.6 per cent. too low.

The separation of mercury from cobalt is attended with still greater difficulty; solutions of mercury (0.225 gm. in 200 cc. of water) and 25—75 per cent. of cobalt, containing 3 grms. of potassium cyanide gave the best results on treatment for 16 hours with a current of 0.5 cc., the error being from  $+0.26$  to  $+0.35$  per cent., with equal quantities of mercury and cobalt and 2.2 grms. of potassium cyanide, the error was  $-0.60$  per cent.

(3.) *Silver from Copper, Zinc, Nickel, and Cobalt.*—In these cases it is only by the use of extremely feeble currents that even an approximate separation can be effected.

In solutions of silver and copper (0.1788 gm. of each in 200 cc. water) containing 4.5 grms. of potassium cyanide, and heated for 16 hours with a current of from 0.15 to 0.40 cc.; the error varied from  $-0.33$  to  $+0.60$  per cent.

No difficulty is experienced in separating silver from zinc; in solutions containing zinc instead of copper and treated in the same way, the error varied from  $-0.05$  to  $-0.5$  per cent. Equally good results were obtained from solutions containing nickel.

The separation of silver from cobalt is approximately complete ( $\pm 0.33$  per cent.) if the amount of potassium cyanide is reduced to 3 grms. for the same amount of metal as before.

The success of the above separations depends upon the exact proportion of potassium cyanide to the metals in solution, and upon the use of the proper current strength.

(4.) *Copper from Cadmium in the presence of Sulphuric Acid.*—The best results were obtained when a solution of copper sulphate (0.1975 gm. of copper) and cadmium

sulphate (0.1828 gm. of cadmium) in 130 cc. of water and 15 cc. of sulphuric acid (sp. gr. = 1.09) is subjected to a current of from 0.2 to 0.3 cc. The error varies from  $+0.05$  to  $-0.65$  per cent. (See also this Journal, 1889, 639, and 1890, 108.)—S. B. A. A.

*The System of Amalgamation of Gold Ores in use at the Massachusetts Institute of Technology.* R. H. Richards.  
Technology Quart. 3, 45—51.

THE object of the system is to make exact quantitative experiments with a small stamp battery. The difficulties to be overcome were in the first place to avoid loss or gain of gold, which always occurs with amalgamated copper plates, a portion of the gold being always retained in the amalgam remaining on the plates. The copper plates first used were  $\frac{1}{32}$  inch thick, and after each operation the plates were dissolved up. This necessitated new copper plates for each operation. As new copper plates amalgamate badly at first until a certain quantity of gold has been taken up, this second difficulty is overcome by amalgamating the plates to begin with, by substituting a silver amalgam for the gold amalgam, and subsequently separating the silver. The silver amalgam is made by dissolving silver in nitric acid diluted with an equal weight of water, and then adding about ten times as much mercury as silver. The silver is all precipitated, forming a pasty amalgam. The amalgam is washed with water, squeezed through chamois skin until it has the consistence of soft putty, and is then applied to the bright surface of the plates with a hog's bristle brush. The author finds that the use of silver amalgam does away with the necessity of dissolving up the plates, as the gold is deposited only on the surface of the amalgam and can be completely cleaned off. The amalgam on the plates should not be allowed to get too hard or too soft. If too hard it may fail to catch the gold; if too soft, mercury will flow at the lower end of the plates. A quantity of mercury varying from 1 to  $1\frac{1}{2}$  oz. should be fed to the stamps for every oz. of gold contained in the ore under treatment. This mercury should be fed a little at a time every half hour. If the plates are inclined to stain, a lump of cyanide of potassium held a moment on the spot will remove it. If an ore contains  $\text{FeSO}_4$  or  $\text{CaSO}_4$ , as a result of oxidation of iron and copper pyrites a little lime should be added along with the ore. The use of oil in the mill should be restricted as this also causes the yellow stain.—J. B. C.

*Some Points in the Determination of Silica in Silicates by Fusion with Alkaline Carbonates.* J. P. Gilbert.  
Technology Quart. 3, 61—68.

THE author used a sample of blast-furnace slag containing alumina and lime and a little magnesia, and first determined the influence of temperature employed in evaporating off the  $\text{HCl}$  to render the silica insoluble after fusion. The temperatures employed were those of the water-bath,  $125^\circ$ , an iron plate over a Bunsen burner and  $280^\circ$ . The results show that the silica was practically rendered insoluble at the temperature of the water-bath, but the amount of foreign matter in the silica was in general perceptibly higher at the higher temperature of dehydration. This is probably due to the alumina being rendered partially insoluble in acid. The next point determined was the effect of salts such as magnesium chloride, which decompose easily, and which might form insoluble silicates at a comparatively low temperature. The slag used for these determinations contained 35 per cent. of lime, 45 per cent. of magnesia, and 15 per cent. of alumina. This series of results differs from the first, mainly in the large amount of impurity in the silica and the large amount of silica in the alumina when the dehydration is effected at a temperature of  $280^\circ$ . The best results were obtained at a temperature of  $120^\circ$ . The increase of silica in the alumina as the temperature increased would seem to be due to the magnesia, for the conditions were in all other respects the same as in the analyses of the other slag. These results indicate that there is no danger of the silica combining

with the magnesia at  $120^{\circ}$ , and that at this temperature the silica is almost completely dehydrated, the calcium chloride probably assisting in the dehydration.

Another series of analyses was made with orthoclase practically free from lime and magnesia. This series differs from the previous one in that the higher temperature is not accompanied with an increased amount of silica in the filtrate. This would seem to furnish a confirmation of the view, that there was a recombination of the silica and the magnesia at the temperature of  $280^{\circ}$  in the magnesian slag; and the fact that the amounts of silica found in the alumina are higher than those in the lime slag indicates the beneficial effect of the calcium chloride on the dehydration of the silica. The author further found that owing to incomplete separation of silica from an alkaline silicate at  $120^{\circ}$ , the amount of silica in a given sample decreases with each successive fusion. A series of experiments was made by Lindo's method. This consists in largely diluting after extracting the fused mass so that on the addition of HCl no precipitation occurs. In this way he obtained on evaporation what he called "vitreous silica." Samples of glass were analysed as follows:—The powdered glass (1 grm.) was fused with five parts of sodium and potassium carbonates and the mass treated with 400 cc. of boiling water, on acidifying with dilute HCl, a clear solution was obtained. This was evaporated to dryness on a water-bath, heated to  $125^{\circ}$ , and the silica thus obtained fused and treated as before. The silica obtained by the second fusion was weighed and treated with HF. The two filtrates were combined and evaporated to dryness, heated to  $125^{\circ}$  for one hour, and the residue insoluble in HCl filtered off. In this case it was found that a considerable portion of silica fails to be dehydrated by one evaporation and heating to  $125^{\circ}$ . The silica is very pure owing probably to it being easily washed, or it may be due to the absence of any large quantity of alumina.

The author has further made a series of determinations, which seem to justify the inference that in presence of much alumina it is not possible to get as pure silica as is easily obtained when only salts of the alkalis or alkaline earths are present. When evaporation is carried out in presence of free sulphuric acid the results are more satisfactory, but the amount of alkaline sulphates introduced seriously interferes with the determination of the bases present. These results seem to justify the recommendation that has been made, viz., that in the analysis of silicates the silica is best determined by dehydration with sulphuric acid when it is admissible, and that the bases should be determined after the decomposition of the silicate by hydrofluoric acid.

—J. B. C.

*A New Reagent for Cane Sugar, Grape Sugar, and Pyrogallol.* E. Mathieu-Plessy. Bull. Soc. Ind. Mulhouse, 1890, 69—72.

THE new reagent is prepared by heating together to  $200^{\circ}$  a mixture of 45 parts of ammonium nitrate, 34 of lead nitrate, and 21 of lead oxide. The fused mass is cast in slabs. Its melting point, namely  $102^{\circ}$ , is lower than that of any of its constituents. It contains lead para-nitrate, which, when the mass is melted, dissolves in the excess of ammonium nitrate. To apply the test, the reagent is gently heated till melted, and then a small quantity (say 5 mgrms.) of the substance to be tested dropped on to it. The following characteristic colourations are so obtained: with glucose a cherry red, with cane sugar a light brown, and with pyrogallol a chrome-green.—E. B.

*On Commercial Turpentine Oil.* A. Wilson. Chem. Trade J. 6, 316. (Compare this Journal, 1890, 330 and 557.)

ADULTERANTS of oil of turpentine are petroleum and shale spirit, coal tar naphtha, rosin oil and rosin spirit, while an equally dishonest addition is that of the inferior Russian oil of turpentine to the French or American product. The author records the limits within which the samples he has examined ranged. Sp. gr. varied between 0.862 and 0.870. The specific rotatory power is not conclusive, although a

sample giving a high value is certainly pure. The maximum value for the D line obtained during the examination of 20 samples was  $+15.29$ , and the minimum  $+12.05$ , the instrument used being a Laurent. French oil of turpentine is levo-rotatory.

Rosin oil may be detected by steam distillation and subsequent treatment of the residue with stannic bromide, which yields a violet colour, or with acetic anhydride and sulphuric acid, giving a red or reddish violet colouration. The halogen absorption of genuine oil of turpentine is about 215 for bromine and 331 for iodine, while petroleum and shale spirit absorb 30 and 70 per cent. of bromine respectively. The behaviour on distillation, especially when a dephlegmator is used, is useful in detecting shale and petroleum spirit, the boiling point of a genuine sample generally lying between  $156^{\circ}$ — $160^{\circ}$  C. Armstrong's method (this Journal, 1890, 557,) for the detection of petroleum spirit by polymerisation of the turpentine with sulphuric acid and distillation in a current of steam is useful, but not applicable for the recognition of rosin spirit, it being attacked by sulphuric acid.—B. B.

*On the Oxidising and Decolourising Properties of Charcoal.* P. Cazeneuve. Compt. Rend, 110, 788—799.

THE decolourising properties of charcoal, animal charcoal, resin charcoal, wood charcoal, &c., are generally attributed to the mechanical fixation of the colouring matter in their pores in somewhat the same way that silk takes a dye. The author has made experiments which tend to show that charcoal, and particularly animal charcoal, have powerful oxidising properties. Calcined and washed animal charcoal placed in solutions of  $\alpha$ -naphthylamine or *p*-phenylenediamine developed characteristic colours produced by the oxidation of these bases. Other similar experiments were made with the same result. Charcoal heated in a current of nitrogen and allowed to cool in that gas has much less powerful decolourising properties than the same charcoal allowed to cool in the air. The colour taken up from dark solutions by charcoal cannot be recovered if the action has taken place some hours prior; it is in fact destroyed by oxidation.

—A. L. S.

*The Colouring Matter of Wines.* Monnet. Bull. Soc. Chim. (1889) 2, 144.

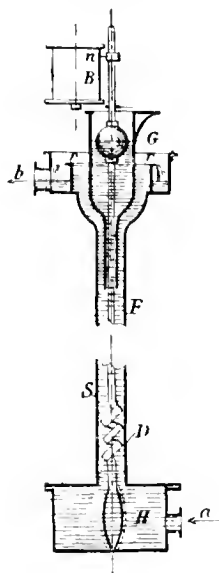
MANY metallic sulphides extract the colouring matter from wine like charcoal. As a rule the colouring matter can only be extracted from the sulphides by alcohol acidified with sulphuric acid. In the case of arsenic sulphide, alcohol alone takes up the colouring matter. The alcoholic extract leaves on evaporation a violet colouring matter, soluble in alcohol and glycerol. If the colouring matter is extracted from the sulphide of arsenic by means of alcohol acidified with acetic acid, the residue on evaporation is only partly soluble in alcohol, and if sulphide of lead be substituted for the arsenic, the blue colouring matter obtained is insoluble in alcohol. The author considers it incorrect to suppose the blue colouring matter to be a normal constituent of wine.—J. B. C.

*Automatic Determination of the Density of Sugar Juice.* Spencer's Handbook for Sugar Manufacturers, p. 4.

Having determined the volume of the juice for a stated period, it is necessary to ascertain the total weight, or the average weight of a unit volume. For this purpose the mean specific gravity must be known. This can either be determined automatically or by the chemist. The latter method is perhaps the more certain of giving accurate results, although the apparatus described below is highly recommended by a prominent beet-sugar manufacturer of Germany for the accuracy of its work. If the sampler is used to collect the juice for the chemist's examination for

density, no lead should be employed. A considerable fermentation affects the density determination but little, hence samples may be stored several hours, if necessary, before their examination.

The automatic determination of the density of the juice depends upon the principle of communicating vessels. A column of juice of an invariable height counterbalances a column of water of a height proportional to the density of the juice (see Figure). By means of a suitable connexion, the small reservoir II is connected with the measuring



tank; the juice from each charge drawn into this latter passes through the reservoir and into the tube S, and overflows at *r*. Inside the tube S is another tube F D, which terminates above in a funnel shaped vessel and below in a flexible bulb F. The interior of this tube, including the bulb, is filled with water, whose height is registered upon a cylinder B by means of a float carrying a pencil. It is evident, from an inspection of this apparatus, that the water in the inner tube will rise in proportion to the density of the juice surrounding and pressing upon the flexible rubber bulb. This rise in the level of the water is registered by the pencil, carried by the float, upon the paper-covered cylinder. This cylinder is revolved by clockwork, making one revolution every 12 hours.

The variable temperatures of the juice have no influence upon the apparatus, provided the column of water is of the same temperature as the juice surrounding it. For this reason the tube F is spiral at its lower end. E. Langen, the inventor of this apparatus, has lately substituted a bundle of fine copper tubes for the spiral. Foam and mechanical impurities do not affect the accuracy of the apparatus. The density is recorded in degrees Brix or Baumé, as preferred.

This apparatus may be attached to the upper or lower tank of the measuring apparatus. A double ball valve should be used to admit the juice and prevent overflow and waste.

#### Schmidt's Process for Converting Oleic Acid into Solid Fatty Acids. R. Benedikt. Monatsh. 1890, 11, 71—83.

SOLID fatty acids, such as stearic and palmitic, are separated from liquid fatty acids such as oleic, by pressing the mixed products of the hydrolysis of tallow and palm oil, and used largely in candle making. The liquid fatty acids are a less valuable by-product, and it is therefore desirable to increase the yield of the solid fatty acids as much as possible. It is found that when the sulphuric acid process of saponification is used, the yield is 15—17 per cent.

greater than is obtained by saponification with lime or high pressure steam (*Stas*), and the author has actually found 15.5 per cent. of iso-oleic acid (discovered by Saytzeff, J. prakt. Chem. [2], 37, 269; this Journal, 1888, 442) in a sample of solid fatty acids produced by this method, showing that the oleic acid undergoes isomeric change to this extent (this Journal, 1886, 452).

Various plans have been proposed to enhance this effect but with small success. Elaidic acid is only formed regularly from *fresh* oleic acid by the action of nitrous acid, and besides is a bad material for candles. Neither the conversion of oleic acid into palmitic acid by fusion with caustic alkalis as proposed by Farrentropp, nor its transformation into stearic acid by heating with a small quantity of one of the halogens according to the process of Wilde and Reyhler, is a practical success. Schmidt heats 10 parts of oleic acid with one of zinc chloride to 180° C., washes first with dilute hydrochloric acid, then with water, and distils in a current of superheated steam, pressing the distillate in the usual manner. It is this process which the author has studied. His raw material was a sample of oleic acid made from beef-tallow without admixture of vegetable fats (as these constantly contain linoleic acid), the purity of which was ascertained by its giving the right iodine number and a lead salt completely soluble in ether. Five hundred grms. of it were heated with 50 grms. of zinc chloride in an oil-bath furnished with a thermostat, the temperature being kept at 180°—185° C.—an important condition, as if 195° C. be exceeded no solid product is obtained. Reaction sets in at 180° C., accompanied by slight frothing from the escape of steam, and the zinc chloride speedily dissolves. A drop of the mixture is withdrawn from time to time, boiled with dilute hydrochloric acid, and cooled, and when it becomes solid on cooling the operation is stopped, dilute hydrochloric acid added to the contents of the flask, and the mixture boiled by blowing in steam, the process being repeated until zinc can no longer be detected in the washings, when after a final washing with water, the fatty acids are distilled under a diminished pressure of 120—150 mm., that plan being more convenient than the use of superheated steam when working on a small scale. A greater reduction of pressure is not advisable, as frothing is then apt to occur. The distillate is yellowish-white, and from it is easily separated a white crystalline solid portion by spreading it on a porous tile. The research divided itself into three parts:—

#### I. Investigation of the Washed Product before Distillation.

—A knowledge of the composition of such fatty mixtures is best obtained from the determination of the figures they give for their "acid number," "saponification number," so-called "ether number" (by which is meant the difference between the first two, corresponding to the amount of alkali consumed in saponifying the glycerides present in an ordinary fat), "acetyl number," and "iodine number," rather than by any attempt to isolate their constituents. In this case the mixture was remarkable as containing a notable proportion of what the author terms "unsaponifiable anhydrides," which are only attacked by alcoholic potash when a temperature of 150° C. was reached, and which he considers to consist chiefly of the anhydride of hydroxystearic acid, besides a considerable quantity of "saponifiable anhydride," found to be identical with the *stearolactone* of Geitel, which, though saponifiable, is regenerated as anhydride on being freed by hydrochloric acid, and does not pass into the corresponding acid. From these results, by a close and ingenious chain of reasoning, too long to produce verbatim, and impossible to compress, the author arrives at the conclusion that the composition of the mixture is:—

	Parts per Cent.
"Fluid unsaponifiable anhydride" (the anhydride of hydroxystearic acid).....	8
Stearolactone (the lactone of $\gamma$ -hydroxystearic acid).....	28
Hydroxystearic acid.....	17
Oleic acid.....	40
*Saturated fatty acids.....	7
	<hr/> 100

\* Calculated acid number for these = 193.

**II. Investigation of the Crude Distillate.**—An unsaponifiable portion was present, but instead of consisting of unsaponifiable anhydrides as in I., was found to contain only a small quantity of oxygenated compounds and to be mainly composed of hydrocarbons. The results are:—

	Parts per Cent.
Unsaponifiable matter (fluid) .....	13.6
Oleic and iso-oleic acids .....	43.3
Stearolactone .....	31.0
Saturated fatty acids .....	12.1
	<u>100.0</u>

Determination of the respective quantities of oleic and iso-oleic acid was not practicable, Saytzeff's method giving unsatisfactory results.

**III. Investigation of the Solid Portion of the Distillate.**—Separation of the valuable solid products is effected on the large scale by hot or cold filter pressing, but for experimental purposes the use of a porous tile sufficed. The product was a hard crystalline mass with a melting point of 41°–42° C., which, when examined by the method indicated under I., was found to contain:—

	Parts per Cent.
Stearo-lactone .....	75.8
Iso-oleic acid .....	15.7
*Saturated fatty acids .....	8.5
	<u>100.0</u>

\* Mean molecular weight = 216.

From this it appears that the action of zinc chloride on oleic acid is analogous to that of sulphuric acid; two isomeric addition products with zinc chloride are formed, and these decompose on heating with dilute hydrochloric acid into zinc chloride and two hydroxystearic acids, identical with those found by Geitel to be produced by the action of sulphuric acid on oleic acid (this Journal, 1888, 218). One of these becomes stearo-lactone with elimination of water, the other becoming converted into its anhydride, which is difficultly saponifiable (needing a temperature of 150° C.). A portion of these hydroxystearic acids suffer change on distillation, yielding oleic acid and solid iso-oleic acids, which appear in the distillate as shown above.—B. B.

**The Analysis of Resins and Balsams.** M. Bamberger. Monatsh. 1890, **11**, 84–86.

BENEDIKT and GRÜSSNER, having already published a series of determinations of the "methyl number" of sundry ethereal oils (Chem. Zeit. **13**, 53; this Journal, 1889, 925), the author supplements their observations with those of certain resins and balsams.

Name of Resin or Balsam.	Methyl Number.	Remarks.
Aloes ( <i>Alöe hepatica</i> ) .....	3.9 ..	
Aloes ( <i>Alöe lucida</i> ) .....	0 ..	
Ammoniacum .....	11 ..	Persia.
Asafetida .....	18 ..	
Gum benzoin ( <i>Siam</i> ) .....	28.5 30	
Gum benzoin ( <i>Sumatra</i> ) .....	16.5 16.2	
Gum benzoin ( <i>Sumatra</i> ) ( <i>Almond</i> ) ..	13.3 ..	
Bellium .....	0 ..	Africa.
Canada balsam .....	0 ..	
Copaiba balsam .....	0 ..	
Copal .....	0 ..	Zanzibar.
Colophony .....	0 ..	
Gum dammar .....	0 ..	
Dragon's blood .....	33.8 ..	Sundainsel.

Name of Resin or Balsam.	Methyl Number.	Remarks.
Euphorbium .....	0 ..	Africa.
Gum elemi .....	0 ..	
Resin ( <i>Pinus taeda</i> ) .....	0 ..	
Resin ( <i>Pinus halepensis</i> ) .....	0 ..	South Austria.
Resin ( <i>Pinus laricio</i> ) .....	49.6 50.9	
Galbanum .....	3.7 4.0	
Gamboge .....	0 ..	Lower India.
Guaicum .....	83.8 84	
Jalap .....	0 ..	
Kauri dammar .....	7.1 ..	
Ladanum .....	0 ..	
Liquidambar .....	0 ..	
Myrrh .....	13.6 13.2	
Mastic .....	0 ..	
Olibanum ( <i>Frankincense</i> ) .....	5.3 ..	Africa.
Oppoponax .....	9.9 ..	Asia Minor.
Balsam of Peru .....	14.4 ..	
Storax calamitus .....	0 ..	Asia Minor.
Scammony .....	0 ..	Smyrna.
Sandarac .....	0 ..	Asia Minor.
Shellac .....	0 ..	
Tacamahac .....	0 ..	
Venice turpentine .....	0 ..	
Balsam of Tolu .....	46.8 ..	
Xanthorrhoea resin ( <i>Yellow</i> ) .....	26.4 ..	
Xanthorrhoea resin ( <i>Red</i> ) .....	32.7 ..	

The numbers in the third column are those of duplicate determinations.

B. B.

**Volumetric Estimation of Tannin.** E. Guenz. Compt. Rend. **110**, 532–534.

A STANDARD solution is made of tartar emetic, 12 grms., and Poirier's green 4.16, 1 gm., to 1 litre of distilled water. The solution of tannin to be estimated is placed in a burette and run into 20 cc. of the boiling standard coloured liquid until complete decolouration just occurs. The standard solution is titrated against a solution of pure oak gall tannin of known strength. Gallie acid does not interfere with this process.—J. M. H. M.

**The Estimation of the Tannic Acids in Sumac.** L. Bruehl. See under VI., pages 607–608.

**Estimation of Acetone in Wood Spirit.** L. Vignon. Compt. Rend. **110**, 534–536.

IN applying Krämer's process, founded on Lieben's iodoform reaction, to the determination of the acetone present in the crude methyl alcohol used for the preparation of methylated spirit, it is necessary to have the iodine present in large excess, and to conduct the operation in the following way: 5 cc. of the wood spirit are dissolved in 200 cc. of distilled water, and the volume made up to 250 cc.; 5 cc. of this mixture are placed in a stoppered and graduated cylinder with 10 cc. of binormal soda and, after shaking, 5 cc. of binormal iodine are added, and the mixture again shaken; 10 cc. of ether free from alcohol are then added to take up the iodoform, and the volume of the ethereal solution noted.

5 cc. of the ethereal solution are pipetted off and evaporated in vacuo, and the iodoform weighed as soon as possible.  $\text{CHI}_3$  corresponds to  $(\text{CH}_3)_2\text{CO}$ . Aldehyde, ethyl alcohol, and other bodies capable of furnishing iodoform must be absent (see following abstract).—J. M. H. M.

*Estimation of Acetone by Iodoform.* E. Arachequesne. *Compt. Rend.* **110**, 642—644.

KRÄMER'S method (see preceding abstract) for the estimation of acetone, based on Lieben's reaction, was intended only for determinations in methyl alcohol, used in the manufacture of methylaniline, containing, at most, 1 per cent. of acetone. With a view to making the method generally useful, the author makes the following proposals:—1. That Krämer's method, as it is, should be used only with pure methyl alcohol. 2. When the liquid to be analysed contains 1·5 to 30 per cent. of acetone, a smaller amount should be employed. For example, with liquids containing 20 to 30 per cent. of acetone, take 5 cc., dilute with water to 500 cc. and of the diluted solution take 5 cc. for the analysis, and multiply the result by 20. 3. With solutions still richer in acetone, 5 cc. is diluted with water to 50 cc. Of this solution 5 cc. is put into a 200 cc. flask, the bottom of which terminates in a point closed with an india-rubber tube and a clamp. Binormal soda (10 cc.) and binormal iodine solution (5 cc.) are added, and the whole shaken. The addition of the soda and iodine solutions (in the same proportions) is then continued until no more iodoform is precipitated. The iodoform is then washed into a burette, having a plug of asbestos at the bottom, and after the excess of solution has run through, is dissolved in ether and weighed as described by Krämer.—N. H. J. M.

*Estimation of Fat in Milk.* Lezô. *Compt. Rend.*, **110**, 647—649.

THE separation of fat in milk is hindered by the casein which retains the globules of butter. The difficulty is overcome by dissolving the casein in acid. The milk is shaken with strong hydrochloric acid (2 to 2·5 parts) and poured into a flask with a long neck graduated in cc. and tenths of cc. It is then heated until the liquid becomes brown, treated with dilute ammonia until the oil is completely separated, and hot water added until the water level reaches the graduated neck of the flask. The volume of melted fat is then read and the number of cc. multiplied by 0·90 (the specific gravity) gives the weight in grms. This correction may be avoided by taking, for instance, 110 cc. of milk instead of 100 cc.; the number of cc. of fat then represents grms. per cent. 44 cc. of milk and 100 cc. of acid are convenient amounts to employ. The mixture is heated to about 80°.

—N. H. J. M.

*The Analysis of the Methylanilines.* H. Giraud. *Bull. Soc. Chim.* 1889, **2**, 142—144.

THE method is based upon that of Reverdin and de la Harpe (this Journal, 1889, 422), and consists in adding acetic anhydride to methylaniline and determining the amount of acetic acid formed. The author, however, instead of employing acetic anhydride alone, employs a mixture of the anhydride with 10 times its volume of dimethylaniline. This mixture keeps better than the anhydride, and the dimethylaniline has no influence on the reaction. The solution is titrated against a standard solution of baryta, litmus or phenol phthalein being used as indicator. The analysis is conducted in the following manner. 1 gm. of the mixed methyl anilines and 10 cc. of the anhydride mixture are brought together in a flask, which is closed with cork. After an hour water is added and the liquor titrated with baryta. The difference between the first and second titration gives the amount of methylaniline or aniline present.—J. B. C.

*On Benedikt's Acetyl Values.* J. Lewkowitsch. *Proc. Chem. Soc.* 1890, 72—74; also 91—92.

IN his paper on a new method of examining fats (*Zeits. für die Chem. Ind.* 1887, 148), Benedikt proposes a method

which is based on the principle that only hydroxylated fatty acids take up the radicle of acetic acid when heated with acetic anhydride, and he asserts that those fatty acids which do not contain the hydroxyl-group remain unaltered; so that the quantities of potash required for neutralising fatty acids before and after treatment with acetic anhydride will be either identical or nearly so, any difference being due to the errors of the method of determination.

While examining a somewhat abnormal sample of tallow, the author was led to doubt the accuracy of this statement, and the following results of experiments which he has made serve to confirm his doubts.

The fatty acids prepared from a sample of fresh town tallow required for neutralisation 19·717 per cent. of potash, or, in other words, their "acid value" was equal to 19·717, corresponding to the molecular weight 284·5. 50 grms. of these acids was acetylated with 40 grms. of acetic anhydride in the manner indicated by Benedikt, and both the acid value and the "saponification" value of the acetylated product was then determined. The values found were 160·5 and 217·5 respectively, corresponding to an acetyl value of  $217·5 - 160·5 = 57$ . A second test gave  $216·3 - 159·9 = 56·4$ .

In the same way the fatty acids of samples of South American tallow and of North American tallow gave the acetyl values 77·8 and 23·93. According to Benedikt the acetyl values ought to have been *nil* or nearly so.

As tallow is considered to consist essentially of a mixture of the glycerides of stearic, palmitic, and oleic acids, it was thought possible that it was the oleic acid which had fixed the acetic acid radicle. A sample of oleic acid obtained from Kahlbaum as pure was therefore examined; it contained, however, some solid acids, which settled out when the acid was kept for some time in a stoppered bottle. To determine the impurities, the acid value and iodine absorption were ascertained: the acid value found was 183, and the iodine absorption 90·14; the theoretical values for oleic acid are 198 and 90·07. Although containing a certain amount of higher homologues, the acid was considered sufficiently pure for the desired purpose: 50 grms. of it when acetylated gave an acid value = 116·50 and a saponification value = 242·05; consequently an acetyl value = 125·55. If the fixing of the "acetyl," however unlikely it might appear, were due to the  $\text{CH}_2\text{CH}$ -group in the molecule of the oleic acid, the acetylated acids ought to have lost the capacity of taking up iodine. However, the iodine value of the acetylated acids was found to be 87·11, proving clearly that acetyl was not taken up by the  $\text{CH}_2\text{CH}$ -group. Therefore, the acetic anhydride must have acted in some other way on the oleic acid, and if so it was not unlikely that palmitic or stearic acid might be acted on in the same way. Experiment fully bore out the correctness of this assumption.

Fifty grms. palmitic acid, the comparative purity of which was proved by an acid value of 213·4 (instead of the theoretical value 219·1), were acetylated and gave an acid value = 143·53 and a saponification value = 226·13; consequently an acetyl value = 82·6.

Fifty grms. stearic acid—showing an acid value of 203 instead of the theoretical one of 197·5—gave, after being acetylated, an acid value = 138·89 and a saponification value = 221·18; consequently an acetyl value = 82·29.

These results were so unexpected that it was determined to verify them by examining other fatty acids: capric, lauric, and erotic acids were therefore acetylated in the manner previously stated. The approximate purity of the acids used was ascertained by determining the quantity of caustic potash required for their saturation.

*Capric Acid.*—The acid value was found to be 318·65, while theory requires 326·2. The acetylated product gave an acid value of 176·4, and a saponification value of 350·4; consequently an acetyl value = 174.

*Lauric Acid.*—The acid value found was 273·02, the theoretical value being 280·5. The acetylated acid gave an acid value = 161·5, a saponification value = 293·99; its acetyl value therefore was 132·49.

*Cerotic Acid.*—The acid value found was 128·4; theory indicates for  $\text{C}_{26}\text{H}_{52}\text{O}_2$  141·6 (or for  $\text{C}_{27}\text{H}_{54}\text{O}_2$  136·8). The

acetylated cerotic acid gave an acid value = 73.87, a saponification value = 142.1; so that the acetyl value was 68.23.

From the approximate coincidence of the acetyl value of capric acid (174) with the acid value of the acetylated acid (176.4), it might be inferred that the acetylated capric acid contains one acetyl-group, but an acid of the formula  $C_{16}H_{31}O_2 \cdot C_2H_5O$  has a theoretical acid value of 262. Similarly the value for a mono-acetyl derivative of lauric acid would be 231, and that for a mono-acetyl derivative of cerotic acid 128 (or otherwise 124). (The same formulae would apply to mixed anhydrides of acetic acid and capric acid, &c.) It was easy to decide whether the action of acetic anhydride on fatty acids affected the  $COOH$  group of the latter, for in that case an alcohol of the  $C_nH_{2n+2}O$  series ought not to become acetylated. The author experimented on cetyl alcohol, which was treated with acetic anhydride. The acid value of the resulting substance was—as is to be expected—*nil*; the saponification value found was 192.65. As a substance of the formula  $C_{16}H_{33}O \cdot C_2H_5O$  has theoretically a saponification value = 198, it is evident that simply etherisation of the cetyl alcohol has taken place.

It was therefore to be supposed, as the mixed anhydrides of the higher fatty acids and acetic acid could not have been formed, that by the interaction of acetic anhydride and the higher fatty acids the anhydrides of the latter had been produced—taking palmitic acid as an example—according to the following equation—



In that case the quantities of caustic potash required by the equation  $(C_{16}H_{31}O)_2O + 2KOH = 2C_{16}H_{31}O \cdot OK + H_2O$  ought to agree with the saponification values found. The following table gives the quantities of caustic potash required by theory, in milligrammes, compared with the quantities actually used in the above experiments:—

	Mol. wt.	Theory.	Expt.
Capric anhydride $(C_{10}H_{19}O)_2O$ ....	326	344	350.4
Lauric anhydride $(C_{12}H_{23}O)_2O$ ....	242	294	293.90
Palmitic anhydride $(C_{16}H_{31}O)_2O$ ..	494	227	226.13
Stearic anhydride $(C_{18}H_{35}O)_2O$ ....	550	204	221.18
Cerotic anhydride { $C_{26}H_{51}O)_2O$ ...	774	145	} 112
	$C_{27}H_{53}O)_2O$ ...	818	137
Oleic anhydride $(C_{18}H_{33}O)_2O$ ....	546	205.4	242

Considering the approximate purity of the acids used, the theoretical values agree very well with the experimental values, and it may be pointed out that with pure material it would be easy by this method to determine which is the formula of cerotic acid.

In the light of this explanation, the "acid values" found for the products of the interaction of acetic anhydride and fatty acids lose every quantitative meaning; as the "acetylated" acids were dissolved in cold absolute alcohol for titration with potash, which at once hydrolysed the anhydrides, hydrolysis ceasing only when a limit was reached which depended on the quantity of alcohol present, and the nature and dilution of the standard solution—in some experiments half normal soda, in others decinormal potash. Had the substances been shaken up with water (hot water does not decompose them), on the first drop of potash falling into the mixture the pink colour would have appeared at once, or very soon when the limit for the system of substances was reached. Experiments carried out in this direction fully bear out the correctness of the author's conclusions. Thus the interaction of  $KOH$  or  $NaOH$  and the anhydrides in aqueous solution affords an elegant illustration of that class of actions which require a measurable time for their completion. The anhydrides of the higher fatty acids are now within easy reach, as they can be prepared in a very short time by means of acetic anhydride.

## ERRATUM.

In the May number of this Journal, page 557, line 7 from top of column 1, for "very oxidisable bodies" read "not very oxidisable bodies."

## New Books.

A TREATISE ON CHEMISTRY. By SIR H. E. ROSCOE, F.R.S., and C. SCHORLEMMER, F.R.S. Vol. III.—The Chemistry of the Hydrocarbons and their Derivatives, or Organic Chemistry. PART II.—New and thoroughly revised edition. London and New York: Macmillan and Co. 1890.

The first appearance of this part of Roscoe and Schorlemmer's great work was in 1884. Since that time the knowledge respecting this department of Organic Chemistry has largely increased. In the present volume will be found, for example, the researches of Curtius on the fatty diazo-compounds; of Kiliani on the constitution of the members of the sugar group; of Emil Fischer on the artificial production of certain members of this group; of Brown, Heron, and Morris on the starches and dextrins; and further, of W. H. Perkin, junr., on the Polymethylene compounds, all this being fresh matter since 1884. These enlargements and additions have made it necessary to defer the consideration of the derivatives of Furfuran, Pyrrol, and Thiophen for another volume, together with the Pyridine and Quinoline derivatives. The price of the volume is 21s. (See this Journal, 1884, 331.)

DIE VERUNREINIGUNG DER GEWÄSSER, EINE DENKSCHRIFT IM AUFTRAGE DER FLUSS-COMMISSION DES VEREINS ZUR WAHRUNG DER INTERESSEN DER CHEMISCHEN INDUSTRIE DEUTSCHLANDS. Bearbeitet von DR. KONRAD WILHELM JURISCH. Privat-Dozent an der königlichen Technischen Hochschule, Berlin. 1890. Berlin: R. Gaertner's Verlagsbuchhandlung. Hermann Heyfelder, S.W. Schönebergerstrasse 26. London: H. Grevel and Co., 33, King Street, Covent Garden.

A QUARTO volume, containing 110 pages of Subject-Matter, Preface, Table of Contents, and Alphabetical Index. The price is 10s. The work is stated to furnish a complete and clear treatise on all the details relating to the Removal of the Waste Waters and Liquors from Works. The following excerpt of the contents of the work will give the clearest idea both of its contents and the nature of the treatment of the subject:—

PART I.—Description of the Waste Liquors or Waters. (1.) Sewage and Waste Waters in Town and Country. (2.) Waste Matters and Waters of Domestic Trades and Mechanical Industries. (3.) Waste Waters of Mining and Metallurgical Industries. (4.) Waste Waters of the Agricultural and Chemical Industries.

PART II.—On the Damage done by Waste Waters and Liquors as they go forth from the Works and Factories. (1.) Waste Liquors containing Nitrogenous Organic Impurities. (2.) Waste Liquors with Solids in suspension. (3.) Waste Liquors with predominating Mineral Matters. Conclusions.



PART III.—The Purification of Waste Waters. (1.) Sewage or Waste Waters containing Nitrogenous Organic Impurities. (2.) Waste Waters containing Solids in suspension. (3.) Waste Waters with predominating Mineral Matters.

PART IV.—The German Rivers.

PART V.—Contaminated River Water.

PART VI.—The Fishery Interests. (a.) The Effects of Sewage and Waste Liquor Contamination on the Fisheries. (b.) An Exposition of other Injurious Influences on the Fisheries.

PART VII.—Importance of the Fishery Question.

PART VIII.—Judgment on the Sewage and Waste Water Question.

PART IX.—Dealing with the Sewage and Waste Water Question.

PART X.—Influence of the Waste Liquors of Works on the Health of the People in the adjacent regions.

PART XI.—Conclusion.

A TECHNOLOGICAL DICTIONARY OF INSURANCE CHEMISTRY. By WILLIAM A. HARRIS, F.R.S.S.A., &c. &c., 1890: Published by the Author, Phoenix Fire Office, Phoenix Chambers, Exchange, Liverpool.

OCTAVO volume, bound in red cloth, and containing, besides preface and introduction, 407 pages of subject matter. Price 21s. The work is described by the author as a "Compendium of the latest information on subjects of vital importance to insurance managers, surveyors, underwriters, government officials in all countries, merchants, captains, shippers, harbour-masters, &c., and home, colonial, and foreign insurance agents; explaining the various risks to be apprehended from spontaneous combustion, oxidation, chemical affinity, fermentation, friction, expansion of gases, inflammability of vapours, dust explosions, steam-heating and drying, oils, fibres, coal, cotton, and mixed cargoes, &c., &c. With actual cases showing where losses have occurred, &c."

The work may be defined as a technological chemical and physical dictionary with special reference to the various causes of superheating, fires, and explosions.

THE PATENTEE'S MANUAL. A Treatise on the Law and Practice of Patents for Inventions, with an Appendix of Statutes, Rules, and Foreign and Colonial Patent Laws, International Convention and Protocol. By JAMES JOHNSON, of the Middle Temple, Barrister-at-Law, and J. HENRY JOHNSON, Solicitor, Assoc. Inst. C.E., &c. Sixth Edition, revised and enlarged. 1890. London: Longmans, Green, and Co., Paternoster Row; Stevens and Sons, Lim., 119 and 120, Chancery Lane.

The fifth edition of this work (see New Books, this Journal 1884, 654) contained 489 pages. The present edition contains 534 pages, and its price is 10s. 6d. Amongst the improvements introduced are the following:—"The provisions of three supplementary Acts of Parliament and of four new sets of Official Rules, now consolidated with the previous Rules, have been noticed, whilst numerous decisions of the Courts during the last six years, many of which have had an important bearing on the Law of Patents, have been cited." "Great additions to and alterations in the Patent Laws of Foreign States and British Colonies have been made of recent years. The Appendix contains summaries of the whole of those laws to date," duly revised in those States or Colonies.

It is stated at the close of the Preface, that "the number of applications for patents increases at the rate of about a thousand a year."

TREATISE ON THE EVAPORATION OF SACCHARINE, CHEMICAL, AND OTHER LIQUIDS BY THE MULTIPLE SYSTEM IN VACUUM AND OPEN AIR, ALSO THE CONSTRUCTION AND WORKING OF THE DIFFERENT SYSTEMS. By JAMES FOSTER, M.I.M.E. Sunderland: Thomas Reed and Co. London: Simpkin, Marshall, and Co.; Hamilton, Adams, and Co. London and New York: E. and F. N. Spou. Glasgow: George Routledge and Sons; W. and R. Holmes. Amsterdam: J. H. de Bussy.

This is an octavo volume, bound in red cloth, containing 600 pages of Subject-matter and 123 pages of Matter consisting of the text of 12 patents. The illustrations or diagrams number 195, and there are 49 large plates. With the text are interspersed 56 tables relating to the matter thereon. The price is 21s. The work is a systematic complement of all of importance that has been done in the direction indicated in modern times, and in his preface the author says its purpose is, "to supply a long felt want for the Industries where Evaporation is required, in which both makers and users are interested." At the end of the treatise is to be found an account of the sugar machinery used in the West Indies up to the year 1848, which the author has reproduced from Leonard Wray's work, entitled "The Practical Sugar Planter," of that date, to show the strides made in the evaporation of sugar juices during the last 40 years.

## Trade Report.

### TARIFF CHANGES AND CUSTOMS REGULATIONS.

(From the Board of Trade Journal.)

#### RUSSIA.

##### Classification of Articles in Customs Tariff.

Mr. J. Michell, Her Majesty's Consul at St. Petersburg, in a report to the Foreign Office, dated the 26th May, encloses decisions of the Russian Department of Customs, to the effect that the under-mentioned articles are classified as follows under the Russian tariff:—

Coal, slag, produce of blast furnaces, reduced to powder, under section 18 of the tariff, as a manuring substance. Duty free.

Phosphorite dust, similarly as bone dust, to be exported from Russia, under section 1 of tariff of exportable goods. Duty free.

Limestone to be cleared similarly as chalk in pieces not purified and not calcined, under section 7, point 1. Duty free.

#### SWITZERLAND.

##### Classification of Articles in Customs Tariff.

Note.—Quintal = 220·4 lb. avoirdupois. Franc = 9<sup>6</sup>/<sub>10</sub>d.

The following decisions affecting the classification of articles in the Swiss Customs tariff have recently been given by the Swiss Customs authorities:—

Sulfocyanure of aluminium.—Category 17. Duty, 1 franc per quintal.

Sulphurous acid compressed, which was formerly in Category 9a, is now classified in Category 18, and pays a duty of 2 francs per quintal.

Grape-sugar (glucose) in the form of syrup.—Category 243. Duty, 7 francs per quintal.

Grape-sugar (glucose) in a solid state.—Category 244. Duty, 7 francs 50 cents. per quintal.

## ITALY.

*The Treatment of Saccharine and its Products.*

The Marquis of Dufferin and Ava, Her Majesty's Ambassador at Rome, in a despatch to the Foreign Office, encloses translations from the *Italian Gazette* of decrees of the King of Italy on the treatment of saccharine and saccharine products.

Decree No. I. provides that the Royal decree of 26th July 1888, by which various changes were introduced into the general Customs tariff, respecting the duty to be paid on the importation of acetic acid, saccharine, and other chemical products not specified, is converted into law.

By decree No. 11., the Royal decree of 29th September 1889, which forbids the introduction or production within the kingdom of saccharine and saccharine products is converted into law.

## UNITED STATES.

*Recent Customs Decisions.*

Certain terra-cotta paste and powder, not a dye-wood extract, but a composed colour, one portion being derived from a vegetable source, and the other from alizarine, either of vegetable extraction or of coal-tar origin, is dutiable at the rate of 25 per cent. *ad valorem*.

Certain so-called "Eikonogen" not an auiline salt, nor an acid, nor a preparation of coal-tar, but a chemical compound, possibly derived from a preparation of coal-tar, is dutiable at the rate of 25 per cent. *ad valorem*.

Certain chlorophyll found on chemical analysis to be a vegetable colour which is principally used in colouring soap, is dutiable at the rate of 25 per cent. *ad valorem*.

Thio-chromogen, a coal-tar colour, is dutiable at the rate of 35 per cent. *ad valorem*.

Certain so-called "Cherry Laurel" is dutiable as medicinal waters, non-alcoholic, at the rate of 25 per cent. *ad valorem*.

## VICTORIA.

*New Customs Tariff.*

The following is a statement of the rates of Customs duty now levied on the importation of articles into the colony of Victoria. The modifications which form the basis of this new tariff were published at pp. 268—75 of the March issue of the *Board of Trade Journal*:—

Classification of Articles.	Rates of Duty now levied.
	£ s. d.
Acids, viz.:	
Acetic, containing not more than 30 per cent. acidity.....	Pt. or lb. 0 0 3
Acetic, for every extra 10 per cent. or part of 10 per cent. above 30 per cent. ....	" 0 0 1
Muriatic, nitric, sulphuric .....	Cwt. 0 5 0
Butterine and oleomargarine .....	Lb. 0 1 0
Candle nuts .....	Free.
Candles.....	Lb. 0 0 2
Charcoal and coal (ground).....	20 % <i>ad val.</i>
Charcoal, animal (ground) .....	Free.
Drugs, viz.:	
Ammonia, carbonate of .....	Pt. or lb. 0 0 2
" liquid.....	" 0 0 2
Chlorodyne .....	25 % <i>ad val.</i>
Cocculus indicus.....	Lb. 0 1 0
Glycerin, pure.....	" 0 0 3
" crude.....	" 0 0 1
Morphia.....	Oz. 0 1 6

Classification of Articles.	Rates of Duty now levied.
	£ s. d.
Drugs— <i>cont.</i>	
Nitrate of silver .....	Oz. 0 0 6
Nux vomica .....	Lb. 0 0 2
Strychnine .....	Oz. 0 1 0
Explosives (except fine meal powder, not sporting, in bulk and in packages of not less than 25 lb. each), viz.:	
Powder, sporting.....	Lb. 0 0 3
" blasting .....	" 0 0 1
Gelatin and gelatin dynamite .....	" 0 0 1
Other explosives.....	" 0 0 4
Gelatin .....	" 0 0 6
Leather (except crust or rough tanned, viz. :— Calf, goat, hogskin, sheep, or kangaroo, when not exceeding seven pounds each skin; and English bend, sometimes called butt).....	" 0 0 6
Do., viz. :—Kid, calf kid, mock kid, and patent calf.....	Free.
Do., being furniture and bookbinding Morocco, roan, and pasto grain skiver.....	Lb. 0 1 0
Medicines, consisting of two or more ingredients mixed ready for use, not being in chemical combination, drugs and chemicals, packed ready for retail sale or for consumption, including medical compounds containing spirits not exceeding the strength of proof by Sykes' hydrometer; and all preparations recommended as beneficial for any portion of the human or animal body, or the cure or the treatment of any disease or affection whatever, and medicine chests or cases, with or without fittings.....	25 % <i>ad val.</i>
Oils, in bulk (except coconut, fish of all sorts, mineral, refined, of which the point of ignition is below 80° F., kerosene, palm, lubricating, of which the chief component part is mineral; and resin).....	Gall. 0 0 6
Do., viz., resin.....	Free.
Do., kerosene .....	Gall. 0 0 6
Do., packed in bottles, jars, or other vessels not exceeding one gallon in size, as under:	
Quarts and over a pint .....	Doz. 0 4 0
Pints and over half a pint .....	" 0 2 0
Half pints and smaller sizes.....	" 0 1 0
Over a quart and not exceeding a gallon..	" 0 12 0
Opium, including all goods, wares, and merchandise mixed or saturated with opium or with any preparation or solution thereof, or steeped therein respectively .....	Lb. 1 0 0
Paints and colours (except artists' colours): Ground in oil, including patent dryers and putty .....	Ton 2 0 0
Mixed, ready for use, from or of any substance .....	" 4 0 0
Perfumery.....	20 % <i>ad val.</i>
Powders, baking, scidnitz, washing .....	20 % <i>ad val.</i>
Salt (except rock salt) .....	Ton 1 0 0
Soap, perfumed, and toilet .....	Lb. 0 0 4
Do., other.....	" 0 0 2
Soda crystals .....	Ton 2 0 0
Spirits, cordials, liqueurs, or strong waters, sweetened or mixed with any article so that the degree of strength cannot be ascertained by Sykes' hydrometer (including all alcohol diluted or undiluted with water or other menstruum and containing in solution any essence, essential oil, ether, or other flavouring or other substance whether of natural or artificial origin).....	" 0 12 0

Classification of Articles.	Rates of Duty now levied.
Spirits, methylated .....	Liq. gall. $\begin{smallmatrix} £ & s. & d. \\ 0 & 1 & 0 \end{smallmatrix}$
Do., perfumed .....	Gall. 1 4 0
Spirits mixed with essential oils, so as to be unfit for human consumption, to be used in the manufacture of soap, under Commissioner's permit, provided the mixing is performed in the presence of an officer of Customs.....	Free.
Starch.....	Lb. 0 0 2
Stearin .....	" 0 0 2
Sugar, the produce of sugar cane.....	Cwt. 0 3 0
Sugar, the produce of sugar cane, bonded on and after 27th day of July 1887, and refined in Victoria in a bonded warehouse, under regulations to be framed by the Governor in Council.....	" 0 2 0
Sugar, the produce of beetroot and all other sugar.....	" 0 6 0
Sugar candy.....	Pt. or lb. 0 0 2
Sugar molasses (except unrefined) .....	Cwt. 0 3 0
Varnish (including lithographic) .....	Gall. 0 2 0
Vinegar, not being acetic acid or crude vinegar, aromatic, or raspberry .....	" 0 0 6

## EXTRACTS FROM DIPLOMATIC AND CONSULAR REPORTS.

### NITRATE TRADE OF CHILI.

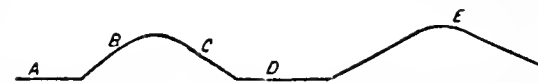
Report by Consul-General Walker, of Bogota.

#### Geographical Position of the Nitrate Deposits.

The chief deposits of the crude nitrate of soda are found in the province of Tarapacá, formerly a part of Peru, but ceded to Chili in 1881 as a war indemnity on condition that after 10 years it may be restored to Peru, provided a majority of the voting population of the province shall so elect, and upon the payment to Chili of 10,000,000 dols. Upon such conditions it is not probable that this territory will ever revert to its former owner; for, in addition to the money difficulty, a serious one in the present disorganised condition of Peruvian finances, the large accessions of population to Tarapacá since its annexation to Chili have been mostly from that Republic, and these new-comers will naturally be averse to restoration.

The province of Antofagasta, acquired by Chili from Bolivia, and joining Tarapacá on the south, and having similar geological and climatic features, is also rich in nitrate deposits. A point a few miles south of Taltal, in this province, in latitude 25° 45' S., may be considered the southern limit of the nitrate belt, its northern being in latitude 19° 12' S.; its extreme north and south length being thus shown to be 260 geographical miles. Its average width is not more than 2½ miles. This narrow strip of nitrate lands stretches along the eastern slope of the coast range of barren, verdureless mountains which wall in the Pacific Ocean, from the northern limit of Peru, to the Straits of Magellan, upon which, for more than 2,000 miles, not a drop of rain ever falls, and upon which there is not a tree, shrub, or any living vegetation. Some of its peaks reach an altitude of 4,000 or 5,000 feet above the sea level, but the usual height of the range is about 2,000. The average distance from the coast to the nitrate beds is about 14 miles, but many of them are not more than 10.

The following profile will give a better understanding of the situation of these deposits:—



A, Pacific Ocean; B, coast range; C, location of nitrate deposits; D, valley of Tamagruel; E, beginning of the main cordillera of the Andes.

#### Nitrate Development.

As early as 1813 some effort had been made to develop these nitrate deposits. During that year the Spaniards exported 22,732 quintals; but, the war of independence breaking out about that time, little was done to develop these mines of wealth until 1852. During that year an Englishman, George Smith, and José Sandes, a Spaniard, established small refining works at Iquique, under the name of the Tarapacá Nitrate Company. Encouraged by their success, numerous other similar establishments sprung up at different points along the coast, and by the year 1875 the nitrate trade had attained so important a development as to attract the attention of the Peruvian Government to it as a source of national revenue. In that year a law was passed providing for the acquisition of the nitrate deposits and refining establishments by the favourite method of "expropriation," and more than 60 different properties, belonging to companies and individuals, were taken possession of by the Peruvian authorities. Certificates were given in payment, to be redeemed through bills of exchange on London, drawn against shipments of nitrate.

The war with Chili broke out before any material portion of this indemnity was paid; but, as the owners of the expropriated property had been fully dispossessed, Chili, during the progress of the war, took possession of it as national Peruvian property. At the end of the war the outstanding certificates issued by Peru, principal and interest, were found to amount to 5,400,000l. (26,276,400 dols.) Chili, having fallen heir to the property, was morally bound to satisfy all mortgages and liens upon it, or to restore it to its original owners. After a few years of unsatisfactory experience in carrying on nitrate production for Government account, she wisely determined to adopt the latter alternative, and on 11th June 1881, the President of the Republic issued a decree ordering the restoration of the expropriated nitrate properties to their original owners. The conditions of this restoration were the delivery to the Chilean Government of a minimum of three-fourths of the certificates in amount given by Peru to the dispossessed owners, and a deposit of money, equal in amount to that of the deficient certificates, to be returned upon the delivery of the latter.

This wise and just measure gave an immediate and powerful impulse to nitrate production, and has proved a very advantageous arrangement for the Chilean treasury. It has not, however, been unattended with some disadvantages and dangers to the commercial prosperity of the Republic. The English companies, organised under the policy of restoration, are making strenuous efforts to secure a monopoly of the nitrate trade. Success in these efforts would signify diminished production to secure high prices, a large falling off of Government revenue from exports, and the ruin of small operators, who now contribute so largely to the aggregate of nitrate production and to the prosperity of the Republic. This is no idle fear, and doubtless the Chilean Government is alive to the danger.

The London nitrate companies, and the fabulous fortunes so suddenly acquired by their skilful manipulations, had their origin in this policy of restoration. Their history is not without interest.

During the short and unsatisfactory experiment made by Chili in carrying on nitrate production for Government account, an Englishman was employed as nitrate inspector, who, by means of his official connexion with the Government, ascertained, it is said, its intention in favour of the ex-owners of nitrate properties long before the public had any such knowledge. Forming a combination with another Englishman, whose name has since become widely known in connexion with the nitrate trade, and with still another

Englishman, the manager of a Valparaíso bank, the combination hastened to Lima, where the Peruvian certificates were mostly held, and succeeded in purchasing, it is said, for less than half their face value, certificates which carried with them the right of redemption of eight or ten of the most valuable of the nitrate properties in the whole province of Tarapacá. For instance, they acquired the property of the Ramírez Company, it is said, for 5,000*l.*, and afterwards sold it in London for 50,000*l.* In this way, and at corresponding prices, they secured the "Peruana," the "Bien Retiro," the "Jaspampa," the "Virginia," and others—the very cream of the Iquique and Pisagua properties. Upon the success of these purchases the combination transferred their operations to London, and within the last six years have floated eight stock companies (limited), with an aggregate capital of 5,875,000*l.* (28,587,750 *dols.*), the shares of which, it is believed, are still largely held by themselves. The splendid showing of profits which they made enabled them to put the stock of these companies on the market at a high rate, notwithstanding the enormous figures at which they were capitalised; and these prices have been well maintained. I have no recent quotations at hand, but I am informed that in most, if not all, of these companies their shares still command a high premium.

In addition to the English companies above alluded to which have a substantial basis in valuable nitrate properties, several self-named nitrate companies have been organised in London, with enormous nominal capital, which are purely speculative, being based on neither Government concessions nor the ownership of nitrate or other property in Chili. To what extent their promoters have succeeded in abstracting sovereigns from the pockets of credulous investors I cannot say.

The wise and liberal policy of the Chilean Government in encouraging small and independent establishments is an obstacle to the formation of a monopolistic "trust" or combination of the English companies, for the control of the nitrate trade.

At the time of my recent visit to Chili as special commissioner a large and imposing representation of these companies arrived at Santiago for the purpose, it was said, of obtaining such concessions and changes in nitrate regulations as would render them masters of the situation and give them a virtual monopoly of the nitrate trade. How far they succeeded in their designs I have no positive information, but I am convinced that the policy of strengthening the hands of the would-be monopolists was fully appreciated at Santiago. On the other hand, I am convinced that the investment of American capital in the nitrate business of Chili would be favourably regarded by both Government and people as tending to impose a wholesome obstacle and check to the monopolistic tendencies of the English companies, which may be considered *pro tanto* under one management.

#### *The Nitrate Deposits.*

The possibility of so strange a fact as the existence in open air of enormous deposits of a salt so deliquescent and easily destroyed by moisture as the nitrate of soda, is partially accounted for by the strange meteorological fact that where they are found it never rains. This rainless region extends from the northern part of Peru, in about south latitude 4°, to about latitude 30° S, and inland from the shores of the Pacific to the main cordillera of the Andes, an average distance of about 90 miles. The country within these limits is a perfect desert, except along the borders of the few streams which, rising in the snow mountains, force their way into and across the desert to the Pacific, furnishing along their course water for irrigation, such as the river Rimac, which runs through the centre of the city of Lima, and irrigates the broad fruitful valley lying between that city and the sea coast.

I have already explained that the nitrate beds are found on the eastern slope of the coast range, which, often precipitous on its western face, on its eastern slopes gently down to the valley of Tamagruel, which separates the coast range from the main cordillera of the Andes. This valley, with an average width of 10 miles, stretches from the hills of Caricoles, which connect the two Andean ranges, in

latitude 22° 40' S., to Aguas Blancas, in latitude 25° 45', south of which point the country becomes very broken, and the nitrate deposits disappear.

This valley is wholly devoid of all the characteristics which we usually associate with the word, such as groves of green trees, verdant pastures, running streams, &c. Instead, the eye wanders over a scene of treeless, verdureless, waterless desolation. It is true, on the eastern side of the valley there is found an occasional weak spring, where feeble attempts at cultivation have been made, but with indifferent success, as the soil is so thoroughly impregnated with salts of various kinds as to render its profitable cultivation impossible, even with a sufficiency of water for irrigation.

The point on the slope of the mountain where the deposits of caliche are found is some 500 or 600 feet higher than the valley, but it diminishes in quantity and richness as the valley is approached, and disappears entirely at the bottom.

An examination of the workings of these beds discloses the following conditions:—

(1.) That the surface, to the depth of 8 or 10 inches, is covered with a layer of fine loose sand.

(2.) That underneath the sand is found a conglomerate of amorphous porphyry, feldspar, chloride of sodium, magnesia, gypsum, &c., cemented by the sulphate of lime into a hard, compact mass to a depth of 6 to 10 feet, called the "costra," or crust.

(3.) That below this crust the caliche, or impure nitrate, is found, presenting to the view a variety of colours—yellowish-white, orange, bluish-grey, &c.

The nitrate deposit being reached, which, like the crust, is found cemented into a rock-like mass of from 4 to 6 feet deep, is quarried by blasting with a coarse-grained powder, of which as much as 150 lb. are sometimes used at a single blast. Neither dynamite nor nitroglycerine are used, as it would shatter and pulverise the caliche so as to occasion a serious loss.

After being brought to the surface, the caliche is carefully assorted by experts, called "particulares," broken into pieces double the size of an orange, and carted to the refinery establishment, situated on the Pampas or on the sea-coast, or carried to Iquique, Pisagua, Patillos, and Antofagasta, by rail, each of these places having connexion, by narrow-gauge railways, with the nitrate deposits, and which, consequently, are rapidly becoming the chief centres of nitrate production and export. Of course, if water and fuel were obtainable near the deposits, it would be more economical to carry on the process of elaboration there; but as these essentials can be had more conveniently at the sea-coast, it would seem to be cheaper to bring the caliche to the fuel and water. The fuel used is bituminous coal, brought by sea, mostly from the coal-beds of southern Chili; while water is obtained chiefly by condensation, and, for domestic purposes, is brought in boats from Arica and other points on the northern sea-coast. Some attempts have been made to obtain water by sinking surface wells, but, as it never rains in these regions, success, of course, was impossible. No efforts, as far as I know, have been made to obtain artesian water, but the fact that the snow mountains are comparatively near seems to indicate that an abundant supply might be obtained at no great depth.

#### *Capacity of the Deposits.*

Mr. George Smith, who did so much for nitrate development in Tarapacá, estimates that there is a sufficiency of crude nitrate in that province alone to yield 63,000,000 tons of the commercial article; while Mr. Billingham, in his interesting pamphlet entitled "Estudio sobre la geografía de Tarapacá," estimates the total capacity of the nitrate beds at 178,011,104 English tons. The mean of these two widely differing estimates would be 120,505,552 tons, an amount sufficient to supply the world's uses, at the present rate of consumption, for many centuries.

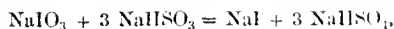
#### *Method of Purification.*

The conversion of impure caliche into commercial nitrate of soda is effected by means of lixiviation, or leaching. For that purpose elaborate and expensive machinery is employed,

a detailed description of which would be out of place in this report. It will suffice to say that the caliche is dissolved in water at a high temperature, in long tanks, from which the solution is carried in pipes to enormous shallow pans, and the water evaporated by artificial heat, the vapour being conveyed to a condenser, and the water thus distilled saved for further use.

#### *Preparation of Iodine.*

It is found that in every 100 kilos. of impure nitrate there are 50 grms. of iodine, and that in the crystallisation of the nitrate the mother-liquor, called, technically, *aqua vieja*, holds it in solution. In order to extract it this mother-liquor is drawn off into a separate tank and charged with sulphite of soda, with (according to Malloché) the following chemical reaction:—



forming the iodide of sodium; and by the subsequent addition of a further quantity of sulphite, precipitating the iodine, which, containing more or less impurities, is refined by sublimation and condensation.

#### *Export of Nitrate and Iodine.*

The importance of the nitrate trade will be seen from the following figures: The export of nitrate of soda from the ports of Taltal, Antofagasta, Iquique, Pisagua, and Topilka, (Iquique furnishing two-thirds of the whole) for the 10 years ending the 31st of December, 1888, was 4,574,440 English tons, valued at 231,411,182 dols., upon which export duties were collected at the rate of 1 dol. per 100 kilos., to the amount of 87,470,622 dols. During the same period the exports of iodine amounted to 1,588,074 kilos., with a total value of 19,333,757 dols., upon which the Government collected in export duties 1,172,576 dols.

The export duties on nitrate for the year 1888, amounted to 17,447,882.12 dols., and it is estimated by the Minister of the Treasury that for the current year (1889) the amount will exceed 19,000,000 dols.

#### *Destination and Uses.*

Of the large export of Chilean nitrate during the year 1888 Germany took 284,000, Holland 33,400, Belgium 84,800, France 156,500, and Great Britain 104,800 tons. The price in the English market varies from 9*l.* 10*s.* to 10*l.* per ton.

The chief use to which it is applied in European countries is as a fertiliser; in Germany and France largely in the cultivation of the sugar-beet. Very considerable quantities, however—as much as 30 per cent. of the whole, perhaps—are consumed in the manufacture of nitric acid, nitro-glycerin, dynamite, gunpowder, &c. In the preparation of the last-named article the soda nitrate is first converted into the nitrate of potash, on account of the latter being less absorbent of atmospheric moisture.

It will be observed that the United States does not figure as an importer of Chilean nitrate, but it might be to the advantage of our agriculturists and manufacturers of explosives to turn their attention to its use.

#### RESOURCES OF PERU.

##### Report by Minister Hicks, of Lima.

#### *Salt Deposits.*

Closely allied to agriculture is the production of salt, and in this industry the natural advantages of Peru are, perhaps, unrivalled in the whole world. On the Pacific coast, 40 or 50 miles north of Callao, is the port of Huacho, where the salt deposits are sufficient to supply half the world. The Government last year sold the exclusive privilege of manufacturing salt at this point for 27,000 dols. Owing to a peculiar formation of the soil, the sea-water on the shore percolates into the earth and is evaporated by the heat of the sun. Salt is thus formed in masses, almost entirely pure, and is cut in the form of blocks resembling ice. This

natural and automatic manufacturing is constantly going on, and the supply is practically unlimited. This crude salt is generally used by the natives, and is shipped in immense quantities to Chili, where it sells for 1.60 dols. per hundred. The table salt, sold in the groceries of Lima, however, is of English manufacture, as there is no refinery for the article in this country.

A similar deposit of salt is found at Sechura, in the north of Peru, where the privilege for manufacturing was sold last year for 30,000 dols. Sechura supplies Ecuador, Colombia, and the northern part of South America.

#### *Sulphur Deposits.*

Immense deposits of sulphur are found at Tumbes in the north and Arequipa in the south. Sulphur is found so nearly pure that samples have analysed at 99.8 per cent. of pure sulphur.

#### *Gypsum Deposits.*

Fields of gypsum exist in the vicinity of Sechura, but its development has not been attempted on a large scale.

#### *Petroleum Deposits.*

Petroleum is found in the north, near Payta. Prescott speaks of the deposits of asphalt, and remains of ancient oil-wells and retorts have been found in that vicinity. An American gentleman has recently purchased a vast tract of land, 12 miles wide and 20 miles long, at Talara, near Payta, covering the site of these ancient oil works and vast deposits of asphalt, which are called by the natives "pitch mines." This gentleman was one of the original operators in the oil regions of Pennsylvania, and he knows well the value of his purchase. He set to work sinking wells, and now has more than half a dozen spouting oil exactly like the famous wells in Pennsylvania. He is erecting a gigantic system of refineries, and in a few years he will have in operation the largest oil-field in the world outside of Pennsylvania.\*

#### *Coal Deposits.*

In the neighbourhood of Chimbote the coal deposits are said to be very large. Both anthracite and bituminous coal exist on the surface and in ridges above the surface, so that it can be mined with but little expense.

#### *Miscellaneous Minerals.*

The mineral wealth of this country is almost fabulous in magnitude, and includes, besides gold, silver, and coal, immense deposits of copper, iron, nickel, platinum, mercury, and many others.

Lack of capital and skilled labour has prevented the development of its agriculture, and a dependence upon foreign importations to the neglect of its own resources has caused several vital industries to remain undeveloped. Its immen-

\* In a subsequent report to the Department (February 11, 1890), Minister Hicks says: "Referring to my No. 76 of January 21, 1890, in regard to the production of petroleum at Talara, near Payta, in northern Peru, I have to say that the refined kerosene manufactured at that point has appeared in large quantities in this city and Callao, and from its cheapness and excellence it seems likely to drive the American article entirely from these markets. A few days since one shipment of 20,000 boxes was reported as arrived at Callao, and the native article is now on sale at many places in that city and Lima. The experiment of burning crude petroleum in place of coal is being tried on the locomotives of the Oroya railroad, and, if it proves successful, it will open a considerable market for the article among the railroads. They now use English coal, and by thus putting into use an article of home production it will give a stimulus to the manufacture as well as curtail the importation of an English staple."

Under date of March 12, Minister Hicks, of Lima, reports further that Mr. Sutherland Hutton, vice-president of the Mining and Supply Company, of Los Angeles, Cal., has been in Peru, and has just completed a contract with Mr. Herbert W. C. Tweddle, owner of the oil-works at Talara, in northern Peru, by which Mr. Tweddle is to ship 1,500,000 gallons of crude oil every month during the life of the contract, which is to continue five years. This is probably but the beginning of this business, and it would indicate a condition of affairs of considerable interest to the oil-producing industry in the United States.

mineral deposits have not been properly appreciated. The attention of its people is now being called to these points, and a noticeable impetus is being given to its various industries.

#### CONDITION OF THE SISAL INDUSTRY IN THE BAHAMAS.

Report by Consul McLain, of Nassau.

One year ago I made a report to the Department upon the culture of sisal hemp in this colony, calling attention to it as a new industry just being introduced, and which promised to bring substantial prosperity to these islands in the near future.

During the year, and especially within the last few months, so many letters have been received at this consulate from various parts of the United States, making inquiries upon the subject, that I am satisfied a statement touching the present condition of the industry would interest many of our people, and I therefore submit the following:

The progress made in the development of sisal culture in the Bahamas during the past 12 months is marvellous. One year ago there was scarcely a dollar of foreign capital, and very little local, invested in this business in the colony, while to-day parties from Great Britain, Canada, and Newfoundland, representing large resources, are interested in sisal, have bought tens of thousands of acres of Government land, and are industriously engaged in clearing and planting the same to the full measure of their ability to procure the material. A local stock company, styled the Bahama Hemp Company, organised and managed by Nassau capitalists exclusively, has also purchased a large tract of land and is developing the same, whilst thousands of acres are being planted in every direction by individual owners of smaller pieces. American capital up to this date, I regret to say, for it is to its own disadvantage, has been conspicuous by its absence. One company, however, styled the Inagua Hemp Company, organised under the laws of the State of New Jersey, with D. D. Sargent, United States consular agent at Inagua, as manager, has lately procured about 1,200 acres at Inagua and has begun operations.

Messrs. Munro & Co., of St. Johns, Newfoundland, have obtained a grant of 18,000 acres of Crown land at Abaco, and are planting the same. Another tract of 20,000 acres has been allotted to a London company on the same island. Mr. Alex. Keith, of Edinburgh, Scotland, has taken 2,000 acres on Andros Island, and is working upon it. But the largest demand has been made lately by two London companies, who are said to be applying for not less than 200,000 acres between them.

Many applications for land have not been reached at all as yet on the files, the Surveyor-General's Department being hard pushed in the matter of surveys and locations, whilst new applications are being constantly received, and have to await their turn for consideration. So much land has been taken up that the Governor, a short time ago, advanced the price of Crown land from 1.25 dols. per acre, the ordinary price, to 4 dols. per acre, withholding also the benefit of the bounty. And lately it has been decided to sell no more large allotments of Crown land at present, the quantity already allotted with a view to cultivation being as great as the condition of labour in the colony will justify. The number of acres of Crown land already disposed of is about 120,000 acres, whilst pending applications on file and not yet reached will amount to at least 200,000 more.

This substantial withdrawal of Crown lands is creating some movement in real estate—as is natural under the circumstances—between private parties, some old properties changing hands at prices double and treble their supposed values two years ago. Persons buying private lands and cultivating them will share in the bounty of 1 per cent. per pound provided by law on all fibre raised and exported. Private lands in New Providence can be bought, unimproved, for from 8 dols. to 12 dols. per acre, and for less on the out-islands.

The employment given to labourers in clearing land and in setting out plants has already put considerable money into circulation, the beneficial effects of which are being felt in various quarters. There has been no special advance in the price of labour, field hands commanding from 40 to

60 cents per day, and finding themselves. Each month, however, witnesses a large increase in the number of those who find remunerative employment, and pleasant relations obtain between employers and employed.

The labour question has been and is one that here, as elsewhere, requires delicate treatment; but it has been skilfully met by Sir Ambrose Shea, the Governor, who long ago perceiving that to permit investors to locate upon adjoining lands would induce sharp competition in wages in thinly settled districts, adopted the plan of scattering the allotments about the different islands, or in localities remote from each other on the same island, so that each settlement should have its share of the benefits of the new industry by obtaining, at fair wages, employment for its local labour. In this way, also, a surplus of labour at one point and a scarcity at some other has been avoided. When the entire labouring population becomes employed, as will happen before long at the present rate of development, a new phase of the labour question will arise; but that time is yet in the future, and the remedy can be applied when the situation demands it.

Small shipments of fibre continue to be made by nearly every steamer, a few old plantings furnishing the material. It is not likely that shipments in any quantity will be possible under two years, but after that time an enormous increase may begin to be looked for, increasing steadily as new fields come into bearing, until the annual exports of the colony, which now average about 600,000 dols., will leap well up into the millions, as a moment's reflection will show. It is a very low estimate to expect half a ton of fibre per acre, and a very low estimate to call it worth 100 dols. per ton, for it is worth over 200 dols. per ton in the world's markets to-day. When even the present quantity of land sold and applied for, to wit, 300,000 acres, is bearing, which ought to happen within five or six years, it will produce 150,000 tons a year, worth 15,000,000 dols.—an increase of prosperity that sounds more like a fairy tale than a strong probability deduced from reasonable figures. And yet 300,000 acres is but a small portion of the uncultivated lands within the limits of the Bahamas.

It is estimated that about 6,000 acres of land have already been planted in sisal (a plantation once started needs no replanting for many years), and that many additional ones have been cleared and made ready for the plants, the obtaining of which has been almost impossible, the industry being seriously retarded thereby. The prices paid for plants have risen from 6 cents per dozen to 36 cents, so great has been the demand; but the price will now decline rapidly, since the supply of plants is developing enormously, about 2,000,000 being now available for planting, and others coming on speedily. The pita plant is being found on all the islands growing wild, and the stock of old plants is very great. From the centre of the old plant rises a pole about 16 ft. in length, on the branches of which small plants grow, averaging a thousand to each pole, and from these poles a vast supply is coming into market, creating a profitable business; for what were two years ago only noxious weeds have all at once become worth 20 dols. apiece for pole plants alone. Quantities of old plants have lately been discovered growing on the cays along the Florida coast, and small schooners are already buying these up and bringing them here for sale. This fact suggests the question whether this new hemp industry, which is about to revolutionise the condition of the Bahamas, may not also be developed in the southern portion of Florida. The plants are found there growing wild just as they are in these islands, and they flourish best in dry sandy soils, fit for little else. I would earnestly call the attention of the Department of Agriculture to this matter, and suggest the propriety of looking into it, and of calling the notice of the people of Florida to this possible source of wealth and prosperity. The conditions of soil, climate, &c. which make its culture a success here, may not obtain there, but the simple fact that the plant is found growing wild in Florida, is of itself a consideration that should warrant an investigation at the hands of the Department.

The unexampled success of the sisal industry in so brief a period in this colony is entirely attributable to the business-like, systematic manner in which it has been



managed by the present Governor, Sir Ambrose Shea, who has all along taken a most earnest interest in the matter. He is a man of large experience in affairs, and has practical knowledge of the proper way to manage industrial enterprises. From the start he realised that this industry would be the salvation of the Bahamas, and, setting his heart upon it, he pushed it forward with great energy and prudence, overcoming numerous difficulties, surmounting obstacles, encouraging the faint-hearted, until now the people are touched with his own enthusiasm, and the industry is fairly afloat. He visited England, and by personal effort enlisted capitalists and procured large investments. To Sir Ambrose Shea the colonists owe a large debt of gratitude, and when the signal prosperity which is already hanging over the islands shall have been developed, to its full measure, they will more perfectly realise how, not only their individual interests, but those of outside investors, have been wisely and prudently promoted and guarded from the very inception of the industry by the practical, discreet, and conservative action of their Governor.

There can be no doubt or question as to the success of sisal culture in this colony. It has passed far beyond the experimental stage, and is giving daily evidence that it will become a source of wealth to all concerned. The combined conditions of soil and climate especially adapted to the growth of first class fibre give this colony a marked advantage over other West Indian islands, where the plant may grow luxuriantly enough, but will be found deficient in good, strong fibre. The poorer and more sterile the soil the better the result, and here the plant flourishes where ordinary vegetation seems almost impossible. It is a plant of unfailling growth; it will live without rain to moisten the soil; you can scarcely exterminate it if you try; it requires but little cultivation, and at an expense below that of almost any other agricultural product; and its value is substantial.

As two-thirds of the trade of the Bahamas is now with the United States; as their only steam communication with the outside world is by a subsidised line of American steamships running between Nassau and New York; as their increased wealth and prosperity means a larger and more profitable commercial intercourse with our own country, we should view this coming development of their material interests with pleasure, and with the warmest wishes for its complete success.

In conclusion, I would add that I have sent by this mail four samples of the Bahama fibre for the information and satisfaction of the State Department, believing that the same would be of sufficient interest to justify me in so doing. These specimens were not specially selected, but are only fair samples of the average fibre which is now being grown and shipped from the colony. Two of them have still attached a stub, or portion of the butt end of the leaf, which was purposely not passed through the machine, showing the character of the sisal plant whence extracted.

#### AMERICAN IMPORTS AT BARRANQUILLA.

Under date of January 7th, 1889, Vice-Consul Whelpley reports that the imports from the United States more than doubled during the year 1889, if landing certificates may be taken in evidence. Probably the most marked increase is on refined sugar, as an article for illustration. During the calendar year 1888 landing certificates were issued for 625 half-barrels refined sugar; the calendar year 1889, by record of landing certificates, shows 40 barrels and 2,824 half-barrels to have been landed; caustic soda, 109 drums, against 70 for the year previous, and other imports in like proportion. The new coffee crop, now in commencement of shipment, bids fair to excel past records.

#### A NEW PROCESS IN GLASS MANUFACTURE.

Report by Consul Monaghan, of Mannheim.

In the manufacture of glass a new and important improvement has been recently reported. It is believed that a complete revolution in the manufacturing methods must take place. Formerly, panes and plates of glass were produced by blowing large hollow cylinders, which were

afterwards cut and pressed. The blowing process, so tiresome and unhealthy to workmen, is to be entirely done away with. The glass manufacturer, Simon, has a new process, by which the tough glass may be run through rolls and made flat, smooth, and of any width and length. The plate-glass so produced is distinguished by greater homogeneity, firmness, toughness, and clearness; besides, the surfaces receive a brilliancy of finish little less beautiful than the finest cut glass. The essential part of the new system consists in the use of peculiarly formed waved hollow metal rolls, which are heated from the inside by means of steam or gas. These rolls take the tough liquid glass direct from the melting pot and run it out into long sheets like those of tin or sheet iron. To avoid sticking of the glass to the rolls the latter are covered with a thin coating of coal-dust, oil, and wax. When one considers the marvellous increase in the demand for sheet and plate glass, and that its former production was at the cost of much money and the health of employes, the present discovery must be regarded as of great importance. It is expected to reduce the cost of production, and consequently the selling price, very materially.

#### THE MANUFACTURE OF PERFUMES AT NICE.

The following is an extract from a report by Her Majesty's Consul at Nice upon the manufacture of perfumes in that district:—

"The art of producing scent is well understood in my district, but can only be acquired by long practice, by which alone the blending of the primary essences in proper proportions becomes thoroughly understood.

"The first process is the extraction of the essences by distillation. A large copper vessel or alembic is filled with water to about two-thirds of its capacity; the flowers to be treated are then introduced, and it is hermetically closed. It is then placed on a fire; usually, in our days, gas or steam is used for this purpose. Steam is generated by this means in the cylinder, and is carried by means of a pipe into a second cylinder, which is constantly kept replenished with cold water, and is furnished with an overflow cock. The pipe, in its passage through this second cylinder, assumes the form of a spiral coil. This coil ends in a cock at the bottom of the cylinder, from which the volatile essence exudes drop by drop as the steam becomes condensed in its passage through the spiral coil. In this manner the essence is collected in a small glass vessel, while at the same time the water containing a small portion of the scent, and which still remains in the alembic aforesaid, is itself perfumed, and becomes the rose water or orange flower water of commerce. All flowers are not susceptible of this treatment—some of them, such as jessamine, violet, cassia, tuberose, &c., containing no essence, have to be treated by a different process, which will be presently described. Of the flowers producing essences the orange flower produces but 1 grm. of essence for 1 kilo. of flowers, or but one-thousandth part. This essence is styled 'neroli,' and is the principal essence produced in the district between the Var and the Italian frontier.

"The following table will show the proportionate yield of the different flowers:—

Neroli .....	1,000 kilos. of flowers	1 kilo. of essence.
Rose .....	25,000 " "	1 " "
Geranium .....	1,000 " "	1 " "
Mint .....	1,000 " "	0.750 " "
Orange leaf (bitter) ..	1,000 " "	1 " "
Lavender .....	100 " "	0.500 " "
Eucalyptus .....	100 " "	0.500 " "

"The volatile essences thus obtained, combined and mixed together with a certain quantity of alcohol, are used in the preparation and as the basis of Eau de Cologne, toilet vinegar, lavender water, &c. There are two processes used for the purpose of extracting perfume from flowers which do not contain the volatile essence. The first may be described as the cold process, and the second as the hot process. The former is generally used for cassia (*Aracia Farneriana*), jessamine, jonquils, tuberoses, violets, and some other flowers.



"Freshly-gathered flowers are placed upon a layer of pure lard, a quarter of an inch in thickness, spread over a sheet of glass about two feet square, which is framed in wood and forms a kind of tray. These trays, sometimes some 40 or 50 together, are then piled upon one another; the flowers are changed every 12, 18, or 24 hours, according to circumstances, and the process is thus continued until the lard is sufficiently charged with perfume. Jessamine and tuberose are frequently changed as often as 50 times before the lard is considered to be sufficiently impregnated, cassie and violets from 30 to 40 times, and jonquils about 20 times only. The fat thus obtained can be packed in air-tight tins and conveyed anywhere.

"When the hot process is resorted to for the purpose of obtaining the impregnated fat, 20 kilos. of grease are placed in a copper vessel, together with some 5 kilos. of flowers; the vessel is then placed over a slow fire, and the contents are well stirred. After allowing the compound to boil for 10 minutes, the vessel is left to cool for some hours; an additional 5 kilos. of flowers are then added, and the process is repeated until the fat has absorbed the requisite amount of perfume; the hot liquid is then poured through a sieve, and the greasy flower-paste that remains is subjected to hydraulic pressure. It is in these two ways that the 'pommades' of trade are produced.

"From these 'pommades' perfumed and alcoholised liquids are extracted by means of grain spirit and also by spirits of wine; these are the 'extraits' of trade, and it is by the judicious blending of the different essences and concentrated perfumes, obtained by the processes above described, that the numerous scents are produced.

"The principal 'parfumeries' of my district are situated at Grasse, but the Bermonds and Lhermines of Nice are well-known houses; the latter of which appears to be at present the most important manufactory in Nice and its neighbourhood."—(No. 164, *Foreign Office Miscellaneous Series*.)

#### TRINIDAD ASPHALTUM.

Under date of November 10th, 1889, the United States Consul for Trinidad reports that the shipments of asphaltum from Trinidad—the greater part to the United States—from January 1st to June 30th, 1889, amounted to 32,460 tons, being a prodigious increase over any previous semi-annual shipments during the history of the colony.

### MISCELLANEOUS TRADE NOTICES.

#### FRENCH MINING INDUSTRY.

The *Economiste Français* for the 3rd May publishes the following information respecting the French mineral industry in 1889, which has been extracted from an official report addressed to the Minister of Public Works by M. Lorieux, *Inspecteur Général*, and M. Keller, *Ingénieur, des Mines* :—

The quantity of coal extracted from French mines in 1888 was not less than 22,603,000 tons, which represents an increase of 1,315,000 tons in the year, and the provisional figures show, for 1889, a further increase, still larger, which approaches two million tons.

In the metal industry the progress is less marked. The production of steel and iron is far from attaining the level reached in 1883, before the crisis. However, the revival in trade which manifested itself in 1887 continued in 1888, and increases have been notified in the manufacture of cast irons and steels in 1889. The forges, properly so called, have suffered for some time from their mutual competition and from that of the steel works. At present, orders for rails are waiting; on the other hand, the sale of bars and of sheets of the same metal share in the growing favour shown to the bars and plates of Bessemer or Martin steel, the price of which is constantly diminishing.

France continues to take from neighbouring countries, from Belgium, England, and Germany, for instance, nearly one-third of the mineral combustible necessary for its consumption. This fact is in itself a sufficient proof that the French collieries stand in need of considerable development. As regards the foreign trade, the state of affairs is

very different for the iron and steel works. They are sufficient for present requirements; several of them have created a market abroad, and export the excess of their production. Everywhere abroad, in England, the United States, Germany, Belgium, and Austria, the extraction of coal has redoubled in activity. More than one million human beings work daily in winning coals from the bowels of the earth. As far as can be judged by the official statistics of the principal civilised countries, the production of mineral combustibles in the whole world increased from 435 million tons in 1887 to 470 million tons in 1888.

For pig iron, the greater part of which forms the basis of the iron and steel industry, the production has increased approximately from 23 million tons to 23½ millions in the same period. The increase has been neither so marked nor so general as for coal. France maintains the fifth rank among the nations, under the double head of the coal industry and of the metal industry.

The number of workmen employed in 1888 in the mines of every kind in France and in Algeria was 116,000; moreover, the working of ores and of quarries has given work to 115,000 persons. Accidents were neither more serious nor more numerous than in the preceding year.

#### HIDE EXPORTS FROM INDIA.

The Calcutta *Englishman* for the 6th May says :—

India furnishes the United Kingdom with more hides and skins than any other country in the world, and during the last three decades the export of hides from Calcutta has trebled. Speaking broadly, the hide trade belongs to Bengal, and the skin trade to Madras, which is due partly to the fact that the total number of horned cattle is greater in the area served by Calcutta than in the area served by the port towns of Southern India, and also to the superior reputation enjoyed by the tanneries of the Madras Presidency in the dressing of skins. Hides and skins for export are gathered from all parts of India as far as the Punjab, and last year the number of hides imported into Calcutta was 120 lakhs.

#### CULTIVATION OF THE SUGAR BEET IN ONTARIO.

The Toronto *Monetary Times* for the 2nd May last has the following :—

An experiment has been in progress in Ontario since May last, undertaken with a view to learn how the sugar beet of Germany and Bohemia can be successfully grown in this country. Beet seed from those countries has been planted in Quebec. Mr Wilfrid Skaife, of Montreal, manager of the Berthier Beet Sugar Company, has been importing the seed, and getting the farmers along the St. Lawrence to cultivate the beet, with only fair success. He has sent for distribution through Ontario a quantity of sugar beet seed, in order to ascertain whether our soil and climate were adapted to produce roots of a quality suitable for the manufacture of sugar. This seed was accordingly distributed, in the hope that Ontario agriculturists would try them on a larger scale and with more pains than the French-Canadians seemed disposed to give. Should they do so, and with good results, Mr Skaife's company might open a branch factory in this province.

A warm interest in the experiment was taken by the Ontario Minister of Agriculture, who ordered a quantity for the model farm at Guelph. Last autumn he got Mr. R. H. Lawder to visit the farmers east of Toronto who sowed the seed and ascertain the results, to procure samples for analysis, and to report to the department. From the House of Assembly reports it is learned that there were drawbacks to the success of these experiments, the principal ones being that, owing to the lateness of the season when seed was sown, generally about May 15, many of the beets had not thoroughly matured at the time in October when the samples were taken; also want of information as to some of the peculiarities of sugar beet cultivation. For instance, close cultivation is necessary to secure rich saccharine quality and the roots need to be kept covered with earth. Our Ontario experimenters, however, set them out far apart, and allowed the roots to grow partly above ground.

The experiment was worth making, however, as proved by the results, which are encouraging. The yield per acre averaged over 20 tons; 80 per cent. of the roots analysed showed a percentage of sugar from 14 up to 17 per cent., but the purity of the sugar leaves room for much improvement, probably for the reasons given above.

The largest plot raised was on the Gulph farm, the yield from which is thought equal to 20 tons per acre, which is a fair yield for good soil in this province. Professor James analysed average beets from this crop, and reports polariscope reading 18 per cent., and the percentage of sugar in beet 17.10, coefficient of purity 83.7. These beets, says the report, were raised in rows 24 in. apart, and thinned out to 7 or 8 in. apart in the rows, and the roots in this case were all kept entirely underground. Such results as these, if they can be attained in various parts of the province, will make certain the success of the beet sugar.

#### INDIAN INDUSTRIAL PRODUCTS.

The following information respecting dyes and tanning materials and lac are extracted from a memorandum on Indian inland trade compiled in the Revenue and Agricultural Department of the Government of India, and which has recently been issued from the Government central printing office at Simla:—

*Dyes and tans* comprise indigo, myrabolams, cutch, turmeric, aniline dyes, and "others"; the first is by far the most important and stands at 323 lakhs of rupees\* in a total trade in dyes and tans valued at 442 lakhs of rupees.

Bengal, the North-Western Provinces and Oudh, and Madras, are the principal sources of commercial indigo, and their combined exports during the year amounted to 289½ lakhs of rupees, viz.: Bengal, 187½ lakhs of rupees; North-Western Provinces and Oudh, 73½ lakhs of rupees; and Madras, 28½ lakhs of rupees. It is also grown rather extensively in the Punjab, but chiefly for local consumption. Elsewhere its cultivation is not unknown, but it is unimportant.

Indigo manufacture in Bengal has a long history marked by many vicissitudes of fortune; but notwithstanding some serious checks, and in later years the competition of faniline and other dyes, it continues to hold its place as one of the great industries of the province. The total area under indigo in Bengal is estimated to be 588,000 acres, and the manufacture is in the hands of European capitalists. The season was fairly prosperous and the exports amounted to 90,616 maunds.

In the North-Western Provinces and Oudh indigo is largely cultivated in the districts to the east of Allahabad, and in the central and western half of the tract lying between the Ganges and Jumna rivers, where canal irrigation has led to a considerable extension of the acreage under this crop. The total area now under indigo in the North-Western Provinces and Oudh averages about 337,000 acres. Unlike Bengal, the manufacture is, except in the eastern districts adjacent to Behar, in the hands of natives. The crop of 1888—89 was not a good one owing to heavy and frequent rainfall, and exports fell from 43,000 maunds in 1887—88 to 40,000 maunds.

Madras indigo is commercially less valuable than that of Northern India; its cultivation is confined to the Northern Circars, but is extending, and the total area is between 400,000 and 500,000 acres. The exports were 23,866 maunds, against 26,000 maunds in the preceding year.

The indigo area in Punjab is returned as 138,000, of which 82,000 are comprised in the districts of Multan, Muzaffargarh, and Dera Ghazi Khan, where the indigo exported from the province is manufactured. The exports go to Sindh and Karachi. During 1888—89 amounted to 14,085 maunds, valued at 12.39 lakhs of rupees.

A noticeable peculiarity of indigo culture is the extent to which the cultivators of Behar depend on the North-Western Provinces and Oudh and the Punjab for seed. The exports of seed from these provinces average 1.5 lakhs of maunds a year.

Myrabolams, the fruit of a species of *Terminalia*, are exported principally from the forests of the Central Provinces and Bombay to Bombay town. During the year the exports of the Central Provinces amounted to 5.31 lakhs of rupees in value, and of the Bombay Presidency to 3.34 lakhs of rupees. With the opening of the railway from Nagpur to Bengal the trade may be expected to increase rapidly.

The Indian trade in cutch is insignificant compared with that of Burma, and the total value was under 8 lakhs of rupees. Of this about 2.5 lakhs of rupees is the value of cutch imported through Calcutta and Madras seaports from Burma. Punjab and Bombay derive their inland supplies from the North-Western Provinces and Oudh.

Turmeric, the roasted root-stock of *Curcuma longa*, is a condiment as well as a dye, and is extensively used by natives and Anglo-Indians for this purpose. The principal localities of production are Bengal, Madras, and the North-Western Provinces and Oudh. 6 lakhs of rupees went to port-towns, 7.5 lakhs of rupees to British provinces, and 2 lakhs of rupees to native States. A larger quantity went to the Punjab (value, 2.34 lakhs of rupees) than to any other province, which is perhaps to be partly accounted for by the fact that yellow is a favourite dye of the Sikh nation.

The growing popularity of aniline dyes is illustrated by the general distribution of the imports by sea among the internal trade blocks. Assam and Sindh are the only provinces which do not appear to import them. The Nizam's territory, Mysore, and Berar take them to a very moderate extent, but these provinces probably get the bulk of their supplies by road.

*Lac*.—Of the three commercial products of the insects (*Coccus lacca*) which causes the secretion of this resin, viz., stick-lac, shell-lac, and lac-dye, the trade in the two former only is separately recorded. Lac-dye, which is obtained from the washing of stick-lac and is a colouring matter derived from the female insect embedded in the resinous secretion, has, since the introduction of chemical dyes, almost disappeared as an article of export. Stick-lac is the crude product encrusting the twigs on which it is formed, but has, it is to be feared, been confounded in the returns in some instances, notably in the case of Assam and Punjab, with the manufactured article. Shell-lac is the pure resin extracted from stick lac, which is pounded and exposed to heat for the purpose. *Coccus lacca* has a wide distribution and affects various trees, but is collected principally from two or three species of fig and *Zizyphus* (ver. Ber) and *Butea frondosa* (ver. Dhak and Palas). The jungles of the Central Provinces, Chota Nagpore, Rewah (one of the Central India States), Mirzapore, and Assam are the chief sources of supply. In Assam the insect is regularly cultivated on two varieties of fig. It is also fairly abundant in parts of the Punjab. The inland trade of the year is valued at 101 lakhs of rupees. Calcutta received 17 lakhs of rupees from Bengal, 32 lakhs of rupees from the North-Western Provinces and Oudh, and 11 lakhs of rupees from Assam. The North-Western Provinces collect the produce of the Central Provinces, Punjab, and Rewah for exportation, and a portion of the consignments from Bengal to Calcutta consists of re-exports of arrivals from Assam.

#### GAMBIER AT SINGAPORE.

Gambier is one of the most important products of Singapore, but although it still maintains its high price, there are many complaints from England that the imported article is heavily adulterated with water. In order to trace the origin of this excess, samples taken from the field, fresh from the boiling-shed, were sent to Mr. Evans, of Bristol, whose analysis, with that as compared with a sample of block-gambier received by him in the ordinary course of trade, was as follows:—Gambier from the field: Tannin, 11.48 per cent.; organic matter, 30.11 per cent.; water, 53.39 per cent.; ash, 4.46 per cent.; less, 0.56 per cent. Trade gambier: Tannin, 14.68 per cent.; organic matter,

\* A lakh of rupees = 1,00,000 rupees.

42.26 per cent.; water, 31.89 per cent.; ash, 6.34 per cent.; loss, 4.48 per cent. This result shows that there is actually less water in the trade article than in the gambier taken directly from the coolies' hands, and negatives the suggestion that the town *towkay* adulterates the gambier after receiving it in Singapore with water to make it heavier. The other suggestion that the gambier has deteriorated of late years from insufficient inspissation, owing to less fuel being used in boiling, seems more probable. In earlier years, when there was no attempt made to protect the forests the destruction of firing was very large, and fuel could be had in large quantities. Now the results of wasteful destruction are being felt, firewood is becoming more expensive and difficult to get, and the gambier is insufficiently boiled and dried. Persons interested in the trade recently conceived the idea of forming a company in Singapore to cultivate gambier on a large scale, but this has fallen through, and there is an idea that this product may be cultivated more profitably—i.e., with European labour—in others of our colonies. Consequently, most botanical establishments have applied to the Singapore Gardens for seeds or young plants, and a large quantity of seed was carefully collected, dried,

and distributed widely, but, as far as has yet been heard, the experiments with it have failed entirely. It seems now certain that gambier seed has a very short duration of life (the Chinese say only 24 hours)—that is, it must be sown as soon as ripe. Thus all attempts to send seed to distant colonies must prove futile. Unfortunately, too, young plants are very bad travellers, and though many have been sent out to different establishments, few appear to survive the voyage. More plants, in as healthy as possible a condition, are now to be sent out to various colonies where gambier is likely to thrive.—*Annual Report of Singapore Botanical Gardens.*

#### RECENT PROGRESS IN SULPHURIC ACID AND ALKALI MANUFACTURE.

##### *Sulphur.*

In spite of the nearly complete abandonment of sulphur for acid-making in Europe, the production of the Sicilian deposits has not declined, as is indicated by the following table:—

PRODUCTION AND DISTRIBUTION OF SICILIAN SULPHUR.

	1883.	1884.	1885.	1886.	1887.	1888.	1889.
	Long Tons.						
Exports to England.....	41,788	40,760	33,402	30,236	31,086	37,455	37,633
Exports to France .....	63,602	65,098	58,264	51,280	56,221	53,927	68,632
Exports to Italy .....	66,810	56,292	49,415	48,658	46,818	48,436	44,317
Exports to America .....	96,629	94,929	90,378	98,590	88,593	128,953	111,838
Exports to all other countries.....	67,563	56,829	74,123	97,691	89,728	86,590	99,592
Total .....	336,392	313,908	314,582	329,455	312,446	355,361	362,012

The United States has for many years imported more sulphur than any other country; the above table shows that more than one-third of the Sicilian product came to America in 1888. In addition to this, constantly increasing quantities of sulphur are imported into the United States from Japan, amounting in 1888 to 6,332 tons. Practically no sulphur was mined in the United States in 1888, the deposits at Cove Creek, Utah, not having been worked, although they produced 3,000 tons in 1887.

Considerable quantities of sulphur, recovered from tank waste in England by the Chance process, have been sent to the United States during the past year, the importations for 1889 amounting to about 1,000 tons. The writer is informed by Mr. George Linder, of Boston, the American agent for the Chance Company, that contracts have been made for 500 tons per month during the present year. The Chance sulphur is nearly chemically pure, and commands a higher price than the Sicilian crude; its chief use thus far is in making sulphur dioxide for the sulphite process of wood pulp manufacture. It would seem particularly well suited for gunpowder making, but has not yet been adopted for this purpose.

The refining of sulphur is still chiefly carried on at Marseilles, where there are now six refineries in operation. These works, with five others at various points in the south of France, produce annually about 60,000 tons of refined sulphur, a very large part of which is used to combat diseases of the vine.

##### *Pyrites.*

The Spanish and Portuguese mines are producing pyrites in undiminished quantities. More than 2,000,000 tons were mined in 1887. The importation of pyrites into England shows no falling off, in spite of the precarious condition of the Leblanc soda process. According to the Board of Trade returns, the quantity of pyrites imported into the United Kingdom during the past six years is as follows:—

##### *Importation of Pyrites into the United Kingdom.*

	Long Tons.
1884 .....	563,073
1885 .....	654,521
1886 .....	556,988
1887 .....	597,595
1888 .....	617,232
1889 .....	643,879

This is practically all supplied by three great companies, the Tharsis and Rio Tinto, in Spain, and Mason and Barry, Portugal. The Rio Tinto Company made efforts some years ago to introduce Spanish pyrites into France and other countries, and to this end established a large plant for the manufacture of Leblanc soda and treatment of pyrites cinders for silver, gold, and copper at Marseilles. These works are still in operation, but owing to the unpromising outlook of the Leblanc process, the project of erecting similar works in Belgium, Austria, and America has been abandoned. French pyrites, containing no copper, mined at various points along the course of the Rhône, is employed at all the chief alkali works in France.

In America the production of pyrites has increased considerably within a few years. In 1888, 54,331 tons of pyrites were mined in the Eastern States, chiefly at the Davis mines, Franklin County, Mass. Large quantities of ore are also imported from Canada and Spain. According to W. H. Adams (*Manufacturers' Record*, February 1890), the mines of Virginia will produce during the present year fully 70,000 tons of pyrites. The same writer estimates the consumption of pyrites for the present year at 250,000 tons. The importations from Canada will probably reach 50,000 tons.

##### *Treatment of Pyrites Residue.*

The treatment of the cinders from burnt Spanish pyrites by the Henderson process is carried on in England on an enormous scale, and is constantly increasing. In 1888

420,415 tons of burnt ore were treated in Great Britain, producing 15,135 tons of copper, 337,500 ounces of silver, and 1,918 ounces of gold.

#### *Present Condition of the Alkali Industry.*

In spite of the strikingly rapid growth of the ammonia-soda industry, there has been no decline as yet in the production of alkali in Great Britain by the Leblanc process. This is shown by the statistics of the importation of pyrites, and by the following table compiled from the latest alkali inspectors' reports.

#### *Salt decomposed by both Processes in the United Kingdom.*

	1884.	1885.	1886.	1887.	1888.
	Tons.				
Leblanc process.....	578,874	598,096	584,323	577,381	590,312
Ammonia-soda process.	89,759	115,032	137,220	158,656	212,181
Total .....	668,633	713,128	721,543	736,047	802,493

These figures show that in Great Britain the older process is holding its own, though the world's growing demand for alkali is supplied by the increase of the new industry. Weldon showed five years ago that soda ash can be produced fully one-third cheaper by the ammonia-soda makers; the survival of the Leblanc process is, therefore, due to the enormous demand for bleaching powder (which has not yet been made economically by any other method), and to the profits gained in the treatment of pyrites cinders. Another important gain to the Leblanc industry is the success of the Chance process of sulphur recovery, which has now been introduced into most of the leading alkali works of Great Britain and the Continent. The effect of this new source of profit will be shown in the returns for the present year. The Leblanc alkali makers have lately devoted themselves almost entirely to the manufacture of caustic soda and soda crystals, for which the older process possesses important advantages over the new.

Outside of England the advance of the ammonia-soda process has been even greater, while the production of the Leblanc works has fallen off in a marked degree. According to the Paris Exhibition circular of the Solvay Company, the world's total production of alkali in 1888, calculated as soda ash, may be estimated at about 900,000 tons. Of this amount 430,000 tons, or about one-half, was made by the ammonia-soda process at the various Solvay works. The latter have increased their production still further during the past year, so that it may probably be safely said that more alkali is to-day produced by the ammonia process than by that of Leblanc.

There are now ten Solvay works in operation, the location and output of which are as follows:—

	Tons.
Belgium (Couillet).....	17,000
France (Varangeville-Dombasle).....	100,000
England (Brunner, Mond and Co., Northwich).....	125,000
Germany (Wylhen, Bernburg, and Saralbe)....	100,000
Russia (Beresniki and Lissitchevsk).....	17,000
United States (Syracuse, N.Y.).....	60,000
Austria (Ebensee, Salzkammergut).....	11,000
	<u>430,000</u>

The works at Geddes, near Syracuse, now supply about one-third of the American demand; in addition to the product of these works there was imported from England in 1888, 125,135 tons of alkali.

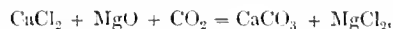
Other modifications of the ammonia-soda process, such as that of Parnell and Simpson, and the Schloesing process, introduced by Bell Bros., Middlesbrough, England, are worked only on an experimental scale, and do not yet play an important part in the alkali industry.

#### *The Hargreaves Process.*

The direct conversion of salt into sulphate of soda by the action of sulphur dioxide and steam has come into increased use during the past few years. There are now five works in England using this process, and also one in Ireland and two in France. During the year 1888 these works produced 43,080 tons of sulphate. The advantages of this method over the ordinary salt-cake process are the avoidance of the cost of making sulphuric acid, and the purity and freedom from iron of the sulphate so obtained. No fuel is found necessary, the heat of the reaction being sufficient after the operation is once started. The disadvantage of the method is the high first cost of the plant. Nearly all the plate glass now produced in Lancashire is made from Hargreaves sulphate.

#### *Chlorine and Bleaching Powder.*

The Weldon-Pechiney process, based upon the decomposition of moist magnesium oxychloride by heat into magnesia, hydrochloric acid, and free chlorine, is in successful operation at Salindres, France, and has been introduced on a large scale at the works of Albright and Wilson, at Oldbury, near Birmingham, England. This method accomplishes the final conversion into free chlorine of more than 90 per cent. of the hydrochloric acid employed, while the Weldon process, now universally employed, yields only one-third of the chlorine in a free state. An interesting application of the new process has been made at Szezakowa, Galicia, in the manufacture of chlorine from the waste products of the ammonia-soda industry. The residual calcium chloride is converted into magnesium chloride by treatment with magnesia and carbon dioxide—



(as in the last stage of the old Schaffner and Helbig method of sulphur recovery). The magnesium chloride is treated by the Weldon-Pechiney process for the production of chlorine, and the residual magnesia used over again. This method is a partial solution, at least, of the great problem of the production of chlorine from salt in connexion with the Solvay process. The great obstacle to the employment of the Weldon-Pechiney process on a large scale is, however, the dilute condition of the chlorine liberated, which makes it unfit for the manufacture of bleaching powder. At present the method is employed solely for the production of chlorates, for which it is especially adapted. Until the problem of the manufacture of bleaching powder from this weak chlorine is successfully worked out, it is not to be expected that the process will have an important influence on the alkali industry.

Other methods for the production of chlorine from the ammonia-soda residues appear to be unpromising. The Solvays are making hydrochloric acid on a small scale, at the Varangeville works, by heating calcium chloride with silica, but the plan makes no progress and is probably very uneconomical. Mond's method of obtaining ammonia and chlorine by passing ammonium chloride vapour over heated nickel or cobalt oxide is still wholly in the experimental stage, though the inventor promises full success in two years at farthest.

#### *Recovery of Sulphur from Tank Waste.*

The Chance process has been introduced at 20 works in Great Britain, one in Austria, and one in France. It was recently stated by the manager of the St. Gobain Company, France, that the sulphur so regained is sold at a price equal to three times its cost in the form of pyrites. Considerable quantities of the Chance sulphur are already being exported to America. An important advantage of the process to English alkali makers consists in its availability for the production of either sulphur or sulphuric acid. If pyrites is cheap, sulphur is manufactured and exported to America and other countries; if pyrites is expensive, the hydrogen sulphide produced from the tank waste is simply burned to sulphur dioxide and used over again for making acid. It is interesting to reflect that the sulphur imported into England

becomes a source of nuisance in the shape of tank-waste, but passes through the various stages of sulphur dioxide, acid, sulphate of soda, tank-waste and hydrogen sulphide, and finally appears at the end of the process as beautiful crystalline sulphur, a product of high commercial value.—*Am. Chem. Jour.*

#### MINERAL EXPORTS OF NEW SOUTH WALES.

The following comparative table, showing the mineral exports from New South Wales during the years 1888 and 1889, is compiled from information given in detail in the statistical register of the colony, the first part of which, for 1889, comprising the figures relating to trade and commerce, has lately been issued. From this it will be seen that there

has been an increase in the value of this class of exports of nearly a million sterling. The Broken Hill silver mines have, of course, the lion's share in bringing about this gratifying result, but they by no means stand alone. The value of silver, silver-lead, and silver ore, exported in 1889, exceeds the figures for 1888 by 828,793*l.*, an increase of about 80 per cent. Next in importance stands coal, with an increase of 214,799*l.*, or 20 per cent. The other lines which show increased exportation in 1889 are antimony and antimony ore, bismuth and its ore, copper ore, gold, both in bars and dust and in coin, kerosene shale, and lead. In the other items which go to make up the list, there was more or less of falling off in the value of the export, caused in some instances chiefly by fall in the market price of the metal:

#### EXPORT OF MINERAL PRODUCTS FROM NEW SOUTH WALES.

	1889.		1888.	
	Quantity.	Value.	Quantity.	Value.
Antimony (auriferous).....	1,516 cwt.	£ 1,094	384 cwt.	£ 553
Antimony ore.....	3,112 cwt.	2,250	3,423 cwt.	2,385
Bismuth.....	206 cwt.	7,060	22½ cwt.	410
Bismuth ore.....	644 cwt.	5,734	349½ cwt.	3,501
Bricks (including fire-bricks).....	266,890 No.	642	232,650 No.	942
Coal.....	2,387,702 tons	1,279,271	1,923,872 tons	1,064,472
Coke.....	9,522 tons	15,043	15,249 tons	24,331
Copper (refined).....	81,296 cwt.	207,000	75,721 cwt.	272,110
Copper (regulus).....	901 cwt.	1,166	1,176 cwt.	2,345
Copper ore.....	3,063 cwt.	2,156	1,090 cwt.	579
Fireclay.....	210 cwt.	37	280 cwt.	37
Gold (bars and dust).....	10,966 oz. 2 dwt.	44,649	6,896 oz. 4 dwt.	28,023
Gold quartz.....	2,163 pkgs.	2,038	6,523 pkgs.	8,426
Gold coin.....	.. ..	277,082	.. ..	138,317
Iron oxide.....	9,781 cwt.	1,329	15,286 cwt.	2,067
Kerosene shale.....	21,386 tons.	59,835	18,331 tons	52,634
Lead (pig).....	10,146 cwt.	6,721	3,575 cwt.	1,941
Manganese ore.....	.. ..	.. ..	460 cwt.	75
Minerals not specified.....	1,917 cwt.	719	2,386 cwt.	3,438
Pyrites.....	171 cwt.	141	64 cwt.	18
Silver (ingots).....	416,895 oz. 7 dwt.	72,001	375,063 oz. 14 dwt.	66,668
Silver-lead.....	691,836 cwt.	1,565,290	362,045 cwt.	911,117
Silver ore.....	839,309 cwt.	333,507	234,787 cwt.	164,620
Tin (ingots).....	88,173 cwt.	403,182	91,242 cwt.	569,182
Tin ore.....	4,687 cwt.	11,805	4,948 cwt.	13,314
Total value.....	.. ..	4,301,061	.. ..	3,343,155

The increase in export values during the past year, 957,906*l.*, is equal to nearly 29 per cent. It is more than two-fifths of the total increase in the exports from the colony which 1889 shows as compared with the previous year. The statistical returns of production are not yet issued, and it is from them and not from export figures that the condition of gold mining during the year is to be judged. The export of gold is not regulated by the output but by the state of exchanges. In regard to other metals the export returns are the best index we have as to the condition of the mining industry. Taken as a whole they are decidedly encouraging. So much attention has been given to silver mining of late, and so much capital directed

towards it, that it would not have been surprising had the production of other metals suffered in consequence. No doubt this has been the case to a certain extent, but happily not sufficiently to prevent the former export from being maintained, and in some cases exceeded. It should be mentioned that the figures given above for gold coin exported have been arrived at by deducting from the total exports the value of coin imported, and that of the bars and gold-dust sent from other colonies to be minted in Sydney. The actual amount of gold coin exported from the colony during 1889 was 3,157,965*l.*—*Engineering and Mining Journal.*

## UNITED STATES CLASSIFICATION OF PHOSPHATE LANDS.

The registrar and receiver of Gainesville, Fla., in a letter of 15th October 1889, asked the Department in Washington if lands containing phosphate came within the law relating to mineral lands. The Department replied on December 11th, 1889, that non-metallic mineral substances generally are classed as mineral; but, in the absence of any specific ruling as to phosphate, and because of regard due to the bona fide claimants under the homestead and pre-emption laws where the patents had not issued, Commissioner Groff directed a full report to be sent him, and meanwhile the receipt of mining applications for phosphate claims to be suspended. The registrar reported, December 20th, 1889, that the phosphate found in Marion and Citrus counties is of the highest quality, that the lands which must be surveyed in order to ascertain the quantity are more valuable for these deposits than for other purposes, and that the phosphate can be marketed at a fair profit. In reply to this report the commissioner rules, April 15th, 1890, that phosphate, included in "all valuable mineral deposits" in section 2319, Rev. Stat., "is subject to disposal under the mining laws as mineral land."—*Engineering and Mining Journal*.

## THE PROPOSED CHEMICAL UNION.

Negotiations have been going on for the formation of the Leblanc alkali manufacturers of the United Kingdom into a chemical union or syndicate on the same lines as the Salt Union. Valuations of works have been made, and the amount of capital has been fixed at something like 6,000,000*l*. It is said that the majority of the manufacturers are in favour of the scheme, but there are about five or six makers who up to the present decline to join the combination. The promoters of the scheme perceive that it would be dangerous if such a number of independent firms continued to work outside the Union; but they are still sanguine of gaining some, if not all, of the dissenting manufacturers into their fold. The Union would control the sales of all the chemicals made at the combined works, and hopes, in this manner, to circumvent the ruinous competition which has been going on for many years amongst the various manufacturers. The articles which it would be most easy for the Union to manipulate are bleaching powder and caustic soda, the former being entirely produced by the Leblanc process, and the latter, although it is possible to make it by the ammonium process, cannot be turned out in practice under that system on account of the higher cost of manufacture as compared with the former process. For soda crystals and soda ash the ammonia soda makers have the sole control of the market, and it is only to use up the sulphite of soda produced in the manufacture of bleaching-powder that Leblanc manufacturers produce these articles, as there is a heavy loss on them, which they must look to bleaching-powder to make good. The quantity of bleaching-powder produced in the United Kingdom in round numbers is about 150,000 tons, and of caustic soda 180,000 tons per annum, so that if the "Chemical Union" be successful, and these articles of manufacture would have to provide the dividends, consumers might expect to have to pay considerably more than they do at present for bleach and caustic. It is also possible that, as many of the Leblanc manufacturers are financially interested in the ammonia-soda companies, some arrangement might mutually be agreed upon to raise the price of soda products so that all round consumers would have to pay more for their chemicals.—*Chemist and Druggist*.

## THE DISTILLING INDUSTRY IN GERMANY.

Official statistics show a production of pure alcohol in the half year, ending April 30th last, amounting to 2,479,161 hectolitres. The exports of raw and refined spirits for the first quarter of 1890 amounted to 204,724 cwt. as against 40,266 cwt. in the corresponding period of 1889.—*Industries*.

## NEW QUICKSILVER MINES.

An important discovery of cinnabar has been made at Kurusz, in the district of Dagestan, in Poland. The ore assays 74·7 per cent. of mercury. Preparations are being made to work those deposits at once.—*Ibid*.

## EXHIBITION AT BARCELONA.

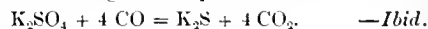
An exhibition of appliances for sprinkling vines with chemicals is to be held in Barcelona this year.—*Ibid*.

## DISCOVERY OF SALT BEDS.

Two important discoveries of salt deposits have been made. The borings which have been going on at Barrow-in-Furness on Walney Island have resulted in the discovery of an extensive bed of salt. Sinking operations, which have been carried on near Winsford, in Cheshire, have also resulted in the discovery of brine at a depth of 74 yards. The brine is equal to that generally found in the Cheshire district.—*Ibid*.

## FORMATION OF ALKALINE SULPHIDES.

M. Berthelot, in a communication to the Academy of Sciences on the reduction of alkaline sulphates by hydrogen and carbon, states that the first effect of hydrogen upon the sulphate—*e.g.*, potash—is to form potassium hydrosulphide and potassium hydroxide. The former in the next stage decomposes into potassium sulphide and sulphuretted hydrogen, and the latter combines with the caustic potash to form again potassium hydrosulphide and water. Another reaction takes place between the alkaline sulphide and water vapour, viz.:— $K_2S + H_2O = KHS + KHO$ . The reduction by hydrogen takes place at a comparatively low temperature. M. Berthelot also shows that solid carbon, even at a very bright temperature, fails to react with the sulphate, but that carbonic oxide at a bright red heat reduces the salt according to the equation—



## CHEMICAL MANUFACTURERS AND THE GERMAN LABOUR LAWS.

The chemical industries of the provinces of Rhenish-Prussia and Westphalia have decided to petition the German Reichstag, through their organisations, against the proposed law on Sunday rest and the liability of employers for accidents to their hands and the sanitary condition of the works. The petition points out that the Bill particularises over 280 different branches of chemical industry as coming under its provisions. Among these are a large number of processes in which it is absolutely necessary to continue working on Sundays and holidays. In some instances uninterrupted labour is required to produce the object of the manufacture, in others the machinery cannot be stopped without serious loss, especially in processes where a high or equal temperature is a necessity. With regard to the length of working hours, it is also pointed out that the course and duration of many chemical processes which require constant supervision cannot be estimated beforehand.—*Ibid*.

## CHEMICALS AND LONDON SEWAGE.

In view of the warm weather the London Council are taking the usual steps for applying chemicals to the sewage in various parts of London, and at outfalls upon the Thames. The expenditure for manganate alone is reported to amount to over 20,000*l*. a year. The condition of the river has decidedly improved between Gravesend and Greenwich, owing to the carrying out to sea of the wet sewage mud, of which it is reported that nearly 250,000 tons has been discharged in the Channel. Meanwhile Sir Benjamin Baker is engaged on the report he has to make upon the whole question of the London sewage system and its disposal.—*Ibid*.

## LANCASHIRE ALKALI WORKS AND THE PRODUCTION OF SULPHUR.

In the House of Commons, on the 22nd ult., Sir Lyon Playfair said: I beg to ask the President of the Local Government Board whether, in view of the recent establishment in Lancashire and elsewhere of an important manufacturing process for the extraction of sulphur from the tank-waste of alkali works, steps will be taken to include this and similar operations involving the production of noxious gases under the provisions of the Alkali Act. Mr. Ritchie: This matter has been receiving my attention, and I have given directions for the preparation of a Bill on the subject.—*Chem. Trade Journal*.

## A NEW RED GLASS.

A new red glass has been recently produced in Germany. Besides its use for the manufacture of bottles, goblets, and vases of various kinds, it will be found applicable in photography and in chemists' and opticians' laboratories. This glass is produced by melting in an open crucible the following ingredients: Fine sand, 2,000 parts; red oxide of lead (minium), 400; carbonate of potash, 600; lime, 100; phosphate of lime, 20; cream of tartar, 20; borax, 20; red oxide of copper—protoxide—9; and binoxide of tin, 13 parts. By a single melting a transparent red glass is said to be obtained of very fine quality, of which various objects can be manufactured directly without the necessity of a second heating to intensify the colour.—*Ibid*.

## THE IRON AND STEEL INDUSTRIES OF THE UNITED STATES.

If anything more were needed to illustrate the material progress made in recent years by the great Republic on the other side of the Atlantic, it is amply provided for by the report—always of considerable interest, but more than usually attractive on the present occasion—on the American iron trade in 1889, which has just been presented by Mr. James M. Swank to the American Iron and Steel Association, the headquarters of which are at Philadelphia. Beginning with pig iron, concerning which reliable data are available, thanks to the efforts of the American Iron and Steel Association and the British Iron Trade Association, we find that the output of crude iron during the last few years in the two countries was as follows:—

Years.	Great Britain.	United States.
	Tons.	Tons.
1882	8,586,980	4,623,323
1883	8,529,300	4,595,510
1884	7,811,727	4,097,868
1885	7,415,469	4,044,526
1886	7,000,754	5,683,329
1887	7,550,318	6,417,148
1888	7,998,909	6,482,738
1889	8,245,336	7,693,842

It will be seen that the British *maximum* production was attained in the year which begins the above table, while the United States are increasing their production of pig iron so steadily that they are evidently destined to become, at an early date, the leading pig-iron-producing country in the world, possibly attaining this distinction in 1890. That the United States are using proportionately more pig iron than Great Britain is shown by the fact that the consumption of crude iron in this country rose from 7,052,291 tons in 1888

to 7,692,230 tons in 1889, while in the United States it jumped from 5,864,950 tons to 6,962,800 tons.

If we turn to the production of steel in the United States and Great Britain, the comparison is even more striking. We subjoin a table showing the production of Bessemer steel ingots (including Clapp-Griffiths steel ingots) and steel rails in Great Britain in the last 13 years, compared with the production of the United States in the same period. For the sake of strict accuracy, it should be observed that in the ingot tonnage for the United States for 1889 is also included the small quantity of Robert-Bessemer steel made in that year.

Years.	Great Britain.		United States.	
	Ingots.	Rails.	Ingots.	Rails.
	Tons.	Tons.	Tons.	Tons.
1877	750,000	508,400	500,524	385,865
1878	807,527	622,390	653,773	491,427
1879	834,511	520,231	829,439	610,682
1880	1,044,382	732,910	1,074,262	852,196
1881	1,441,719	1,023,740	1,374,247	1,187,770
1882	1,673,649	1,235,785	1,511,687	1,284,067
1883	1,553,380	1,097,174	1,477,345	1,148,709
1884	1,299,676	784,068	1,375,581	996,983
1885	1,304,127	706,583	1,519,430	959,471
1886	1,570,520	730,343	2,269,190	1,574,793
1887	2,689,403	1,021,847	2,936,083	2,101,904
1888	2,032,794	979,083	2,511,161	1,886,277
1889	2,140,793	943,048	2,930,204	1,510,057

The race between the two largest steel producers of the world has been keen, but the United States have now for some years eclipsed this country in the output of Bessemer steel ingots and rails. In the production of ingots, the race was won by America only in 1884, while with regard to Bessemer rails, the supremacy of the United States was asserted in 1879. Since that year, the construction of railways proceeded at a tremendous pace in the States, the mileage built reaching a total of 11,569 miles in 1882, after which, the rate of building fell off somewhat; but the total rose to 12,872 miles of new track in 1887, the largest annual record ever made by any country.

If we consider all kinds of steel made by the two great steel-making countries, the United States were ahead of their European rival in 1886 and 1887. In 1888 and 1889, however, Great Britain again asserted her supremacy. She now excels as a steel producer, because of her large annual output of open-hearth steel, which is extensively used in ship-building in this country, a field in which the United States is idle, owing to its protective tariff having killed its mercantile marine.

The figures for the last six years' production of open-hearth steel are:—

Years.	Great Britain.	United States.
	Tons.	Tons.
1884	475,250	117,515
1885	583,918	133,876
1886	694,150	218,973
1887	981,104	322,069
1888	1,292,742	314,318
1889	1,429,160	374,543



The production of steel ignots of all kinds in the two countries in 1889 may be summarised as follows:—

Description.	Great Britain.	United States.
	Tons.	Tons.
Bessemer steel.....	2,140,793	2,930,204
Open-hearth steel.....	1,421,039	370,543
Crucible steel (about) .....	100,000	75,865
Other steel .....	Nominal	5,120
Totals .....	3,661,832	3,385,732

The English statistics of finished iron do not cover the same ground as those published by the American Iron and Steel Association, and therefore it is difficult to make comparisons. The output of puddled bars in Great Britain was as high as 2,841,534 gross tons (of 2,240 lb.) in 1882; it fell to 1,616,701 tons in 1886, rising again to 1,701,312 tons in 1887, 2,031,473 tons in 1888, and 2,253,756 tons in 1889. The production of rolled iron of all descriptions in the United States in 1882 was 2,493,831 net tons (of 2,000 lb.); 1883, 2,348,874 tons; 1884, 1,957,307 tons; 1885, 1,804,526 tons; 1886, 2,283,622 tons; 1887, 2,588,500 tons; 1888, 2,411,654 tons; 1889, 2,586,385 tons. In gross tons the quantities were 2,153,263 in 1888, and 2,309,272 tons in 1889.

Reference was made above to the large use of open-hearth steel in this country for ship-building purposes. When it is considered that the shipyards of the United States constructed but 34,354 tons of new shipping in 1887, 36,719 tons in 1888, and 53,513 tons in 1889, while the totals for Great Britain in those years were 577,327, 904,329, and 1,288,251 tons respectively, the insignificance of the American ship-building industry is obvious. The backward state of the United States with regard to the manufacture of tin plates, which is still in its experimental stage in that country, will probably not continue if the provisions of the Tariff Bill now before Congress are retained when the Bill is finally passed. It is surprising that no efforts have been made to "domesticate" such an important industry in the United States, as the use of tin plates is larger there than in any other country, and that the imports, from this country chiefly, are enormous. The British imports of tin plates into the United States have steadily grown from 82,969 tons in 1871 to 331,311 tons in 1889; the total quantity of imports in those 19 years being 3,293,404 tons, and their total foreign value 283,671,246 dols. (56,724,249*l.*) irrespective of the freights and duties paid by the importers. Possibly the increase in the tin plate imports may still continue, for the requirements of provision "canners" and petroleum "packers" are still growing, and they do not look with favour on the increase of duty now proposed.—*Chem. Trade Journal.*

### THE LAW OF PATENTS: SUGGESTED AMENDMENTS.

The Chemical and Allied Trades Section of the Manchester Chamber of Commerce has for some time had under consideration the bearing and application of the Patents, Designs, and Trade Marks Acts of 1883—88.

The Committee of the Section has arrived at the conclusion that certain provisions of the Patent Act of 1883 are mischievous and even impracticable when applied to chemical patents. The Committee is also of the unanimous opinion that some provision ought to be made in the law for distinguishing between mechanical and chemical patents, for the reason that however applicable the existing statutes may be to the former, it frequently happens that its provisions are absolutely impracticable when they come to be applied to the latter.

This fact has already been fully recognised in the German Patent Law by the enactment of special clauses and provisions for the administration of the law in the case of chemical patents. The Committee is further of opinion that if the principle were adopted of distinguishing between mechanical and chemical patents, the probability of litigation in the case of chemical patents would be considerably decreased, and greater justice would be done to litigants.

For the present the attention of the Committee has been directed to the anomalies which result from the operation of section 18, leaving for future deliberation other sections and provisions of the Patent Acts of 1883 to 1888. This section gives power to a patentee from time to time to disclaim, correct, or explain his specification. There is practically no safeguard for the public against the far-reaching power thus granted. It is true that the second clause of this section gives any person the right to oppose an application for leave to amend, and also that the eighth clause apparently limits the rights granted to the applicant by clause 1; but, in fact, both these protective clauses are altogether ineffectual to prevent unscrupulous patentees from evading both the letter and spirit of the law.

According to the existing law a patentee invalidates his patent—amongst other reasons:—

1. If he describes in his specification a process that does not produce the result claimed.
2. If he specifies one or more alternative processes for obtaining similar or equivalent results, and one of those processes does not produce the result claimed.
3. If, in his specification, he uses ambiguous, misleading, or evasive language.
4. If his description is insufficient to enable those skilled in the art to carry out the invention without other aid than that afforded by the specification.

The Committee holds that these just and equitable principles have been made absolutely inoperative by the application and effect of section 18, and that a patentee may now commit breaches of the four principles first set forth, and may abuse a monopoly and privilege which, in their opinion, is already far too easily granted to him, without running the slightest risk of vitiating his patent, because he can confidently trust to his benevolent friend, section 18, to supply him with all the protection of which he may stand in need, and to obtain for him condemnation of his former abuse of privilege, and thus enable him to make good a patent, to the possession of which he was never entitled.

A patentee may now specify and claim almost anything that his speculative and imaginative mind may suggest, without ever having made a single experiment to ascertain the possibility of the realisation of his dreams, so long as he is clever enough to make his original specification sufficiently wide and comprehensive, and to formulate it in such a manner as to permit him, in accordance with section 18, from time to time, to disclaim, correct or explain, without in any way enlarging his claims, or, after such amendment, making the invention substantially different from or larger than the original specification. Favoured by section 18, the speculative faculties of some patentees have of late years been so marvellously extended, that it now frequently happens that a patentee specifies, and claims as part of his invention, chemical products or substances which he has never even seen, much less made or produced.

It may, therefore, well be asked what are the consequences of such an anomalous state of things, in which we have, on the one hand, well-established law and practice, tending to prevent the abuse of a privilege, and on the other, laws to make their operation ineffective.

Great facilities are afforded by section 18 to unscrupulous patentees for evading the law, and the Committee trust they have succeeded in establishing the conviction that this section in its present form operates hurtfully and injuriously on our trades and industries. They now submit their propositions for preventing this abuse of privilege, without doing any injustice or restricting in any way the rights of honest inventors. These proposals are:—

1. To add to Part II., section 5, sub-section 4, of the Act of 1883, the following provisions, which shall be known as sub-section 4b:—"When generic terms are used in chemical

patents, the patentee shall specify *by name or otherwise* each substance or compound which he intends to include within such term or terms, and he shall only be entitled to claim as his invention those substances or compounds which have been so specified by him."

2. Sub-section 4c:—"There shall be deposited at the Patent Office, along with the complete specification of chemical patents, duplicate samples sufficient for analysis or examination of the result or results claimed, and of all raw and intermediate products used in obtaining such result or results, unless such raw or intermediate products shall be ordinary articles of commerce easily obtainable in this country."

3. Sub-section 4d:—"The complete specifications of all patents relating to coal-tar colours, in addition to the samples of the colouring matters themselves, and the intermediate substances used in obtaining them, shall be accompanied by dyed specimens of cotton-wool or silk, showing the results obtained with 1 per cent. and 2 per cent. respectively of the dyes claimed. A complete statement of the method employed in dyeing in each case shall also be given."

The Committee are further of opinion that the words in clause 1, section 18, "from time to time," shall be altered, in the case of chemical patents, into "two years from date of application."

The Committee recommends that a copy of this report be sent to all members of Parliament representing the Manchester district, with the request that they will take the first opportunity of bringing the questions of chemical patents and the working of section 18 before the House of Commons, and that the co-operation of other Chambers of Commerce be invited, especially of all those which are established in chemical centres, such as London, Liverpool, Glasgow, Newcastle, &c.

## BOARD OF TRADE RETURNS.

### SUMMARY OF IMPORTS.

	Month ended 31st May	
	1889.	1890.
	£	£
Metals.....	1,691,075	1,888,375
Chemicals and dyestuffs.....	810,562	649,493
Oils.....	438,509	460,957
Raw materials for non-textile industries.....	3,296,396	3,595,116
Total value of all imports ....	31,835,103	33,311,005

### SUMMARY OF EXPORTS.

	Month ended 31st May	
	1889.	1890.
	£	£
Metals (other than machinery) ....	3,220,679	4,278,321
Chemicals and medicines .....	667,892	828,279
Miscellaneous articles.....	2,812,786	3,102,118
Total value of all exports.....	20,421,394	22,910,779

### IMPORTS OF METALS FOR MONTH ENDED 31ST MAY.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
			£	£
Copper:—				
Ore..... Tons	14,713	9,115	83,762	64,938
Regulus .....	9,878	9,453	234,427	218,970
Unwrought .... "	3,758	2,581	151,106	119,563
Iron and steel:—				
Iron ore .....	362,904	345,113	253,100	261,577
Iron bolt, bar, &c. "	7,182	6,913	61,109	81,324
Steel, unwrought.. "	1,297	1,131	10,997	8,610
Lead, pig and sheet "	12,899	11,698	165,222	185,673
Pyrites .....	58,464	37,853	95,538	106,490
Quicksilver..... Lb.	139,252	934,631	11,201	124,351
Tin .....	49,061	41,788	222,703	195,360
Zinc .....	2,917	2,710	59,661	60,197
Other articles ... Value £	..	..	349,834	407,522
Total value of metals	..	..	1,691,075	1,888,375

### IMPORTS OF CHEMICALS AND DYESTUFFS FOR MONTH ENDED 31ST MAY.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
			£	£
Alkali..... Cwt.	4,448	3,369	3,612	2,295
Bark (for tanners, &c.) .....	63,138	52,961	50,771	28,038
Brimstone .....	56,433	34,003	12,737	7,005
Chemicals..... Value £	..	..	144,984	125,894
Cochineal .....	535	567	3,372	2,349
Cutch and gambier Tons	1,709	2,752	43,367	78,362
Dyes:—				
Aniline .....	..	..	20,102	23,956
Alizarine .....	..	..	59,692	23,317
Other .....	..	..	1,989	3,614
Indigo .....	3,592	1,829	59,294	27,538
Madder .....	1,260	1,426	1,758	1,833
Nitrate of soda.... "	109,567	237,220	177,912	92,617
Nitrate of potash . "	39,927	26,127	26,622	23,540
Valonia .....	3,002	1,511	47,292	50,687
Other articles... Value £	..	..	207,598	178,698
Total value of chemicals	..	..	810,562	649,493

## IMPORTS OF OILS FOR MONTH ENDED 31ST MAY.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Cocoa-nut..... Cwt.	2,029	2,012	£ 2,672	£ 2,508
Olive ..... Tuns	1,796	1,204	66,017	56,218
Palm ..... Cwt.	88,797	57,520	87,934	59,330
Petroleum ..... Gall.	5,651,699	7,364,122	131,625	172,723
Seed ..... Tons	1,611	2,197	90,838	57,176
Train, &c..... Tuns	1,678	2,116	36,802	46,637
Turpentine ..... Cwt.	3,819	3,012	6,122	3,765
Other articles .. Value £	..	..	63,539	62,390
Total value of oils ...	..	..	438,509	460,957

## IMPORTS OF RAW MATERIALS FOR NON-TEXTILE INDUSTRIES FOR MONTH ENDED 31ST MAY.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Bark, Peruvian .. Cwt.	11,211	8,830	£ 31,806	£ 19,917
Bristles..... Lb.	273,851	271,285	38,538	37,671
Caoutchouc..... Cwt.	15,871	18,333	151,526	214,712
Gum:—				
Arabic..... "	5,880	6,059	21,312	13,974
Lac, &c..... "	6,758	9,212	21,631	26,758
Gutta-percha .... "	3,457	6,705	43,456	65,513
Hides, raw:—				
Dry..... "	65,724	42,884	182,792	119,862
Wet..... "	51,852	50,591	120,591	115,629
Ivory..... "	288	970	9,293	51,995
Manures:—				
Guano..... Tons	1,639	2,871	11,745	14,660
Bones..... "	1,309	6,311	20,908	33,681
Paraffin..... Cwt.	32,813	58,312	36,098	76,731
Linen rags..... Tons	3,387	3,619	35,292	37,915
Esparto, &c. .... "	19,060	19,467	93,670	90,062
Pulp of wood .... "	16,281	13,468	55,185	68,711
Rosin..... Cwt.	86,702	148,287	191,130	35,817
Tallow and stearin "	155,264	130,991	196,991	158,664
Tar..... Barrels	2,184	8,329	1,552	4,794
Wood:—				
Hewn..... Loads	206,416	208,963	£ 0,836	£ 763,05
Sawn..... "	327,653	339,885	837,254	828,458
Staves..... "	16,181	13,375	70,141	51,546
Mahogany ..... Tons	3,834	3,914	34,815	37,715
Other articles..... Value £	..	..	802,966	813,303
Total value .....	..	..	3,293,506	3,395,146

Besides the above, drugs to the value of 97,305*l.* were imported, as against 69,818*l.* in 1889.

## EXPORTS OF METALS (OTHER THAN MACHINERY) FOR MONTH ENDED 31ST MAY.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Brass..... Cwt.	9,549	9,002	£ 43,678	£ 43,013
Copper:—				
Unwrought..... "	64,551	81,441	112,194	219,938
Wrought..... "	25,728	21,660	74,296	80,885
Mixed metal .... "	17,116	33,639	43,676	92,796
Hardware..... Value £	..	..	250,195	248,987
Implements..... "	..	..	108,383	124,250
Iron and steel..... Tons	379,117	422,080	2,579,585	3,131,553
Lead..... "	7,719	6,700	110,019	93,231
Plated wares... Value £	..	..	31,828	30,099
Telegraph wires... "	..	..	86,926	57,081
Tin..... Cwt.	11,452	9,792	51,365	47,116
Zinc..... "	23,337	20,969	15,292	19,241
Other articles .. Value £	..	..	77,242	90,151
Total value .....	..	..	3,620,679	4,278,321

## EXPORTS OF DRUGS AND CHEMICALS FOR MONTH ENDED 31ST MAY.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Alkali..... Cwt.	521,917	517,211	£ 132,942	£ 181,193
Bleaching materials ..	135,808	168,101	59,962	48,440
Chemical manures. Tons	22,113	21,223	163,268	168,028
Medicines..... Value £	..	..	93,285	87,917
Other articles .... "	..	..	257,497	313,601
Total value .....	..	..	697,892	828,279

## EXPORTS OF MISCELLANEOUS ARTICLES FOR MONTH ENDED 31ST MAY.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Gunpowder..... Lb.	1,683,900	869,500	£ 30,182	£ 18,020
Military stores.. Value £	..	..	85,796	122,080
Candles..... Lb.	935,100	1,178,900	17,069	21,894
Caoutchouc ..... Value £	..	..	93,014	100,611
Cement..... Tons	61,858	70,434	125,782	143,973
Products of coal Value £	..	..	99,081	128,052
Earthenware ... "	..	..	184,069	201,472
Stoneware..... "	..	..	18,614	19,991
Glass:—				
Plate..... Sq. Ft.	341,434	346,617	20,923	23,298
Flint..... Cwt.	9,288	9,227	23,605	22,500
Bottles..... "	105,035	89,633	49,239	42,906
Other kinds.... "	15,369	21,354	12,447	20,126
Leather:—				
Unwrought..... "	12,082	13,315	116,495	123,808
Wrought..... Value £	..	..	27,365	22,474

EXPORTS OF MISCELLANEOUS ARTICLES, &c.—*cont.*

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Seed oil..... Tons	7,376	5,555	£ 154,149	£ 138,710
Floor cloth ..... Sq. Yds.	1,787,800	1,536,800	82,064	73,122
Painters' materials Val. £	..	..	137,910	150,576
Paper ..... Cwt.	85,043	82,916	174,784	144,248
Rags..... Tons	5,190	5,564	41,171	38,833
Soap..... Cwt.	42,371	52,217	42,017	52,409
Total value .....	..	..	2,812,386	3,102,198

## THE TYNE CHEMICAL TRADE.

The exports of chemicals from the Tyne for the past month show a heavy falling off as compared with the corresponding month of last year. Caustic soda has not hitherto been recorded separately, being formally included under the heading of "other chemicals" :—

	May 1889.	May 1890.
	Tons.	Tons.
Alkali and soda ash .....	1,349	1,572
Bicarbonate of soda .....	10	8
Bleaching-powder.....	2,141	2,317
Manure.....	67	622
Soda crystals .....	2,593	1,511
Sulphate of soda .....	7	53
Caustic soda .....	..	1,240
Other chemicals .....	3,766	1,183
Total tons .....	9,963	8,516

—*Chemist and Druggist.*OUR EXPORTS OF DRUGS DURING THE LAST FIVE YEARS.  
(The figures are given in thousands of pounds) :—

Exports in 1,000l. to	1885.	1886.	1887.	1888.	1889.
Germany (with Holland and Belgium).....	979	1,005	980	976	1,036
France .....	354	407	510	551	627
Russia.....	92	79	85	86	83
Italy .....	93	109	145	134	187
Spain and Portugal.....	83	87	79	82	129
Other parts of Europe.....	97	101	83	82	81
Turkey and Levant.....	26	23	26	20	22
United States.....	952	730	879	890	867
Spanish America (including Brazil).....	222	243	263	283	317
China and Japan .....	71	80	102	79	109
British India and Ceylon ...	230	220	256	266	267
Australasia .....	446	398	401	439	441
British North America .....	76	86	91	87	103
British West Indies .....	60	61	65	73	65
South Africa.....	78	67	101	131	170
Other Colonies.....	13	14	14	46	16
Total 1,000l.....	3,877	3,710	4,089	4,235	4,523

## Monthly Patent List.

\* The dates given are the dates of the Official Journals in which acceptances of the Complete Specifications are advertised. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the said dates.

## I.—GENERAL PLANT, APPARATUS, AND MACHINERY.

## APPLICATIONS.

7886. T. Norman and H. T. Simpson. Improvements in furnaces to promote combustion. May 22.

8167. G. Moffat and S. Stuttaford. An improved apparatus for consuming smoke and saving fuel in furnaces. May 24.

8168. G. Moffat and S. Stuttaford. Improvements in apparatus for delivering air into the furnaces of steam boilers. May 24.

8192. G. E. Sherwin. Improvements in apparatus for heating, purifying, cooling, distilling, and evaporating water and other liquids. May 27.

8295. B. Versen. Improvements in the production of refractory linings for converter bottoms, and in apparatus therefor. Complete Specification. May 28.

8499. H. Schluzer-Berge and F. Schluzer-Berge. Improvements in air pumps. Complete Specification. June 2.

8523. E. F. Paddon. Improvements in furnaces. June 2.

8527. H. H. Lake.—From K. Müller, Germany. Improvements in air-compressors. June 2.

8544. W. H. Johnson and C. C. Hutchinson. Improvements in filter-presses. Complete Specification. June 3.

8551. C. Eitle. Apparatus for charging retorts. June 3.

8563. W. P. Thompson.—From J. A. Bowden, United States. Improvements in filters. Complete Specification. June 3.

8707. J. von Langer and L. Cooper. Improvements in and connected with gas furnaces for metallurgical and other industrial purposes. June 5.

8790. W. R. Watson and R. A. Robertson. Improvements in apparatus for evaporating, concentrating, and distilling liquids. June 7.

8915. O. Guttman. See Class VII.

9181. The Lucigen Light Co., Lim., and T. M. Jarmain. An oil flame furnace. June 13.

## COMPLETE SPECIFICATIONS ACCEPTED.\*

1889.

9321. F. M. Maynard. See Class XVII.

11,352. R. C. Garton, C. H. Garton, and W. Lawrence. Apparatus for evaporating, distilling, or effecting interchange of heat. June 11.

11,518. A. T. Clarksen and J. B. Spurge. Apparatus for regulating the flow of compressed gases. June 11.

11,600. A. G. Brown. Apparatus for separating water from steam. May 28.

11,865. L. Perkins. Refrigerating and freezing apparatus. June 4.

12,809. W. Maxwell. See Class XVI.

13,343. H. H. Lake.—From Kalle and Co. Transferred to Class IV.

18,153. J. Foster. Vacuum distilling and evaporating apparatus. June 11.

19,539. W. P. Thompson.—From Bordman. Apparatus for reducing mineral fuel, &c. to powder, and supplying same mixed with air to furnaces. June 18.

1890.

6107. J. C. Mewburn.—From C. C. Peck. Evaporation of brine and other solutions, and apparatus therefor. May 28.

## H.—FUEL, GAS, AND LIGHT.

### APPLICATIONS.

7757. C. P. E. Elmann. An apparatus for the production of gas from naphtha and kindred oils. May 19.

7830. B. C. Sykes and G. Blamires. The conversion of solid into gaseous fuel, and its purification. May 20.

7851. R. D. Rowman. Improved process and apparatus for the production of oxygen gas. May 20.

7889. W. Dyson. Improvements in the process of making gas and a product arising therefrom. May 22.

8109. E. Fremd. Improvements in the manufacture of gas. May 23.

8170. W. Clark. An improvement in the manufacture of illuminating gas. May 24.

8274. P. Shuttleworth. Improvements in the manufacture of fire kindlers. May 28.

8294. L. B. Lagoutte and G. de Velna. A process for solidifying mineral oils and applying them for combustion. May 28.

8319. J. C. Jopling. Appliance to increase combustion of gases and smoke in steam boilers. May 29.

8373. E. W. Walker. Improvements in and apparatus for the manufacture of gas for lighting and other purposes. May 30.

8535. S. Pitt.—From E. J. Jerzmanowski, United States. A process and apparatus for the manufacture of illuminating gas. June 3.

8752. W. Cryer. Improvements in means or apparatus for use in producing or separating oxygen and nitrogen gases from atmospheric air, also applicable for manufacturing other gases. June 6.

8909. H. Woodall. Improvements in apparatus for charging inclined gas retorts. June 9.

8930. L. S. d'Izoro. Improved machinery or apparatus for charging and drawing gas retorts. June 10.

8943. W. P. Thompson.—From O. Paternoster, France. Improvements in the manufacture of agglutinants, applicable for use in making coal blocks and other like conglomerates. June 10.

8958. J. Finay. An improved fire lighter, and machinery for manufacturing same. June 10.

9199. J. Bowing. Improvements in the manufacture of fuel from coal slack and similar substances, and in obtaining tar products. June 13.

### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

10,658. W. P. Thompson.—From A. Mason. Method and apparatus for burning culm or pulverised coal. June 4.

10,659. W. P. Thompson.—From A. Mason. Process and apparatus for burning coal and hydrocarbon fuel. June 4.

13,390. W. Fritsch. Coke ovens. June 18.

1890.

6584. J. J. Shedlock and T. Denny. Gas producers. June 4.

6877. J. J. Hiertz and A. S. Garnett. Combustible coal bricks. June 11.

## III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, ETC.

### APPLICATIONS.

7754. A. H. Allen and W. W. Staveley. Improvements in the production and application of the metallic compounds of phenoloids. May 19.

8728. F. Fenner and F. W. Colls. Improvements in the manufacture of naphthalene, and in means or apparatus employed therein. June 5.

### COMPLETE SPECIFICATION ACCEPTED.

1889.

12,028. J. D. Starck. See Class VII.

## IV.—COLOURING MATTERS AND DYES.

### APPLICATIONS.

7926. J. Y. Johnson.—From F. von Heyden, Germany. The manufacture and production of dioxynaphthalene carbon acids. May 22.

8215. J. Hall. Improvements in the manufacture and production of colouring matter. May 27.

8243. W. R. Lake.—From Wirth and Co., agents for A. Leonhardt and Co., Germany. Improvements in the manufacture of colouring matters. May 27.

8303. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik, Germany. Improvements in treating alizarine blue for the obtaining of new compounds thereof, and alizarine blue S. May 28.

8389. A. Remy, R. Kramer, and W. Herking. Improvements in the manufacture of colouring matters or dyes. Complete Specification. May 30.

8411. B. Willeox.—From The Farbenfabriken vormals F. Bayer and Co., Germany. Improvements in the manufacture and production of coumarin colouring matters. May 30.

8506. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. Production of a yellow colouring matter. June 2.

8530. B. Willeox.—From The Farbenfabriken vormals F. Bayer and Co., Germany. Improvements in the production of azo colours upon fibres. June 2.

8541. J. J. Hart. Improvements in the development or treatment of colouring matters in textile fabrics or materials. Complete Specification. June 3.

8725. B. Willeox.—From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture of new derivatives of alizarine and its analogues. June 5.

8726. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik, Germany. The manufacture and production of a new compound, and of artificial indigo from phenylglycecol or analogous compounds, or derivatives thereof. June 5.

8895. R. Holliday and Sons, Lim., and T. Holliday. Improvements in the manufacture of azo colours. June 9.

9001. B. Willeox.—From The Farbenfabriken vormals F. Bayer and Co., Germany. Improvements in the manufacture and production of nitroso combinations, and of colouring matters therefrom. June 11.

9080. J. Dawson and R. Hirsch. The manufacture of phenol ether and oxidiphenyl, their homologues and analogues. June 12.

9200. R. W. E. MacIvor. An improvement in the manufacture of ultramarine. June 13.

9258. O. Imray.—From H. K. Marburg, Germany. Improvements in the production of colouring matters. June 14.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

11,666. O. Imray. — From The Farbwerke vormals Meister, Lucius, and Brüning. Production of orange-red colouring matters from flavopurpurine. June 4.

12,031. O. Imray. — From The Farbwerke vormals Meister, Lucius, and Brüning. Manufacture of dyeing compounds consisting of a mixture of nitrite with basic amido-azo compounds, or with nitro compounds of aromatic monamines in the form of a paste; and application of such paste for diazotising. May 28.

12,191. C. Kolbe. Obtaining carbon acids of meta-amidophenol and its alkyl derivatives. June 4.

13,343. H. H. Lake. — From Kalle and Co. Production of quinazoline derivatives. June 18.

## V.—TEXTILES, COTTON, WOOL, SILK, Etc.

## APPLICATIONS.

7999. W. Gray and W. P. Thompson. Improvements in or appertaining to the separation or purification of cotton seed from its adherent fibre and also of other seeds or grains from external adherent matter or coating. May 22.

8148. J. B. Coleman, W. S. Coleman, and S. E. Coleman. Improvements in the sizing or treatment of lace and the like. May 24.

8296. The Serrell Automatic Silk Reeling Co., Lim. A new or improved process of preparing cocoons for reeling. Complete Specification. Filed May 28. Date applied for April 1, 1890, being date of application in France.

8420. R. C. Anderson. Improvements in the treatment of cloth and other absorbent materials or surfaces. May 30.

8452. E. Knecht. A process for the separation of certain textile fibres from each other. May 31.

8520. P. A. Favier. Improvements in machines for decorticating ramie or other textile plants in a dry or a green state. June 2.

8689. E. Sutcliffe and G. E. Sutcliffe. Improvements in coloured yarns. June 5.

## COMPLETE SPECIFICATION ACCEPTED.

1889.

9904. R. S. Burn. Treating cotton and other fibres preparatory to their being worked up into yarn. June 18.

## VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

## APPLICATIONS.

7786. A. F. S. George. A method of, and apparatus for, colouring and inlaying coloured designs upon and through floor and wall covering materials, and manufacturing the same. May 19.

7787. A. F. S. George. Colouring and inlaying coloured designs upon and through linoleum and other materials. May 19.

7841. H. A. McColl. Improvements in and relating to the printing of designs on handkerchiefs and other piece goods. May 20.

8183. J. M. Campbell. Improvements in colouring, sizing, and otherwise treating paper, and in apparatus therefor, also applicable for filling and treating woven fabrics. May 27.

8238. J. Bell. Improvements in dyeing and finishing of black stiff goods. May 27.

8270. E. Sutcliffe and G. E. Sutcliffe. Improvements in apparatus for washing, dyeing, and treating textile materials. May 28.

8530. B. Willeox. — From The Farbenfabriken vormals E. Bayer and Co., Germany. See Class IV.

8541. J. J. Hart. See Class IV.

8799. A. North. Black piece dyeing for improving the colour of the cotton warp or weft in mixed fabrics of wool and cotton. June 7.

8809. W. L. Wise. — From Favre and Braun, Germany. Improvements in the treatment of fabrics printed or dyed with several colours. June 7.

8896. R. Holliday and Sons, Lim., and T. Holliday. Improvements in the treatment of cotton dyed with certain azo colours. June 10.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

9692. A. Graemiger, W. T. Whitehead, S. Mason, jun., and E. A. Leigh. Machines for dyeing, bleaching, and treating yarn in cop, &c. May 28.

10,360. E. Maertens. Dyeing, &c., yarn or other fibrous material, and apparatus therefor. June 4.

11,098. T. O. Arnfield. Apparatus for dyeing, washing, &c., yarn in hanks. June 18.

11,382. G. B. Sharples. Apparatus for the continuous bleaching, soaping, &c., yarns of cotton, wool, and silk, &c. May 28.

1890.

5285. C. Kellner. Process and apparatus for bleaching vegetable fibres. June 18.

## VII.—ACIDS, ALKALIS, AND SALTS.

## APPLICATIONS.

7764. D. J. Playfair. Improvements in and connected with the obtainment of cyanides and ferrocyanides. May 19.

7983. J. C. Butterfield, T. C. Batchelor, and T. Fairly. Improvements in the manufacture of ammonium nitrate. May 22.

8090. J. D. Pennock and J. A. Bradburn. A new or improved process for manufacturing bleaching powder and caustic soda. Complete Specification. May 23.

8150. J. Leith. Improvements in the treatment of alkali waste, and in the manufacture of sulphuretted hydrogen and sulphide of ammonium, and in the arrangement of plant therefor or connected therewith. May 24.

8332. A. G. Greenway. Improvements in or appertaining to the treatment of galvanisers' waste acids or other similar waste liquors. May 29.

8454. T. H. Williams and W. H. Simons. A solution of calcium phosphate in carbonic acid water. May 31.

8733. W. Bruce. An improved method and plant for the manufacture of carbonic acid gas. June 6.

8786. G. Leon. A new process for the manufacture of fuming and anhydrous sulphuric acid. June 6.

8836. E. Blinkhorn. A process for rendering nitrate of ammonia non-hygroscopic. June 7.

8901. R. H. Davidson and R. H. Davis. Improvements in the treatment of alkali waste for the production of sulphuretted hydrogen. June 9.

8914. A. Crossley and J. W. Jones. Improvements in the process of, and apparatus for, producing ferro-ferrie and ferrie oxides. June 9.

8915. O. Guttman. Improvements in apparatus for the condensation of nitric acid and other distillation products. Complete Specification. June 9.

8942. J. Leith. Improvements in or connected with the manufacture of sulphuretted hydrogen and sulphide of ammonium and of alkali, and in the arrangement of plant therefor. June 10.

9112. F. Ellershausen. Improvements in the manufacture of caustic soda. June 12.

9198. W. L. Wise.—From The Chemische Fabrik Buckau Actiengesellschaft, Germany. Treatment of impure or waste potassium salts for the manufacture therefrom of soda, potash, hydrochloric acid, magnesia, gypsum, and sulphur or sulphuric acid. June 13.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

12,028. J. D. Starek. Process and apparatus for the production of fuming sulphuric acid or sulphuric anhydride, from the residues of the petroleum, paraffin, and tar industry. June 4.

13,264. J. Farmer. Manufacture of acetic acid and apparatus therefor. June 18.

1890.

4095. E. Bergmann. Production of cyanide compounds from ferrocyanide compounds. June 18.

### VIII.—GLASS, POTTERY, AND EARTHENWARE.

#### APPLICATIONS.

7822. D. Y. Cliff. Improvements in the means of effecting correct designs in decorative tiles. May 20.

7849. J. A. Jobling. Improvements in pots for melting glass. May 20.

8917. C. T. Maling. Improvements in the manufacture of china or earthenware mugs, jugs, and other vessels. June 9.

8977. J. Jacobs. Improvements in and relating to illuminating tiling. Complete Specification. Filed June 10. Date applied for December 12, 1889, being date of application in United States.

9172. R. J. Withers. Improvements in roof and hanging tiles. June 13.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

9841. F. C. Clare. Manufacture of terra-cotta porcelain castor bowls, &c. June 4.

11,979. H. H. Lake.—From A. Gilbert. Enamelling articles of copper. May 28.

13,577. M. Sugar.—From C. Lasalle and C. Mautter. Ornamenting glass. June 18.

1890.

6231. A. Fielding. Kilns for firing or hardening pottery. May 28.

### IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

#### APPLICATIONS.

7942. E. H. Edwards. Improvements in construction and working of cement kilns. May 22.

7962. R. Watkins. The utilisation of slate or slate refuse for making Portland cement. May 22.

8033. G. H. Skelsey. Improved means for the removal of the free lime and heat in newly-ground cement. May 22.

8108. F. Sarg. Improvements in concrete to be used in the construction of vaults, roofs, arches, floors, and the like. May 23.

8355. W. E. Keevil. Improvements in the treatment of Fuller's earth and in apparatus therefor. May 29.

8413. J. Hartnell. Improvements in the manufacture of artificial stone. May 30.

8519. O. Prinz. Improvements in the manufacture and treatment of cement. June 2.

8606. S. G. T. C. Bryan. Improvements in the treatment of slag for a variety of important uses, such as building blocks, paving blocks, &c. June 3.

8723. G. Ollier. Improvement in or relating to fire-resisting cements. June 5.

8801. J. D. Carr. An improved material or composition for covering walls, and for other decorative and useful purposes. June 7.

8919. D. Wilson. Improvements in apparatus for use in the manufacture of cement, and improvements connected with the said manufacture. June 9.

8993. V. L. Daguzau. A new or improved cementing material. June 10.

9239. D. Hart. Improvements in bricks for building purposes. Complete Specification. June 14.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1890.

4741. W. A. Gibbs. Cement kilns. May 28.

4995. G. A. Mansfield. Improvements in paving. June 18.

6855. G. Higham. Composition for use in connexion with the manufacture of Portland cement. June 11.

### X.—METALLURGY, MINING, Etc.

#### APPLICATIONS.

7820. F. G. Bates. Improvements in, and preparation of, materials for use in converting iron or low steel into high grade steel by the cementation process. Complete Specification. May 20.

7827. S. Fox. The application of water-gas to the manufacture of iron and steel in connexion with Siemens' and other gas furnaces. May 20.

7916. F. T. Thomas. An improved method of pickling and cleaning metal plates and sheets, and apparatus therefor. May 22.

8005. H. H. Lake.—From H. T. Smith, United States. Improvements relating to the formation of ingots for the manufacture of plated wire. May 22.

8032. W. H. Dowland. Improvements in the recovery of gold and silver by means of chlorine. May 22.

8295. B. Versen. See Class I.

8324. H. L. Wethered and A. E. Morgans. An improved method of and apparatus for recovering mercury and amalgam after treatment with mercury of gold and silver ores. May 29.

8352. W. McDermott. Improvements in apparatus for concentrating or separating ores and other substances. May 29.

8357. S. O. Cowper-Coles. Improvements in coating metallic surfaces with tin. May 29.

8399. H. Aitken. Improvements in means for the treatment of iron ores containing hydrocarbons, carbon, nitrogen, or sulphur. May 30.

8572. T. L. Thomas and J. B. Hillman. Improvements in or relating to the art or process of galvanising metal tubes, bars, plates, castings, ornamental ironwork, and other articles, and in means or appliances for use therein or relating thereto. June 3.



8606. S. G. T. C. Bryan. *See Class IX.*
8655. E. C. Miller.—From The Magnolia Anti-friction Metal Co., United States. Improved manufacture of compositions or alloys for anti-friction purposes. Complete Specification. June 4.
8707. J. von Langer and L. Cooper. *See Class I.*
8759. J. von Langer. Improvements in the manufacture of iron and steel. June 6.
8820. J. Clark and G. W. Clark. Obtaining alloys of aluminium with other metal or metals. June 7.
8826. J. Coombs. Improvements in gold extraction. June 7.
8867. W. Beardmore. Improvements in the manufacture of armour plates. June 9.
8884. J. W. MacFarlane. A method of extracting gold from auriferous ores by an improved process of amalgamation. June 9.
8900. J. C. Butterfield. Improvements in the treatment of sulphurised ores. June 9.
8908. F. G. Jordan. Improvements in the process of, and machinery for extracting gold from refractory ores. June 9.
8916. P. Ellershausen. Improvements in the extraction of metallic lead from ores containing the same. June 9.
8922. T. Peters. A process for the application of iron residues which result from the reduction of organic nitro-compounds. June 9.
8929. S. O. Cowper-Coles. Improvements in coating metallic surfaces with alloys. June 10.
8989. H. H. Lake.—From G. W. Cummins, United States. An improved process for treating copper and its alloys to prevent oxidation and deoxidation thereof during heating and annealing. Complete Specification. June 10.
8991. H. H. Lake.—From J. D. Brown, United States. Improvements relating to the manufacture and treatment of iron and steel, and to compounds for use therein. Complete Specification. June 10.
8992. H. W. Lash and J. Johnson. Improvement in the reduction of ore. Complete Specification. June 10.
9115. R. D. Bowman and H. J. Anderson. Process for the treatment of ores containing precious metals. June 12.
9123. A. Dauber. Improvements in making iron of any quality, pig iron, wrought iron, cast steel, rough steel. June 12.
9171. H. J. Smith. Improvements in and relating to the production of castings in ingot iron, steel, and other metal. June 13.
9182. C. R. Western. Improved means and apparatus for extracting gold and other precious metals from their ores. June 13.
9247. E. Edwards.—From E. Honold, Germany. Improvements in apparatus for and process for desilverising lead. June 14.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

6882. F. Knowles.—From T. C. Sanderson. Separation of gold from antimony. June 4.
8678. M. R. Couley and J. H. Lancaster. Apparatus for deoxidising, dephosphorising, desulphurising iron and other ores, and collecting and burning the gases therefrom. June 4.
9301. J. Gill. Blast furnaces. June 4.
- 10,279. R. E. Evenden. Apparatus for washing and separating gold and other minerals. May 28.
- 11,393. T. Parker. Manufacture of iron and steel. June 18.
- 11,707. T. Parker and A. E. Robinson. Manufacture of sodium, potassium, and magnesium. May 28.

- 11,882. S. Trivick. Roasting certain ores to obtain metals therefrom, and apparatus therefor. June 4.
- 11,883. S. Trivick. Extracting precious metals from their ores, and apparatus therefor. June 18.
- 12,113. C. L. Bath. Apparatus for forming copper ingots. June 4.
- 12,500. T. Twynam. Manufacture of iron and steel. June 11.
- 12,648. J. Clark. Obtaining alloys of aluminium with other metals. June 11.
- 12,811. T. Hydes and S. W. Wilkinson. Apparatus for casting molten metal, slag, &c. into pigs and blocks. June 18.
- 12,839. J. C. Bromfield. Manufacture of iron and steel, and utilisation thereof of waste products. June 18.
- 19,759. J. T. Penny and W. H. Richardson. Apparatus for triturating and amalgamating gold, silver, and other ores. June 11.

1890.

237. R. Brown and G. H. Irvine. Process and apparatus for extracting gold by the wet method from finely divided quartz ores, &c. June 11.
1794. C. Pfeiffer. Amalgamator for extraction of gold and silver from the ore by the action of mercury. June 18.
5003. A. K. Eaton and K. A. Vanderbilt. Obtaining chromium and chromium alloys. June 18.
5659. G. W. Marble and M. W. Simons. Forging metals, and machinery therefor. June 18.
5894. A. J. Boulton.—From J. W. Bookwalter. Converters. May 28.
6529. J. H. Bramwell. Hardening steel. June 4.
7461. H. H. Lake.—From The Russel and Erwin Manufacturing Co. Annealing metals. June 18.

#### XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

##### APPLICATIONS.

7853. W. P. Thompson.—From C. L. Coffin, United States. Improvements in welding metals electrically, and apparatus therefor. Complete Specification. May 20.
7876. H. H. Lake.—From E. A. Colby, United States. Improvements in methods of and apparatus for melting, refining, and casting metals and other substances by electricity. Complete Specification. May 20.
7988. C. L. Coffin. Improvements in welding metals electrically. Complete Specification. Filed May 22. Date applied for November 6, 1889, being date of application in United States.
7989. C. L. Coffin. Improvements in welding metals by electricity. Complete Specification. Filed May 22. Date applied for November 12, 1889, being date of application in United States.
7990. C. L. Coffin. Improvements in electric welding. Complete Specification. Filed May 22. Date applied for November 19, 1889, being date of application in United States.
7991. C. L. Coffin. Improvements in electric welding. Complete Specification. Filed May 22. Date applied for November 19, 1889, being date of application in United States.
7992. C. L. Coffin. Improvements in electric welding. Complete Specification. Filed May 22. Date applied for November 19, 1889, being date of application in United States.
7993. C. L. Coffin. Method of welding metals electrically. Complete Specification. Filed May 22. Date applied for December 6, 1889, being date of application in United States.

7994. C. L. Coffin. Method of welding metals electrically. Complete Specification. Filed May 22. Date applied for December 12, 1889, being date of application in United States.

8086. M. Bailey and J. Warnes. Improvements in secondary batteries. May 23.

8233. W. A. A. Dowden. The extraction of gold from its ores, consisting of a form of copper plate amalgamated with mercury, the action of which in retaining the gold upon it is aided by the passing of a current of electricity. May 27.

8242. W. R. Lake.—From H. Lemp, United States. Improvements in and relating to electric welding or metal working apparatus. Complete Specification. May 27.

8254. R. Haddan.—From F. Pescetto, Italy. Improvements in the manufacture of plates for electric accumulators. May 27.

8394. W. Wright. Improvement in battery compound. May 30.

8442. Sir C. S. Forbes, Bart. Improvements in primary batteries and in apparatus connected therewith. May 31.

8469. The Mining and General Electric Lamp Co., Ltd., and D. G. FitzGerald. Improvements in or connected with voltaic battery elements. May 31.

8534. N. de Benardos and Lloyd. Improvements in secondary batteries or electrical accumulators. June 2.

8578. J. Y. Johnson.—From C. Hering, France. Improvements in electrical accumulators or secondary batteries, and in the manufacture and production of plates or elements therefor. Complete Specification. June 3.

8703. W. P. Thompson.—From C. L. Coffin, United States. Improvements in electric welding. Complete Specification. June 5.

8716. E. Edwards.—From T. Lange, Germany. A new or improved process for obtaining, by electrical decomposition, metallic zinc from solution of sulphite of zinc. June 5.

8800. W. P. Thompson.—From I. Pearl, Spain. Improvements in or relating to electric accumulators. June 7.

8933. G. H. Felt. Improvements in the method of and apparatus for depositing metals by electro-deposition. Complete Specification. June 10.

9030. C. H. Freedman. Primary battery for open circuit work. June 11.

9257. S. Wohle. Process for effecting electrolytic deposit of aluminium. June 14.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

11,528. C. Beyer and G. Hagen. Electrodes for accumulators. June 18.

12,906. E. Hermite, E. J. Paterson, and C. F. Cooper. Electrolytic treatment of vegetable matters for the production of starch. June 18.

14,162. A. de Méritens. See Class XVII.

1890.

2302. H. Weymersch and R. McKenzie. Electric batteries working with two liquids or electrolytes. June 18.

3924. H. H. Lake.—From The Crosbig Electric Co. Electric batteries. June 18.

4087. H. T. Cheswright. Secondary voltaic batteries. June 11.

7383. J. N. Levens. Battery element. June 18.

7444. A. Million and P. Bony. Galvanic batteries. June 18.

## XII.—FATS, OILS, AND SOAP MANUFACTURE.

### APPLICATIONS.

7851. A. W. Rhenstrom. Improvements in the manufacture of soap. Complete Specification. May 20.

7891. T. H. Olyer. Hop soap. May 22.

7976. A. T. Hall. Improvements in refining vegetable oils. May 22.

8077. G. Allen. Improved composition of soap. Complete Specification. May 23.

8123. C. R. Hingworth. Improvements in soaps for skin disease. May 24.

8470. J. B. Koenig. Manufacture of soap combined with mineral oils. May 31.

8484. W. B. G. Hogg and J. G. Lorrain. A method of and apparatus for purifying oils and fatty substances. May 31.

9062. C. D. Hestrom. Improvements in the separation of fats from emulsions. Complete Specification. June 11.

9176. H. H. Lake.—From F. de la Croix, Belgium. Improvements in apparatus for desiccating and sterilising waste animal substances, and for extracting fat therefrom. June 13.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1890.

4809. J. Y. Johnson.—From M. Duclos. Manufacture of soap and glycerin. May 28.

5375. E. Noppel, B. Grosche, and J. Bigler. Apparatus for refining oil. June 11.

## XIII.—PAINTS, PIGMENTS, VARNISHES, AND RESINS.

### APPLICATIONS.

8250. G. L. Hille. Improvements in the treatment of waste india-rubber and in apparatus therefor. May 27.

8668. B. Piffard. Improvements in the manufacture of varnishes. June 4.

9108. S. Springer and J. R. R. Hawkins. A protective compound. June 12.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

8513. T. Hughes. Method and means for protecting submerged structures from effects of moisture, applicable also to construction and decorations of buildings. May 28.

12,292. R. Condy. A new compound to be used in paint or pigment. June 4.

12,756. R. Dick. Preparing and treating gutta-percha compounds. June 18.

1890.

5387. D. Fulton.—From D. F. Schmid. Anti-corrosive compound. June 4.

## XV.—AGRICULTURE AND MANURES.

## APPLICATIONS.

7890. W. Dyson. A new composition for tillage purposes. May 22.

8064. R. Fullarton. Improvements in treating or purifying phosphatic minerals. May 23.

8262. Sir E. R. Sullivan, Bart. Improvements in the manufacture of manures. May 27.

9129. H. H. Lake.—From A. Nenninger, United States. Improvements relating to the manufacture of acid phosphates for use as artificial manure. Complete Specification. June 12.

## XVI.—SUGARS, STARCHES, GUMS, Etc.

## APPLICATION.

8029. E. Stieger, E. Schulze, and C. Auer-Schollenberger. An improved process for treating vegetable cellular membranes with the object of obtaining glutinous substances. May 22.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

12,021. A. Chapman.—From C. A. Matthey. Process and apparatus for extracting sugar from sugar-cane, &c. June 4.

12,809. W. Maxwell. Multiple-effect apparatus for concentrating sugar juice, &c. June 18.

## XVII.—BREWING, WINES, SPIRITS, Etc.

## APPLICATIONS.

7878. F. König. Improved process of fermentation, and means and apparatus therefor. May 20.

7880. H. T. Brown, G. H. Morris, and E. R. Moritz. A process for preparing raw grain or other amylaceous substances for mashing in brewing or distilling operations. May 20.

8408. A. Walker. The improved mode of treating the wash in distilleries. May 30.

8850. E. J. Taylor. Improvements in the manufacture of whisky and other spirits. June 7.

9183. C. R. C. Tichborne, A. E. Darley, M. F. Pureell, and S. Geoghegan. Method of and appliances for the collection and utilisation of the carbonic acid gas and other products given off during the process of all fermentations. June 13.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

9321. F. M. Maynard. Valve, &c. for regulating flow of steam or liquids, especially for use with brewers' attenuators. June 11.

10,740. E. Larsen. Facilitating the drying of distillers' spent wash and similar matter. May 28.

10,891. J. Sleeman. Method of malting grain, and apparatus therefor. June 11.

12,186. P. C. Rousseau, M. J. de Chanterae, and M. J. D. A. de la Baume. Purifying alcohol, wine, and other alcoholic liquids. June 4.

12,187. L. Lederer. Manufacture of yeast. June 11.

13,054. L. Meyer. Producing ferments. June 4.

14,162. A. de Méritens. Treating and rectifying or ageing alcohol or alcoholic liquors by electricity. June 4.

1890.

3579. C. Hof.—From L. Rubsam. Production of malt. June 4.

6480. G. Schneider. Producing and storing beer without contact with the atmosphere. June 11.

6587. W. T. Whiteman.—From F. Ilawek. Process and apparatus for distillation and rectification of alcohols. June 18.

## XVIII.—CHEMISTRY OF FOODS, SANITARY CHEMISTRY, AND DISINFECTANTS.

## APPLICATIONS.

*A.—Chemistry of Foods.*

7551. A. W. Stokes. Improvements in the means of obtaining acidity or alkalinity in milk or other substances. June 3.

8018. J. White. The manufacture of a jelly powder. Complete Specification. May 22.

8354. W. C. H. Jessen. Improvements in preserving eggs. Complete Specification. May 29.

8804. W. H. Gover. Improvements in and connected with the manufacture of substances of food, especially relating to the manufacture of bread, bismits, cakes, infants' food, viz., hygienic phosphated food, bread, &c. June 7.

*B.—Sanitary Chemistry.*

7840. W. C. Young. Improvements in the purification of water by distillation. May 20.

7963. J. W. Lodge. Improvements in the method of, and apparatus for, purifying or treating river and sewage water, and recovery of solid matter found therein. May 22.

8239. W. S. West. Improvements in and relating to apparatus for handling and treating sewage. Complete Specification. May 27.

8498. J. Macfarlane and O. Prescott Macfarlane. The treatment of sewer gas and sewage. June 2.

8685. J. Chaffer and L. Simms. An improved method of and means for purifying sewage and other impure matters. June 5.

9203. A. R. Millbourn. The separation and recovery of all the component parts of various waste products. June 14.

*C.—Disinfectants.*

8838. D. C. Mackenzie. An absorbent deodorising and disinfecting powder for a boot and shoe powder, toilet, and other purposes. Complete Specification. June 7.

8938. E. Leman. An improved compound for preserving meat, fish, and poultry. June 10.

8997. G. Walker. Improvements in the preparation of disinfectants for more convenient transmission, carriage, storage, and general application. June 10.

9103. J. Y. Johnson.—From F. von Heyden, Germany. Improvements in the manufacture of disinfectants or antiseptics. June 12.

## COMPLETE SPECIFICATIONS ACCEPTED.

*A.—Chemistry of Foods.*

1890.

6421. A. G. Cunningham. Bread for dyspeptics. June 18.

*B.—Sanitary Chemistry.*

1889.

11,798. C. J. Moor. Treatment of sewage for purification and production of valuable substances. June 4.

## XIX.—PAPER, PASTEBOARD, Etc.

## APPLICATIONS.

7761. R. A. Sloan and J. E. L. Barnes. Improvements in machinery for the manufacture of wood fibre or wood wool. May 19.

7816. J. A. Loudon. The treatment of peat fibre for the manufacture of brown paper, wrappers, millboards, &c. May 20.

7971. F. W. Crowther. A new or improved metallic foil coated airtight and grease-proof paper. May 22.

8422. O. Evenstad and O. Senstad. Improvements in apparatus for use in the manufacture of wood-wool. Complete Specification. May 30.

8504. F. Butterfield and W. Renton. Improvements in the method of and apparatus for finishing paper. June 2.

8553. J. Hawke. Improvements in the manufacture of copying paper. Complete Specification. June 3.

9051. J. Luke. Improvements in and relating to the manufacture of paper for mill wrappers, lappings, boxes, and such like, and in machinery connected therewith. June 11.

9135. J. Craig. Improvements in the manufacture of paper. June 13.

## COMPLETE SPECIFICATION ACCEPTED.

1890.

4960. C. Kellner. Process and means for purifying paper pulp. June 18.

## XXI.—PHOTOGRAPHIC PROCESSES AND MATERIALS.

## APPLICATIONS.

8193. W. S. Wardle. Improvements in photography. May 27.

8214. M. A. Wier. New or improved means for developing photographic films in actinic light. May 27.

9218. F. W. Hayward. Improvements in colouring and softening photographs on paper. June 14.

## COMPLETE SPECIFICATIONS ACCEPTED.

1890.

6028. P. Rudolph. Photographic objectives. May 28.

6029. E. Abbe and P. Rudolph. Photographic objectives. May 28.

## XXII.—EXPLOSIVES, MATCHES, Etc.

## APPLICATIONS.

7839. W. Cross. Improvements in the manufacture of matches, and in boxes therefor. May 20.

8181. P. Ward and E. M. Gregory. An improved priming and detonating composition. Complete Specification. May 31.

8717. E. Binkhorn. New or improved manufacture of explosives suitable for use in lieu of fulminate of mercury or the like for detonating purposes. June 5.

8811. H. Koff. Improvements in the manufacture of gunpowder. June 7.

8843. E. Muller. An apparatus for nitrifying cotton, cellulose, straw, and similar material. Complete Specification. June 7.

9197. P. P. Alexander. Improvements in electrical fuses. June 13.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

11,664. Sir F. A. Abel and J. Dewar. Manufacture of explosives. May 28.

11,667. Sir F. A. Abel, J. Dewar, and W. Anderson. Process and apparatus for manufacturing explosives in the form of wires or rods and forming same into cartridges. June 18.

## XXIII.—ANALYTICAL CHEMISTRY.

## COMPLETE SPECIFICATION ACCEPTED.

1889.

18,856. A. W. Gerrard. Apparatus for estimating the quantity of glucose or grape sugar in fluids. June 4.

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Members who require extra sets or back numbers of the Journal are requested to make application to the General Secretary only, to whom also changes of address should be communicated.

Authors of communications read before the Society, or any of its Local Sections, are requested to take notice that under Rule 41 of the bye-laws, the Society has the right of priority of publication for three months of all such papers. Infringement of this bye-law renders papers liable to be rejected by the Publication Committee, or ordered to be abstracted for the Journal, in which case no reprints can be furnished to the author.

Notice is hereby given, for the information of members and advertisers, that the advertisement columns of this Journal have been contracted for by Messrs. EYRE and SPOTTISWOODE, the Society's printers and publishers, to whom all communications respecting them should be addressed.

The Society's Office will be closed from the 5th to the 16th August inclusive.

The Secretary is instructed to negotiate for the purchase of copies of the Society's Journal for January 1883, and January, February, and April 1886. Members possessing odd copies of these numbers are particularly requested to communicate at once, stating price required, with Mr. Cresswell. The stock of all other numbers is at present sufficient for the Council's requirements.

#### MEMBER ELECTED, 9th JULY, 1890.

Mendeleeff, Prof. D., St. Petersburg, professor of chemistry.

#### LIST OF MEMBERS ELECTED, 23rd JULY, 1890.

Bairstow, John, 32, Sealand Road, Chester, chemical works manager.

Brierley, J. T., 249, Bolton Road, Chorley, Lancashire, analytical chemist.

Clark, Franklin S., 527, Madeira Avenue, New York City, U.S.A., manufacturing chemist.

Devey, Arthur C., c/o La Société Hermite, 4, Rue Drouot, Paris, bleacher.

Donald, Saml., Corporation Gasworks, Dalmarnock Road, Glasgow, analytical chemist.

Frankenstein, L., 32, Dover Street, Oxford Road, Manchester, india-rubber manufacturer.

Frusher, T., White Abbey Dye Works, near Bradford, Yorks, master dyer.

Goodall, Geo., 182, Mansfield Road, Nottingham, hosier.

Goulding, W. J., 25, Eden Quay, Dublin, fertiliser manufacturer.

Harris, Sydney J., Mechanics' Institute School of Science, Leeds, head master.

Hodgson, Wm., 8, Victoria Buildings, St. Mary's Gate, Manchester, oil broker.

Johnson, Robt. W., New Brunswick, N.J., U.S.A., manufacturing chemist.

Kirkland, John, 42, Eland Road, Lavender Hill, S.W., bread works manager.

Mond, Alfred M., Winnington Hall, Northwich, analytical chemist.

Santer, Ernest H., 176, High Street, Redcar, Yorks, analytical chemist.

Thomson, W. T., 156, Bath Street, Glasgow, analytical chemist.

#### CHANGES OF ADDRESS.

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Bartlett, F. L., 1/o Portland; Canon City, Colorado, U.S.A.

Beckett, J. H., 1/o Wilmslow; Corbar Hill House, Buxton.

Bernard, Jas., 1/o Glasgow; Tinoen, Lim., Casal das Rollas, Poco do Bispo, Lisbon.

Brown, R. J., 1/o Davenport; Technical School, Stockport.

Carmody, Prof. P., 1/o Somerset House; Government Laboratory, Port of Spain, Trinidad.

Chamberlain, Jno., 1/o Melbourne; Brunswick Gasworks, near Melbourne, Victoria.

Elmore, A. S., 1/o Rothwell; Spring Grove, Low Road, Hunslet, Leeds.

Fletcher, R. Steele, 1/o Abbey Walk; c/o Fletcher Bros. and Co., Grimsby.

Griffith, D. Agnew, Journals to c/o Cowan and Son, Hammersmith Bridge Works, Barnes, S.W.

Hailes, A. J. de, 1/o Hornsey; 92, Manor Road, Stoke Newington, N.

Horton, Wm., 1/o Edge Lane; 38, Belvidere Road, Prince's Park, Liverpool.

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#### CHANGE OF ADDRESS REQUIRED.

Huy, Alex., 1/o Kirby House, Arboretum Avenue, Lincoln.

#### Death.

T. R. Ogilvie, 1/o Mill Lane House, Plymouth, at Melbourne.

## PROCEEDINGS

OF THE

## NINTH ANNUAL MEETING.

THE Annual General Meeting of the Society was held on Wednesday, the 9th July, in the Chemical Lecture Theatre of University College, Nottingham.

The President, Sir LOWTHIAN BELL, took the Chair at 11 o'clock, and called upon the General Secretary to read the Minutes of the last Annual Meeting. The Minutes having been duly confirmed—

The Mayor (Alderman GOLDSCHMIDT) said it afforded him much pleasure to offer the Society a hearty welcome to Nottingham. He hoped that the meeting would be a successful one in every respect, and that the members would carry away none but pleasant recollections of their visit.

The President said that it was a great pleasure to him to offer the hearty thanks of the members present for the very kind welcome which had been extended to them. His own recollection of Nottingham carried him back to 25 years ago, since which time he had not had the pleasure of being in the town. On that occasion he had enjoyed the hospitality of the then Mayor, and from the lively recollection which he had preserved of that visit he had no doubt that the pleasure which he had then experienced would be repeated on the present occasion. He wished to tender to the Mayor and Corporation the thanks of the members, not only for his kind words of welcome, but also for the vigorous measures (which he had no doubt would prove successful) that had been taken to conduce to their pleasure.

The Mayor then left the meeting.

Dr. C. R. ALDER WRIGHT moved: that Messrs. S. V. Holgate and E. N. Butt be appointed scrutators. The motion was seconded by Mr. T. Tyrer, and carried unanimously.

The ballot was then declared closed.

The President then called upon the General Secretary to read—

## THE REPORT OF THE COUNCIL.

The Council has the honour to report that the membership of the Society has increased from 2,445, as reported at the last meeting, to 2,595, or a net increase of 150 members. During the year 298 members have been elected, and 148 have been removed by death, resignation, and other causes.

During the past session 73 papers and 14 communications have appeared in the Journal as against 55 papers and 5 communications the year before. To the authors of these the Council would offer its best thanks.

The Journal is now recognised as the leading authority on Applied Chemistry in the English language, and forms an admirable work of reference. The number of subjects treated under the 23 headings of the Journal and Patent Literature amounts at present to about 60, and is not likely to increase much in the future. Attention will now be devoted to the consolidation and perfection of each group in order that the specialist may find there a full and complete record of contemporary work.

On the 23rd June last the Treasurer reported the sum of 1,720*l.* 6*s.* in the bank, as against 1,214*l.* 4*s.* 5*d.* a year ago. The investments of the Society remain as last year, viz. 3,688*l.* 15*s.* 9*d.*; but it is intended to add to this sum shortly 165*l.*, derived from 11 Life Composition Fees, which

have been received during the last two years. The revenue last year, as will be seen from the Treasurer's report, exceeded the expenditure by 607*l.* as against 918*l.* 14*s.* 9*d.* in 1888, but this reduction was foreseen last year. It is mainly due to diminished returns from advertisements and increased expenditure upon the Journal, which is money well spent.

The Council has the pleasure to announce that, in constituting the permanent governing body of the Imperial Institute, the interests of the chemical industries have not been passed over, and the appointment of one member has been allotted to the Council of this Society.

Mr. E. K. MUSPRATT, in moving the adoption of the report, said that he was glad to see that the Society continued to prosper, and he hoped that the coming year would bring it even greater prosperity than that shown by the present report.

Professor CLOWES seconded the motion, observing that the report must be felt to be eminently satisfactory. It showed progress, which was all that could be desired. The motion having been carried unanimously,

The Hon. Treasurer (Mr. E. RIDER COOK) presented the accounts for the year, printed on page 690. In doing so he said that the printed figures would speak for themselves, and that all he need do was to add a few words supplementary to what had been said in the Annual Report of the Council as to the increase in the membership. That increase was a practical one, for he had already received 2,259 subscriptions as against 2,152 at the same date last year, and as against 2,233 for the whole of 1889, so that he had already in hand 26 more subscriptions than the total for last year. He would, however, warn the members that, the Council having taken a somewhat extravagant fit as regarded expenditure, they must not expect to find in the coming year a surplus equal to that shown in the accounts now presented. If he could contrive to save 600*l.*, he was satisfied that it would be due to the large increase in the membership, and not to a decrease in the expenditure. He was glad to be able to say that members had taken the hint he had given last year with respect to sending receipts for subscriptions by return of post; and although the commencement of the current year had brought him the usual rush of work, he was thankful to say that members had forborne to complain of the consequent delay in getting their receipts.

The PRESIDENT, in moving the adoption of the accounts and a vote of thanks to the Hon. Treasurer, said that he wished to say a few words in commendation of the Hon. Treasurer's report. It was not often that a Society was fortunate enough to possess a treasurer who would bear admonition and act upon it in the conscientious way that Mr. Rider Cook had done. He would not like to measure the utility of such an establishment as the Society of Chemical Industry by the amount of money it had in the bank. He did not by any means wish to have an overdrawn account, but he believed that any balance they might have would be most usefully employed in helping to perform the functions of the Society; and unless there should be a prospect of requiring a large sum of money for some immediate purpose, such as building a palace for the Society, the best thing they could do with their revenue was to spend it for the benefit of the present members.

Mr. L. ARCHBUTT (in the unavoidable absence of Sir John Turney) seconded the motion, which was carried by acclamation.

The PRESIDENT then said that no one who took the trouble to examine the result of the labours of the Council and of the Society could fail to be impressed with the high honour which was involved in being appointed to fill the office which had fallen to his lot. Under these circumstances he was sure that the meeting would excuse him for trespassing upon their time in order that he might express his thanks to his colleagues and the members generally for having appointed him to fill the post of President during the past year.





TABLE I.

	A. Cold Blast.	B. Hot Blast.	C. Hot Blast.	D.	E.	F.	G.	H.
Height and cubic capacity of furnace.....	48 ft. 6,000 c. ft.	48 ft. 6,000 c. ft.	80 ft. 15,500 c. ft.	90 ft. 32,488 c. ft.	80 ft. 15,500 c. ft.	76 ft. 20,454 c. ft.	65 ft. 10,300 c. ft.	42 ft. Sweden. 1,888 c. ft.
Temperature of blast.....	25° C.	485° C.	485° C.	710° C.	704° C.	765° C.	536	200° C.
" escaping gases.....	430° C.	451° C.	310° C.	222° C.	250° C.	301° C.	301° C.	222° C.
Consumed per ton of pig iron :—	Cwt.	Cwt.	Cwt.	Cwt.	Cwt.	Cwt.	Cwt.	Cwt. Charcoal.
Coke.....	40·76	28·50	22·50	19·69	19·99	19·49	21·06	15·44
Limestone.....	18·25	16·00	11·00	10·50	11·00	..	9·95	4·36
Time.....	..	..	..	..	..	CaCO <sub>3</sub> 6·87 = 12·28	..	..
Ore.....	46·20	48·00	48·00	48	48	48	34·61	36·44
Description of ore.....	Clay ironstone.	Lias claystone.	Lias claystone	Lias claystone.	Lias claystone.	Lias claystone.	West Coast Hem.	Hematite, Sweden.
	Cwt.	Cwt.	Cwt.	Cwt.	Cwt.	Cwt.	Cwt.	Cwt.
Weight of blast per ton of iron.....	180·46	126·05	100·41	84·01	87·15	79·37	87·31	58·38
" escaping gases per ton of iron..	233·92	168·37	135·20	115·89	119·47	106·81	118·62	81·04
" slag made per ton of iron.....	34·15	31·00	28·00	28·00	28·00	28·50	15·02	12·01
Pig iron :								
Weekly make.....	90 tons	220 tons	350 tons	700	350	613	600	..
Quality.....	No. 3	No. 3	No. 3	No. 3	No. 3	No. 3	Bessemer	Forge gray
Make per 1,000 ft. cubic capacity....	15·0 tons	36·7 tons	22 tons	20·9	22 tons	30	..	..
Appropriation of heat produced, viz.....	111,180 cal.	104,336 cal.	91,194 cal.	85,912	88,577	79,958	79,056	61,365
Constants per ton of pig iron:—	Calories = Cwt. Coke.	Calories = Cwt. Coke.	Calories = Cwt. Coke.	Calories = Cwt. Coke.	Calories = Cwt. Coke.	Calories = Cwt. Coke.	Calories = Cwt. Coke.	Calories = Cwt. Chrl.
Reduction of Fe <sub>2</sub> O <sub>3</sub> in ore.....	34,176 = 12·532	33,108 = 9·04	33,108 = 8·17	33,108 = 7·59	33,108 = 7·47	33,108 = 8·07	32,565 = 8·67	32,565 = 8·22
" metalloids in pig.....	1,600 = 0·587	4,174 = 1·14	4,174 = 1·03	4,174 = 0·96	4,174 = 0·94	4,174 = 1·02	521 = 0·14	521 = 0·13
Dissociation of CO.....	1,440 = 0·528	1,440 = 0·40	1,440 = 0·35	1,440 = 0·33	1,440 = 0·33	1,440 = 0·35	1,920 = 0·51	1,920 = 0·48
Fusion of pig iron.....	6,800 = 2·420	6,800 = 1·60	6,600 = 1·63	6,600 = 1·51	6,600 = 1·49	6,000 = 1·61	6,600 = 1·77	6,600 = 1·67
Sum of constants.....	43,816 = 16·067	45,322 = 12·38	45,322 = 11·18	45,322 = 10·39	45,322 = 10·23	45,322 = 11·05	41,608 = 11·09	41,606 = 10·50
Variables, per ton of pig iron:—	Calories. Cwt.	Calories. Cwt.	Calories. Cwt.	Calories. Cwt.	Calories. Cwt.	Calories. Cwt.	Calories. Cwt.	Calories. Cwt.
Evaporation of water in coke.....	124 = 0·045	400 = 0·11	313 = 0·08	275 = 0·06	275 = 0·06	275 = 0·07	1,270 = 0·34	1,000 = 0·25
Decomposition of water in blast.....	3,780 = 2·119	3,740 = 1·02	2,720 = 0·66	2,380 = 0·55	2,380 = 0·54	2,380 = 0·58	3,550 = 0·66	1,360 = 0·35
Expulsion of CO <sub>2</sub> in limestone.....	6,752 = 2·476	5,920 = 1·61	4,070 = 1·00	3,885 = 0·89	4,070 = 0·92	677 = 0·16	2,681 = 0·98	1,636 = 0·40
Reduction of this CO <sub>2</sub> to CO.....	7,005 = 2·570	5,144 = 1·68	4,224 = 1·04	4,034 = 0·92	4,224 = 0·95	704 = 0·17	3,808 = 1·01	1,564 = 0·41
Fusion of slag.....	18,782 = 6·887	17,050 = 4·66	15,400 = 3·81	15,400 = 3·53	15,400 = 3·48	15,775 = 3·84	8,261 = 2·20	6,605 = 1·67
Carried off in escaping gases.....	24,148 = 8·855	18,224 = 4·99	10,058 = 2·49	6,174 = 1·41	7,166 = 1·60	7,715 = 1·90	8,569 = 2·29	4,139 = 1·05
Heat lost by radiation and expansion of blast, tyvere water, and balance to make both sides agree.....	4,770 = 1·749	7,596 = 2·05	9,087 = 2·24	8,442 = 1·94	9,740 = 2·21	7,110 = 1·72	9,361 = 2·49	3,355 = 0·81
Sum of variables.....	67,364 = 24·701	59,014 = 16·12	45,672 = 11·32	40,590 = 9·30	43,255 = 9·76	34,636 = 8·44	37,450 = 9·97	19,759 = 4·94
Total. Calories of cwt. fuel per ton pig..	111,180 = 40·768	104,336 = 28·50	91,194 = 22·50	85,912 = 19·69	88,577 = 19·99	79,958 = 19·49	79,056 = 21·06	61,365 = 15·44

Table I. contains consumptions of materials and generally the conditions under which the different furnaces, therein specified, were working.

The manner in which the heat was generated is given in Table II. at the end of the address, where also the calorific value of each unit of fuel is shown.

In Table I. is inserted the number of calories considered as absorbed by each item in the process. Different observers have given the results of their experiments undertaken with the view of obtaining the quantity of heat required for the purposes against which they are placed in the *Appropriation* account. The actual figures, being more or less an average, are as follows:—

	Calories.
Reduction of peroxide of iron.....	1,780
Dissociation of CO reckoned on C precipitated.....	2,400
Fusion of pig iron .....	330
Evaporation of water in coke.....	540
Decomposition of water, reckoned on H .....	34,000
Expulsion of CO <sub>2</sub> from limestone .....	370
Decomposition of this CO <sub>2</sub> in limestone reckoned on C present	3,200
Fusion of slag .....	330

Carried off in gases—estimated from weight, temperature, and specific heat.

Balance—to include loss by radiation, convection, &c.; by heat in tuyere water, &c.

The specific heat of the escaping gases is averaged in all cases at  $-24^{\circ}\text{C}$ .

Do. do. blast is taken at 237.

Heat evolved by the oxidation of carbon is taken at as follows:—

	Calories.
C to CO .....	2,400
C to CO <sub>2</sub> .....	8,000
C in CO to C as CO <sub>2</sub> .....	5,600

G represents a furnace working on English hæmatite ore, inserted as a comparative test of general correctness of mode in the other calculations.

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	A. Cold Blast.	B. Hot Blast.	C. Hot Blast.	D.
Height and cubic capacity of furnace . . .	48 ft. 6,000 c. ft.	48 ft. 6,000 c. ft.	80 ft. 15,500 c. ft.	90 ft. 33,488 c. ft.
Temperature of blast . . . . .	25° C.	485° C.	485° C.	710° C.
„ escaping gases . . . . .	430° C.	451° C.	310° C.	222° C.
Consumed per ton of pig iron : —	Cwt.	Cwt.	Cwt.	Cwt.
Coke . . . . .	40·76	28·50	22·50	19·69
Limestone . . . . .	18·25	16·00	11·00	10·50
Lime . . . . .	..	..	..	..
Ore . . . . .	46·20	48·00	48·00	48
Description of ore . . . . .	Clay ironstone.	Lias claystone.	Lias claystone.	Lias claystone.
	Cwt.	Cwt.	Cwt.	Cwt.
Weight of blast per ton of iron . . . . .	180·46	126·05	100·41	84·01
„ escaping gases per ton of iron . .	233·92	168·37	135·20	115·89
„ slag made per ton of iron . . . . .	34·15	31·00	22·00	28·00
Pig iron :				
Weekly make . . . . .	90 tons	220 tons	350 tons	700
Quality . . . . .	No. 3	No. 3	No. 3	No. 3
Make per 1,000 ft. cubic capacity . . .	15·0 tons	36·7 tons	22 tons	20·9
Appropriation of heat produced, viz. . . . .	111,180 cal.	104,336 cal.	91,194 cal.	85,912
Constants per ton of pig iron :—	Calories = Cwt. Coke.	Calories = Cwt. Coke.	Calories = Cwt. Coke.	Calories = Cwt. Coke.
Reduction of $\text{Fe}_2\text{O}_3$ in ore . . . . .	34,176 = 12·532	23,108 = 9·04	33,108 = 8·17	33,108 = 7·59
„ metalloids in pig . . . . .	1,600 = 0·587	4,174 = 1·14	4,174 = 1·03	4,174 = 0·96
Dissociation of CO . . . . .	1,440 = 0·528	1,440 = 0·40	1,440 = 0·35	1,440 = 0·33
Fusion of pig iron . . . . .	6,600 = 2·420	6,600 = 1·80	6,600 = 1·63	6,600 = 1·51
Sum of constants . . . . .	43,816 = 16·067	45,322 = 12·38	45,322 = 11·18	45,322 = 10·39
Variables, per ton of pig iron :—	Calories. Cwt.	Calories. Cwt.	Calories. Cwt.	Calories. Cwt.
Evaporation of water in coke . . . . .	124 = 0·045	400 = 0·11	313 = 0·08	275 = 0·06
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Reduction of this $\text{CO}_2$ to CO . . . . .	7,008 = 2·570	6,144 = 1·68	4,224 = 1·04	4,034 = 0·92
Fusion of slag . . . . .	18,782 = 6·887	17,050 = 4·66	15,400 = 3·81	15,400 = 3·53
Carried off in escaping gases . . . . .	24,148 = 8·855	18,224 = 4·99	10,058 = 2·49	6,174 = 1·41
Heat lost by radiation and expansion of blast, tuyere water, and balance to make both sides agree . . . . .	4,770 = 1·749	7,536 = 2·05	9,087 = 2·24	8,442 = 1·94
Sum of variables . . . . .	67,364 = 24·701	59,014 = 16·12	45,872 = 11·32	40,590 = 9·30
Total. Calories of cwt. fuel per ton pig . .	111,180 = 40·768	104,336 = 28·50	91,194 = 22·50	85,912 = 19·69

## THE SMELTING OF IRON ORES CHEMICALLY CONSIDERED.

BY

SIR LOWTHIAN BELL, BART., F.R.S.

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### INTRODUCTORY.

It is only natural, in a Society occupying itself with chemical industry, that the subjects referred to in a presidential address should be largely dealt with from a chemical point of view. Fifty years ago, when I was commencing my manufacturing career, the choice of subjects would have been comprised within a comparatively narrow compass, not so much from the smaller extent of this country's industrial pursuits, as from a supposed want of connection between some of them and the science of chemistry. The manufacture of iron was one of these. I recollect my disappointment in travelling among the furnaces and mills at home and abroad, to hear so little importance attached to the studies to which I had been applying myself for some time in Edinburgh and in Paris. Notwithstanding this indifference on the part of the ironmasters themselves, men of science, particularly in Germany and France, had devoted much attention to those branches of knowledge more directly bearing on metallurgical questions. Behind both these nations in that sphere of learning, which our Society and others are now doing their best to advance, were the iron manufacturers of our own country. It is a little remarkable, however, that during the half century we are considering, Great Britain in point of production and in manufacturing invention and proficiency, occupied the foremost rank as an iron-making country.

If we take an article like soda ash, an undoubted chemical product in the popular sense of the word, we find it may contain, besides sodium carbonate, 15 per cent. of foreign matter, without, for most purposes to which it is applied, the commercial value of its alkali being seriously impaired. If, however, pig iron has notably above one part of phosphorus in two thousand, 0.5 per cent., it would be rejected by the Bessemer steel maker as unsuitable for his purpose. Cases of this description, proving the extraordinary effects of minute quantities of foreign substances on the quality and properties of steel, sometimes in one direction, sometimes in another, might easily be multiplied. How to deal with these, or how to remove them, comes clearly within the range of chemical investigation, and entitles the subject of my address to a place among your transactions. So much for the metal when we have obtained it: but it possesses other claims on the attention of the Society of Chemical Industry, viz., in the phenomena which accompany its production.

Many ores of iron as they are delivered to the blast furnace contain 30 or 40 per cent. of earthy ingredients. To these have to be added the ash

contained in the fuel and the lime of the flux, employed for the fusion of the silica and alumina brought in by the other minerals. From this considerable weight of sterile matter pig iron is obtained by means of one of the most successful examples of heat utilisation with which we are acquainted. The crude metal, however, has not run the gauntlet, as it were, unhurt, through substances capable of materially affecting its properties.

To those influences it has been exposed, at an intense temperature, in some cases for 48 hours and upwards, and it is therefore not surprising that some carbon, silicon, sulphur, and phosphorus should be found as constituents of our pig iron. The metal thus contaminated is transferred, still liquid by the heat which has produced it, to the Bessemer converter. In it a very rapid current of highly compressed air oxidises the metalloids, or a greater part of them, with such an evolution of heat that the mass, 99 $\frac{2}{3}$  per cent. of which consists of pure iron, is raised to whiteness of dazzling splendour. By a small addition of carbon and manganese, in about a quarter of an hour from leaving the blast furnace, we may thus have 10 to 15 tons of cast iron changed into steel of great malleability and strength.

These considerations—but there are many others—justify me, I hope, in asking your assent to the proposition of the work of the blast furnace being one of the most interesting and successful applications of chemical science known in industrial enterprise.

This view of my subject was apparently shared by your Council, which, together with the fact of Nottingham occupying a central position of some very important seats of the iron trade, induced them to honour me with a request to prepare an address on a subject to which I had devoted a good deal of attention. Perhaps I may be here permitted to say that their wishes were willingly complied with by myself. The application of scientific investigation to an operation carried on in the manner and on the scale of a huge blast furnace, is beset with many disturbing causes and, in consequence, with many difficulties. For their satisfactory solution lengthened experience and frequent observation are necessary, and I gladly embraced an opportunity of submitting to so competent a tribunal as the members of this Society, my more recent views on the chemical questions involved in smelting the ores of iron.

#### RATIONALE OF THE SMELTING PROCESS.

So far as I am aware the action of the blast furnace continues to be thus described even in the latest work on chemistry.\* “A large number of investigations have been made on the chemical changes which occur in the blast furnace, but in spite of these our knowledge of this subject is still far from complete. The fuel uniting with the oxygen of the blast burns with formation, in the first place, of carbon dioxide, and this is reduced to carbon monoxide by contact with glowing carbon. This latter gas, coming in contact with the constantly descending charges of ore, reduces the ferric oxide to spongy metal. . . . The zone in which this reduction occurs is situated at a higher or lower part of the furnace according to the nature of the ores, and its temperature varies from 600—900° C. As the spongy iron descends it arrives at the hotter parts of the furnace. . . . At this point the finely divided spongy iron begins to take up carbon, and it may

\* “Treatise on Chemistry,” by Sir H. E. Roscoe, F.R.S., and C. Schorlemmer, F.R.S., 1889, Vol. II., p. 53.

therefore be termed the zone of carburisation." The zone in which fusion takes place is then described along with the reduction of certain other metals and metalloids. Allusion is subsequently made\* to experiments of Caillaetet, in which "it appears that even in the hottest parts of the furnace and at the point at which the combustion is most perfect, carbon exists in the solid state from the dissociation of carbon monoxide into carbon dioxide and carbon, brought about at very high temperatures."

My own experiments and investigations have led me to conclude that the action of the blast furnace is somewhat less simple than might be inferred from this description. That the greater portion of the reduction of the ore takes place in the upper zone is doubtless beyond all question. Nothing, however, is said of the important part played by a distance of 30 to 60 feet in the depth of the furnace in intercepting the heat which, but for it, would be wasted. To such an extent is this duty performed, that I have estimated in a modern furnace that for every unit of heat evolved at the tuyeres,  $2\frac{1}{2}$  units are returned to this point from the accumulation in the descending current of materials. It will also be shown that besides the deposition of carbon at high temperatures mentioned by Caillaetet, this action commences with the reduction of the ore, say, at  $150^{\circ}$  C. It will then be shown that if reduction be ever completed in the upper zone, which is more than doubtful, the spongy iron then produced would, during its passage through what I have designated a zone of heat interception, and in presence of an intensely deoxidising atmosphere, be reoxidised by the action causing the deposition of carbon. However this may be, there seem strong reasons for believing that as much as 25 per cent. of the oxygen required to form  $\text{Fe}_2\text{O}_3$  sometimes remains in the ore near the point where complete fusion takes place.

#### ON THE CHANGE EFFECTED BY REDUCTION OF THE ORE ON THE CO GENERATED IN THE BLAST FURNACE.

When carbon monoxide deprives an oxide of iron of all or any part of its oxygen, a corresponding portion is converted into carbon dioxide. Now, as the heat evolved by carbon passing into the state of the latter compound is more than three times as much as when the former oxide is generated (as 8,000 is to 2,400), it is obviously of very great importance that the proportion of carbon dioxide in the gases, as they leave the blast furnace, should be as large as the nature of the operation will permit.

As preliminaries to this inquiry, we have to consider the circumstances which conduce to the formation of carbon dioxide, and those which, when it is formed, tend to preserve its existence.

We do not require to be reminded that carbon dioxide, if produced at the tuyeres, cannot be long continued as such, and the same observation is applicable to the greater part of the height of the furnace. Indeed, experimentally I have proved, and the composition of the gases at its different levels shows, that where the carbon dioxide leaves the limestone, the temperature is such that this gas is resolved, by the incandescent coke, into carbon monoxide. Assuming each 20 parts of pig iron to contain 18.6 of iron, there will be liberated in the reduction of this quantity of the metal 7.97 parts of oxygen, equal to converting 5.97 parts of carbon in the form of carbon monoxide into carbon dioxide. Whatever the exact nature of the changes may be, my

\* Ibid., Vol. II., p. 54.

own experience, and that of all other observers whose work I have examined, have led me to infer that 5.97 cwt. of carbon as carbon dioxide per ton of iron made, is all we can calculate on meeting with in the gases escaping from furnaces engaged in smelting iron ores like those of Cleveland. It is perfectly true that carbon monoxide can be entirely converted into dioxide by contact with oxide of iron, but this happens under conditions, as to temperature, &c. which do not obtain in the case of the blast furnace. Hence we may infer that whenever 5.97 cwt. of carbon as carbon dioxide per ton of iron made is exceeded, it does not represent an *average* amount of the quantity we may expect to find in this form of combination.

#### ON THE QUANTITY OF C AS CO IN THE BLAST FURNACE.

If the correctness of this proposition be conceded, it follows as a necessary consequence that the remainder of the heat must be made up by the combustion of carbon to the condition of carbon monoxide, supposing the air driven into the furnace has the temperature of zero of the centigrade scale. What this quantity of heat may be depends on the nature of the ore under treatment, and the more or less perfect character of the furnace in which the smelting operation is conducted.

For the purpose of making this clear I have constructed two tables. The first table contains a statement of the weights of the various substances used and of those produced in the process, with how much heat, and from it what quantity of coke, has been consumed for each section of the duties it has to discharge. The factors made use of to estimate the quantity of heat are given in an explanatory note affixed to the table itself. In the second table, the quantity of heat evolved by the combustion of the coke in each case is set forth; and here it may be observed that all the calculations are based on the supposition that coke is the fuel employed. It is true, coal is frequently used in its raw or uncoked condition, but this means that the coking operation is effected in the blast furnace, and it is not until the volatile parts have been separated from the remaining portion that the actual work of the furnace begins. A few marginal words are added to table No. II. in respect to the method pursued in the calculations for ascertaining the production of heat. The difference between the two sides of the account, *i.e.*, the excess of heat generated beyond that absorbed, is considered as escaping through the sides of the furnace, by expansion of the blast and trifling errors in the figures themselves.

#### ON THE RELATIONS BETWEEN C AS CO AND C AS CO<sub>2</sub> IN A FURNACE 48 FEET IN HEIGHT WITH A CAPACITY OF 6,000 CUBIC FEET, BLOWN WITH AIR AT ATMOSPHERIC TEMPERATURE.

The work of what is technically known as a cold-blast furnace is set forth under columns A. in the two tables. The production is 90 tons per week with a consumption of 40.76 cwt. of coke, 46.20 cwt. of calcined ironstone, and 18.25 cwt. of limestone. The production of heat is 2,727 calories per unit of coke, and it was made up by each unit of carbon in the gases as CO<sub>2</sub>, being accompanied by 6.12 units as CO. The total quantity of heat so obtained was 111,180 calories, of which no less than 24,148 calories were carried off in the escaping gases, representing 8.85 cwt. or 21.7 per cent. of the heating power of the coke employed.



THE EFFECT OF HEATING THE BLAST TO  $485^{\circ}$  IN A FURNACE OF THE SAME DIMENSIONS AS THAT PRECEDING, VIZ., 6,000 CUBIC FEET.

Neilson, the inventor of the hot blast, was abundantly satisfied on obtaining a temperature of  $330^{\circ}$  C. in the air driven into the furnace. At the Clarence Works and elsewhere,  $485^{\circ}$  was obtained in the cast-iron stoves modified somewhat in construction from those in early use for the purpose. The effect of the change from cold to hot air of this temperature, is set forth in columns B. of the two tables. To secure this a quantity of fuel was burnt in the stoves, representing 35,000 to 40,000 calories, but of this, in the particular instance before us, only 14,488 calories reached the furnace, the remainder being lost by radiation from the stoves and at the chimneys, &c.

Notwithstanding this great escape of heat, 60 to 70 per cent. of the whole, it will be seen that by heating the air, a far greater reduction in the quantity of coke necessary for producing a ton of iron has ensued, than would at first sight have been expected. So much so, that it is a question whether Neilson's want of scientific knowledge on the subject was, at the time, not a matter of congratulation. Had he pursued his inquiries in this direction he would, no doubt, have been landed in the difficulties encountered by able chemists in their attempts to assign a cause for the great saving of fuel, and in consequence might have abandoned any practical work in connection with his valuable invention. Certain it is that the men of science, for nearly 40 years after its application, adopted a somewhat dangerous line of conduct in their attempts to explain the action of the hot blast by greatly over-estimating the actual saving it had effected. Dr. Percy, writing in 1864, quoted, and no doubt quoted truly, that after heating the air to  $300^{\circ}$  F. ( $149^{\circ}$  C.) the total consumption of coal per ton of iron at the Clyde Iron Works was reduced from  $161\frac{1}{4}$  cwt. to  $103\frac{1}{4}$  cwt. In this no doubt was included the great and needless waste in coking the coal, and many other matters having no connexion whatever with the actual work performed within the furnace itself. In reality therefore, the saving at Clyde did not much exceed one ton of coal, but this, or adopting  $12\frac{1}{4}$  cwt. of coke as given in the case before us, obtained by burning 4 or 5 cwt. of coal outside the furnace in the hot air stoves, affords sufficient ground for speculation and inquiry. I hope, however, to be able to convince this meeting that there is nothing mysterious in this apparent anomaly.

The first thing which strikes us on examining the figures is the large increase of heat rendered by a unit of coke, viz., 3,660 calories instead of 2,727, when cold blast was used, or an increase of 34 per cent. This difference is made up as follows: by a small accidental superiority in the coke; by a larger quantity of carbon raised to dioxide; and by the heat contained in the blast.

It will be readily understood that the vast volume of gases, largely expanded by the high temperature of the furnace, may arrive at the point of escape before they have parted with all the heat they are capable of communicating to the solid materials. In other words, there is an unnecessary waste of heat from this cause. When we come to examine the sources of heat in the hot-blast furnace it will be perceived that there is an increase of 409 calories, due to a larger quantity of carbon converted into carbon dioxide than in the cold-blast furnace. Any deficiency in this respect, when using cold air, may be due either to the velocity of the gaseous current not affording time for the conversion of the full quantity of carbon into dioxide, or from the

temperature of the upper region of the furnace being such, that carbon dioxide if formed, is reduced to the monoxide by incandescent coke. It is also possible, indeed probable, that the deficiency of carbon dioxide may be the result of a combination of these two reactions. Besides the improvement in the hot-blast furnace from a higher state of oxidation of the carbon there is an increase of 481 calories contained in the air. These amount together to 890 calories obtained without any addition to the volume of the gases. The effect of this change is that the quantity of gas for a given quantity of pig iron is about 40 per cent. larger in the cold- than in the hot-blast furnace. This diminution of volume will, however, not suffice for the object I have in view, because the hot-blast furnace produces 220 tons of metal while the other is only turning out 90 tons. The net result, therefore, is that in a given time, which means velocity, 75 per cent. more gas is passing through the hot-blast than through the cold-blast furnace.

It will probably occur to some that if there be any loss of heat from this increased velocity of the current of gas, the remedy would be to reduce the speed of the work performed, by regulating the admission of blast. Experience has taught the contrary, and it is conceived for the following reason. If we have a current of heated gas meeting and passing through one of cold solids, there is certain speed which affords the best results in heating the latter and cooling the former. Imagine a tall vessel filled with fragments of metal or stone, and, for ease of motion, we will suppose them to be spherical and of the size of playing marbles. Into the lower end of this vessel a current of hot air is introduced and, after a time, 20 of the marbles per minute are withdrawn, and are found to be very hot. In spite of this, the number is not sufficient to rob the air of all its heat, and in consequence we have the escaping air passing off from the top at a high temperature. To avoid this loss we commence to remove 200 of the marbles per minute, by which the loss of heat in the gases above is effectually checked, but the temperature of the solid spheres is insufficient for the purpose for which they are required. It is clear from this that there is a rate of speed expressed by something between 20 and 200, which, everything considered, is what has to be aimed at. This in the cold-blast furnace is achieved when 90 tons of iron per week are being run, say 15 tons per 1,000 ft. of furnace capacity, and in one driven with hot air a speed represented by 220 tons per week, or 37 tons per 1,000 cub. ft. per week, both making Cleveland iron, is the most successful. It is worthy of note also that the hotter the blast is the quicker the furnace can be driven, and the cooler are the escaping gases.

The saving of fuel in smelting iron ore with hot air is due to two causes. Each unit of fuel has, as we have seen at furnace B, its calorific power raised from 2,727 calories in the cold-blast to 3,660 calories in the hot-blast furnace, while the total quantity of heat required has fallen from 111,180 calories in A to 104,336 calories in B. Let us examine how this last difference of 6,844 calories has been brought about, and for this purpose our attention must be applied to the "Appropriation" of the heat produced in the manner already described. The appropriation referred to is given in two divisions, "Constants" and "Variables," so called because the former, for a given quality of iron in respect to the metalloids it contains, is supposed to call for the same quantity of heat; the latter varies with changes in the quantity of coke and limestone used, and with the percentage of earthy matters in the ore.

To the quantity of heat required for each item in the appropriation account is added its equivalent of coke, and although the actual number of calories

to satisfy the constants remain unaltered, their representation in coke is necessarily changed by the improved efficiency of the latter. In this way an economy by the hot blast of 3.687 cwt. of coke per ton of iron has been effected upon 45,322 calories engaged. In the variables 59,014 calories were absorbed, and approximately it will be found that of the saving of 8.581 cwt. of coke under this head, 5.161 cwt. may be set down to the enhanced power of the coke, and the remaining 3.52 cwt. to the diminished amount of work to be performed. As regards the cause of this diminution, using less coke gives less water to be evaporated, and less atmospheric moisture to be decomposed. A reduced quantity of coke ash lessens the limestone for fluxing, which in its turn needs less heat for the expulsion and dissociation of its carbon dioxide, along with a smaller weight of slag to be melted. Economy in fuel and in limestone tells greatly on the volume of escaping gases, thus effecting a saving of 5.924 cwt. of coke, in furnace B, under this item alone.

#### ON THE ADVANTAGES DERIVED FROM INCREASING THE HEIGHT OF THE BLAST FURNACE.

With the knowledge afforded by the previous examination of the action of the hot blast, it may be said that heating the air is equivalent to an increase in the dimensions of the furnace, *i.e.*, not only is there a great economy in the consumption of coke, but, as we have seen, 1,000 ft. of furnace space affords 37 tons of iron per week instead of 15 tons, as was the case when cold air was used. The natural sequence of this admission would be the probability of some advantage being derived from an enlargement of the furnace as it existed 28 years ago. This brings us back to the time when John Vaughan built a furnace 71 ft. in height, not however from any inference he drew from the reasoning just adduced, because his mind in reference to the chemical action of the blast furnace was very similar to that of Neilson when he proposed heating the blast. Vaughan's idea was to increase the make of the furnace, and in this practical view of the question this very practical man was not disappointed; but his enlarged furnace did more than this, it saved a notable quantity of coke, something like 4 cwt. per ton of iron, if my memory is correct. This height was increased at the Clarence Works and elsewhere to 80 ft. with a cubic capacity varying from 12,000 to 25,500 cub. ft. This advantage, however, has only been obtained by a sacrifice in the output, because in smelting Cleveland stone the production does not exceed 22 tons per 1,000 cubic ft. of capacity, using air heated to 485°. This is precisely the same temperature the blast had in the 48 ft. furnace, and by referring to the figures contained in column C. in the two tables the effect of the additional furnace space will be readily appreciated. The limestone consumed and the weight of escaping gases are considerably reduced, while the efficiency of the coke is considerably increased by the larger quantity of carbon as dioxide per ton of iron. The consequence of this is that a unit of coke now represents 4,053 calories instead of 3,660, as happened in the smaller furnace. This shows an improvement of about 11 per cent., while the requirements have, for the reasons given above, fallen from 104,336 to 91,194, equal to 13 per cent. The net result is that, instead of 28.5 cwt. of coke being burnt per ton of iron, the quantity fell to 22.5 cwt.

In confirmation of what has been advanced respecting the similarity of effect in heating the blast or increasing the dimensions of the furnace, there is placed in adjoining columns the results obtained in the cases A and B in

the tables. To these are added the particulars of a much loftier furnace than that given under B, but, like A, blown with cold air:—

	A.	B.	—
Height in feet .....	48	48	70
Cubic capacity .....	6,000	6,000	7,500
Blast .....	Cold	485°	Cold
Calined ironstone used per ton of iron ..... Cwt.	46·20	48·00	50·00
Limestone used per ton of iron ..... ..	18·25	16·00	13·00
Coke used per ton of iron ..... ..	40·76	28·50	32·00
Weekly make per 1,000 cubic feet of furnace capacity ... Tons	15·0	36·7	19·3

Thus, by a mere alteration in dimensions, a saving has been obtained of 8·76 cwt. of coke per ton of iron, and the produce per 1,000 cubic feet of capacity approximates to that obtained from a modern Cleveland furnace. In all probability, had the height been increased to 80 feet, the consumption of coke would have been as favourable as that given under column B.

After what has been said in explanation of the items of saving realised under columns B., it cannot be necessary to explain in detail the advantages due to the enlargement of the furnace C as compared with B. These will be readily understood by a comparison of the figures in the tables.

The benefit derived from the addition of 32 ft. to the height of a furnace 48 ft. high having been settled beyond all questioning, an ironmaster, who had built four 80 ft. furnaces, named to me his intention of erecting a pair 100 ft. high. This led me to reflect on the nature of the action in the upper zone. A reference to the figures under column C. will indicate that of the total heat (79,656 calories) generated by the combustion of the carbon 33,432 calories, or 42 per cent., are due to the burning of C as CO to CO<sub>2</sub>. It has further to be remarked that this oxidation, or combustion, begins almost immediately on the introduction of the ore, and is completed, as far as it can be, in the first 15 ft. from the upper surface of the materials in an 80 ft. furnace. Practically, however, it may be said, very little of this heat will manifest itself in the sensible form in the gases, because a large proportion of it (the greater part of the 33,108 calories) is absorbed in tearing away the oxygen from the iron in the ore. I proved experimentally, however, that by arresting the office of reduction in the upper zone there was a marked decline in the temperature of the gases. This was done by withdrawing the ore from the charges and substituting a mixture of slag and flints in its room. In a short time the temperature of the escaping gases began to fall, and when this was stationary the new mixture was discontinued and the ore resumed. In due course the gases became hotter, and speedily regained their normal temperature. The difference was estimated at 220° C., and taking the gases at 137 cwt. per ton of iron, this would represent 7,138 calories, or nearly 8 per cent. of the entire heat generated in such a case as the example given under column C.

The inference I drew from this experiment was that any addition to the height of a furnace after a certain altitude would prove useless, and of this I

warned the owner, who, however, disregarded my advice. He built his two furnaces 103½ ft. high, and to my surprise reported he was making a ton of iron with 18 cwt. of coke. This was a saving of about 4 cwt., which was 30 per cent. more than the entire loss of heat in the waste gases in an 80 ft. furnace, working in the manner set forth in column C.

My friend, acting in perfect good faith, willingly allowed me to examine the condition of these two monster furnaces, and to compare them with other furnaces at his works 80 ft. in height, and using the same minerals. The following were the results of this examination:—

The temperature of the escaping gases between the two types of furnace only differed by 7° C.

The quantity of carbon in the gases, as judged by its relation to the nitrogen present, was precisely the same in both.

Samples of the gases were taken over 2½ to 3 hours. In the 80 ft. furnace the carbon as CO<sub>2</sub> was as 1 to 2·87 as CO, and in the 103½ ft. furnace it was as 1 to 2·86.

From this I concluded that it was a mistake to imagine that there was any such difference in the consumption of coke as had been supposed, an opinion which was confirmed by subsequent events. It is perhaps only right to observe that the loftier furnaces were being driven below the speed I should have thought best adapted for economy of coke, that is, instead of running 22 tons per week per 1,000 cub. ft. of capacity, they were only giving 18 tons.

We may therefore infer, that raising a furnace beyond a certain height, such height varying according as the iron oxide parts more or less easily with its oxygen, is useless. The reason is that the zone of reduction, and therefore of heat generation, has its position raised with each needless increase in the altitude of the structure, whereby the temperature of the gases remains unchanged, and the loss from this cause continues unaltered.

Before leaving the question of capacity of furnace I would say that, while there may be some advantages connected with colossal furnaces, I greatly doubt whether economy of fuel can be justly reckoned among them. In my opinion a height of 80 ft., and a capacity of 16,000 cub. ft., suffice for all we can expect in this direction, when smelting the ore of Cleveland.

#### ON RECENT INCREASES IN THE TEMPERATURE OF THE BLAST.

When it was ascertained at the Clarence Works that air at a temperature of 535° C. had enabled us to produce a ton of iron in our 48 furnaces with about 25 cwt. of coke, experiments were set on foot to introduce the regenerative system, as it had been named by Siemens, for heating the blast. This was done in the hope that an amount of heat could be introduced into the furnace beyond the power of iron pipes to supply it. Mr. E. A. Cowper, largely assisted by Mr. Charles Cochrane had, I afterwards learnt, made considerable progress in the same direction previous to my trials, and to these gentlemen we are indebted for the successful introduction of what is now well known as the "brick stoves."

Impressed as I had been and still was with the desirability of an alteration in the construction of the hot blast stoves, I never could see my way to adopt the opinions entertained by some of my colleagues in the trade, as to what was capable of accomplishment by this new form of apparatus. From 13 to 16 cwt. of coke of average Durham quality was not infrequently spoken

ot as sufficing to produce a ton of Cleveland iron, my own expectations being limited to about 20 cwt.

The facts and figures which have been set forth in these pages render it easy to judge of the difficulties in reducing the coke consumed below a certain point. For the purpose of illustrating this I have followed the same mode of calculation as that adopted in the tables, on the supposition that 18 cwt. per ton of metal was the weight consumed. With the reduction in the quantity of coke, 9 cwt. of limestone instead of  $10\frac{1}{2}$  and 11 cwt. have been assumed as the quantity to be used. It is not necessary that more should be done than remind the members of the changes consequent upon using a smaller quantity of coke and flux. What does concern us is, that the 18 cwt. of coke producing the theoretical quantity of  $5.97$  cwt. of carbon in the form of dioxide, and the remainder as monoxide, will give 1 of C as  $\text{CO}_2$  to  $1.81$  as  $\text{CO}$ , in respect to the possibility of which some doubt may be entertained in smelting Cleveland ore. One thing, however, is certain, the blast would be considerably lower in quantity than anything hitherto reached in Cleveland under similar circumstances. The weight would be only  $77.97$  cwt. per ton of metal, and to supply the complement of heat required, 16,900 calories would be needed in the blast, equal, therefore, to a temperature of  $914^\circ \text{C}$ . This, I take it, would be extremely difficult with the present hot-air apparatus, even of brick, to secure. To show how rapidly this obstacle increases with each diminution in the coke, I have estimated that for 16 cwt. of coke to the ton of iron, which has been named as possible, the blast would weigh only  $67.72$  cwt., and that then a temperature of  $1,605^\circ \text{C}$ . in it would have to be secured.

#### ECONOMY OF FUEL IN FURNACES OF MOST MODERN CONSTRUCTION.

We may now abandon mere speculation as to what may be done in further economy of fuel in smelting ore of the Cleveland character, and confine ourselves to what has been accomplished. Column D. affords such an example when in a furnace 90 ft. in height, and a capacity of 33,488 cub. ft., a ton of iron was said to have been produced with  $19.69$  cwt. of coke. It may further be remarked that the analysis of the gases, made by the Clarence chemist Mr. Rochall, confirmed this statement. The temperature of the blast was ascertained to be  $710^\circ$ , but it does not appear, for a complete understanding of the workings of this furnace, more need be said than a reference to the particulars as they are to be found in the two columns D. in Tables I. and II.

The column E. is one of the Clarence furnaces, 80 ft., with a capacity just about half that of D., 15,500 cub. ft. The coke consumed is a little higher than under D. ( $.30$  cwt.), but it has to be observed that when working with such high loads of mineral upon the coke, we may expect some trifling fluctuations in respect to the fuel consumed. Still, I am not prepared positively to deny that the extra dimensions of D. may, at such high blast temperatures, have a trifling advantage. At the same time, this is doubtful.

#### QUANTITY OF FUEL REQUIRED IN CONNECTION WITH THE USE OF LIMESTONE OR LIME.

In directing our attention to the manner in which the heat in the blast furnace is disposed of, we cannot fail to be struck with the share rendered

necessary by the presence of the carbon dioxide liberated from the limestone. This is particularly evident in the old type of furnace as set forth in columns A. and B. of the tables. There is the loss of carbon of the coke due to the action upon it of the carbon dioxide set free, and also the heat required to expel and decompose this gas.

The sum of the items connected with the use of limestone amounts in the cold blast furnace A. to 5·046 cwt. of coke, and in B., blown with hot air, to 3·29 cwt. This is exclusive of a loss of carbon carried off by the liberated dioxide from the flux. It is to be feared that in olden time we did not realise the full extent of the evil, but knowing generally that heat was necessary to drive off the CO<sub>2</sub> in a limekiln, it was assumed that a saving might be effected by previous calcination of the flux, without much or indeed any calculation what that saving might amount to. It happened in the old type of furnace—say, 48 ft. in height—that the heat carried off in the gases represented 4·99 cwt. of coke in the hot-blast furnace and 8·85 cwt. in that blown with cold air. There was, therefore, heat enough and to spare with such a loss at the throat to effect at all events some economy. In a paper read before the Iron and Steel Institute in 1875, I showed that the saving was under 1½ cwt. of coke, and it was then proved that a portion of the difference between practice and theory, *i.e.*, imperfect theory, arose from the imperfect mode of burning the lime, by which a considerable portion of the carbon dioxide was still left in the flux. We shall see when we come to consider the action of a furnace using well calcined lime to what this difference between calculation and experience was owing. Still 1½ cwt. of coke was worth something, at the same time it must be remembered that this saving was only effected by burning probably double this weight of coal in the limekiln, probably nearer 4 cwt. than 3. But paying 3s. or 4s for small coal and 10s for coke left a margin, and therefore the plan was not infrequently adopted. When the large furnaces came into use it was perhaps to be expected, if for no other use than for information's sake, that burnt lime should be tried in them. At the same time, as the heat carried away in the gases was only about half its former quantity, there was no longer the margin out of which the additional work could be performed, *i.e.*, instead of the heating power of 4·99 cwt. of coke being in the escaping gases as happened in a 48 ft. furnace blown with air at 485° C., there was only that of 2·49 cwt. in the gases of an 80 ft. furnace with blast at the same temperature. The trials at the Clarence Works, of which full particulars were published at the time, gave such results as led to their abandonment by my firm, which was followed by my late friend Edward William, who confirmed the conclusions laid down in the discussion which followed the reading of the paper on the subject.

A dozen years afterwards the use of caustic lime again found favour in Cleveland, but it speedily disappeared. This was admitted by its former advocates upon a recent occasion, when entire success attending its application was alleged to have been achieved at a Middlesbrough furnace. I will shortly give the reasons for my believing that the results then communicated were not conclusive of any economy having been realised in this new trial.

The experiment was made in a furnace 76 ft. high, with a capacity of 20,454 cub. ft. When working upon raw limestone, it was alleged to have consumed 23·28 cwt. of coke per ton of metal, and on lime, 19·49 cwt., the saving thus stated being 3·79 cwt. The comparison involves, in my judgment, a fallacy at the outset, for such a furnace, using limestone, ought, with its blast heated to 765°, to have produced a ton of iron with less than 21½ cwt. of coke. The saving then, in reality, was less than 2 cwt., instead



of 3·79 cwt. So far back as 1875, I not only showed that in 80 ft. furnaces there was no economy in using caustic lime, but the reasons of its failure in this respect were described.

Caustic lime, as is well known, on exposure at ordinary temperatures, attracts  $\text{CO}_2$  from the mere trace contained in the atmosphere. Of course, this absorption is much more rapid in gases containing, like those escaping from a blast furnace, about 12 per cent. of their volume of this gas, and the rate of absorption is further greatly accelerated at higher temperatures, but short of what is required to convert limestone into lime. What takes place then in the upper region of the blast furnace, when caustic lime is used, is a re-absorption by the lime of the carbon dioxide produced by the reduction of the ore. The calcium carbonate thus formed, on reaching the hotter zones of the furnace, loses its  $\text{CO}_2$ , and an action is set up precisely the same as if limestone had been used, so far as the dissociation of carbon dioxide is concerned. The figures given in the paper descriptive of these fresh trials, however, indicate the decomposition of a larger quantity of this higher oxide of carbon than when limestone in its native condition is employed. This, in all probability, is increased by an undoubted generation of heat produced in the reducing zone by the union of carbon dioxide with the lime. However this may be, there is the fact of the particular furnace in which the trial was made containing per ton of iron only 4·33 cwt. of carbon as dioxide in the gases, instead of a quantity approaching 6 cwt. A consequence of this disturbance, in spite of a very high temperature of blast, is a great falling off in calorific power of the coke. I give the results of columns E. and F. to illustrate the nature of this decline of power:—

Sources of Heat per Unit of Coke.	E. Clarence Furnace of 80 ft., 15,500 cub. ft., Blast 704, using 19·39 Cwt. of Coke per Ton of Iron.	F. Furnace, 70 ft. High, 20,451 cub. ft., Blast 765, and using 19·49 Cwt. of Coke per Ton of Iron.
Conversion of C into CO .....	2,032	2,120
Conversion of portion of this C as CO into $\text{CO}_2$ .....	1,672	1,244
Heat in blast for each unit coke.....°.....	727	738
	4,431	4,102
Weight of C as CO to each unit of C as $\text{CO}_2$ in the gases .....	} 2·18	2·94

It happens that the Cleveland stone contains usually such a quantity of lime that something like 3 cwt. of this earth, from this cause, finds its way into the furnace for each ton of iron produced. It appears, however, that the lime is in such a state of combination as not to be affected by  $\text{CO}_2$ . This was proved experimentally when a mere trace of the carbon dioxide was absorbed by the calcined ore.

#### GENERAL CONCLUSIONS.

In answer to those who, in order to effect further economy in the consumption of coke in the blast furnace, recommend the use of furnaces of

great height and large dimensions, who advocate the use of caustic lime, and insist on imparting unmanageable heat to the blast, I would beg attention to the lessons taught by the example given under column E. There, a furnace of 15,500 cub. ft. capacity, blown with air at a comparatively moderate temperature, and using raw limestone, compares favourably with furnaces twice its size with a much hotter blast and deriving such advantages as calcined limestone is regarded as being capable of affording.

I am very far from undervaluing the very great importance of the lessons taught by the various trials made with the view of reducing the cost of producing pig iron. There is no doubt the iron trade is under very great obligations to those who, at considerable expense and trouble to themselves, have given us the benefit of their experience, without which almost all speculation is useless.

#### EXPERIMENTAL WORK, AND INFERENCES DRAWN THEREFROM.

When the feasibility of heating the air to  $750^{\circ}$  had been demonstrated, and the great expectations as to its future influence on the iron trade was dwelt upon, I ventured to urge the difficulty just referred to: the difficulty being a rapidly-increasing temperature in the blast chiefly arising from a reduced volume of air having to carry a larger quantity of heat. There were, however, other questions, but of a purely chemical character, which it seemed to me might impose a limit to the economy expected from the use of a more intensely heated blast than had hitherto been in use. This limit I conceived might be established from the known oxidising power of carbon dioxide on metallic iron, particularly when the latter is in the spongy state. This evidently, under certain conditions as to temperature, could not fail to weaken if not to arrest the reducing power of the carbon monoxide. There was also the faculty possessed by heated carbon to decompose the dioxide generated by the reduction of the oxide of iron. The full force of this reflex action is easy to perceive when it is remembered that for every unit of carbon so treated the heat of  $2.33$  units in the form of monoxide is required to take its place.

I propose now to give a brief abstract of some experiments upon which the conclusions set forth in these pages have been founded.

It was ascertained that at  $200^{\circ}$  C., the ferric oxide in Cleveland calcined ironstone begins to lose its oxygen, but the action is faint; at  $210^{\circ}$  it became marked, and at  $225^{\circ}$  it was rapid.

The following shows the starting points of the action of reduction on different varieties of oxide:—

—	Lowest Temperature at which $\text{CO}_2$ appeared.	Temperature of Marked Action.
	$^{\circ}$ C.	$^{\circ}$ C.
Pure precipitated $\text{Fe}_2\text{O}_3$ .....	141	149
$\text{Fe}_2\text{O}_3$ obtained by calcining $\text{FeNO}_3$ .....	145	154
Calcined Cleveland ore ( $\text{Fe}_2\text{O}_3$ ) .....	199	210
$\text{Fe}_2\text{O}_3$ obtained by calcining $\text{FeSO}_4$ .....	208	216

The percentage of oxygen lost in seven hours by specimens simultaneously heated in a vessel to about  $417^{\circ}$  C. and exposed to a current of CO :—

	Original Oxygen Removed, Per Cent.
Calcined Cleveland ore.....	9.4
Fe <sub>2</sub> O <sub>3</sub> got from calcined iron sulphate.....	61.7
Fe <sub>2</sub> O <sub>3</sub> precipitate.....	66.7
Fe <sub>2</sub> O <sub>3</sub> from calcined iron nitrate.....	72.7

Exposure, in a similar vessel, to CO at a temperature of  $421^{\circ}$  gave the following results :—

	Percentage of Iron.	Original Oxygen Removed, Per Cent.
Calcined spathose ore.....	51.7	15.4
Elba specular ore.....	66.8	16.8
Lancashire hematite.....	66.6	35.9
Calcined Cleveland ore.....	40.0	37.3
Pure precipitated Fe <sub>2</sub> O <sub>3</sub> .....	70.0	49.2
Fe <sub>2</sub> O <sub>3</sub> from calcined FeSO <sub>4</sub> .....	70.0	60.8

By slowly passing CO over an excess of Cleveland calcined ore, the gas was converted into CO<sub>2</sub>, but of the oxide of iron only 5.25 of its oxygen was removed.

At a temperature of  $300^{\circ}$  pure carbon dioxide was passed over pure spongy iron without change, but at the melting point of zinc ( $417^{\circ}$ ) the action was very rapid. The metal was oxidised and the gas was almost entirely converted into carbon monoxide.

Hard Durham coke, silvery in appearance, is only slowly affected by carbon dioxide at a bright red heat, but at the temperature near the tuyeres of a blast furnace the change may be regarded as instantaneous. On the other hand, soft dull-looking coke is affected by carbon dioxide in the much lower temperature of the reducing zone. The usual proportion of C as CO<sub>2</sub> to C as CO is as 1 to 2.6 in an 80 ft. furnace receiving its blast at  $485^{\circ}$ . Under the same circumstances, but using soft coke, it became as 1 to 2.82.

A mixture of 2 vols. of CO and 1 of CO<sub>2</sub> (1 C as CO<sub>2</sub> to 2 C as CO by weight) and another of 3 vols. of CO to 1 of CO<sub>2</sub>, were passed through a vessel containing the following substances, the temperature being  $417^{\circ}$  C. :—

	LOSS PER CENT. OF ORIGINAL OXYGEN.			
	2 Vols. CO, 1 Vol. CO <sub>2</sub> .	2 Vols. CO, 1 Vol. CO <sub>2</sub> .	3 Vols. CO, 1 Vol. CO <sub>2</sub> .	3 Vols. CO, 1 Vol. CO <sub>2</sub> .
	5½ Hours Exposure.	11½ Hours Exposure.	5 Hours Exposure.	10½ Hours Exposure.
Calcined Cleveland ore .... Loss of O	Per Cent. 0.9	Per Cent. 4.3	Per Cent. 1.9	Per Cent. 29.3
Fe <sub>2</sub> O <sub>3</sub> from pure nitrate ... ..	2.4	16.2	..	..
Precipitated Fe <sub>2</sub> O <sub>3</sub> .....	2.9	14.0	26.6	38.6
Fe <sub>2</sub> O <sub>3</sub> from calcined FeSO <sub>4</sub> . ..	3.4	..	..	..

Recently the following experiments were made. A mixture of 1 vol.  $\text{CO}_2$  and 2 vols. CO were passed for 40 hours over calcined Cleveland ore; the temperature varying from  $400^\circ$ — $480^\circ$ , the average being  $441^\circ$ :—

—	Composition before Exposure.	No. 1 Composition after Exposure.	No. 2 Composition after Exposure.
Iron .....	41.44	43.62	43.20
Oxygen .....	17.71	11.69	13.22
Carbon deposited.....	Nil.	1.70	1.00
Earthy matter.....	40.85	42.99	42.58
	100.00	100.00	100.00

In No. 1 the loss of original oxygen was 33.99 per cent. in 40 hours; and in No. 2, 25.35 per cent. in 15 hours, the temperature in the latter varying from  $430^\circ$  to  $470^\circ$ . In it 2 grms. were exposed to a current of the mixed gases, continued for 5 hours, during which 550 cc. were passed over. This was done three times upon the same specimen of ore, the loss of oxygen being as follows:—

	Grms.
After the first exposure .....	.0338
After the second exposure.....	.0046
After the third exposure .....	.0001
Total loss .....	.0385

A mixture of 36 vols. of  $\text{CO}_2$  and 64 of CO was passed over spongy iron for  $13\frac{1}{2}$  hours, the temperature being one of bright redness. In about six hours the composition of the gas was 31 vols. of  $\text{CO}_2$  and 69 vols of CO (1 of C as  $\text{CO}_2$  to 2.22 of C as CO by weight), after which no further change occurred.

The same experiment was tried at a white heat, the mixture at the commencement being 36 vols. of  $\text{CO}_2$  and 64 of CO. In two hours the composition was 10 of  $\text{CO}_2$  and 90 of CO, or 1 to 9 of C by weight in the two gases.

A large number of experiments of a similar character were performed in the laboratory, all of course tending to show the antagonistic tendencies of the two gaseous oxides of carbon and the conditions which increased or diminished the activity of each. From this I inferred that in actual practice, when smelting Cleveland pig iron in the most perfect furnaces we possess, the neutral point was being approached. This opinion received confirmation from the fact that on exposing Cleveland calcined ironstone as a coarse powder to the action of the escaping gases, with their composition of, say, 1 of C as  $\text{CO}_2$  and 2.20 as CO, and having a temperature of about  $400^\circ$ , we were approaching the limit to which carbon monoxide could deprive such an ore of its oxygen. By this is meant that the action should proceed with sufficient rapidity for reduction to be largely effected before the ore passed into a zone of the furnace, where from the elevation of temperature the carbon dioxide generated would resolve into CO ( $\text{CO}_2 + \text{C} = 2 \text{CO}$ ).

When a mixture of equal volumes of CO and  $\text{CO}_2$  was passed at a bright red heat over factitious  $\text{Fe}_2\text{O}_3$ , calcined Cleveland ore, Lancashire hæmatite, calcined spathose ore, and pure spongy iron, all the ferric oxides were reduced to ferrous oxide, at which they remained stationary, while the spongy iron was

oxidised to the extent of becoming also FeO, after which no further change took place.

The following trials were made with the CO<sub>2</sub> in excess, and in which Cleveland calcined ore was exposed under the conditions named. The last specimen was Cleveland ore in which the iron had been completely reduced by H.

CO.	CO <sub>2</sub> .	Temperature.	Loss of original O.	Period of Exposure.
Vol. 1	Vols. 1.25	420° C.	Per Cent. 5.25	Hours. 1 $\frac{3}{4}$
1	2.2	Visibly red.	11.70	1 $\frac{1}{2}$
1	4.0	Full red.	15.70	1 $\frac{1}{2}$
1	6.0	Low red.	5.80	$\frac{1}{2}$
1	6.0	Red heat.	6.00	$\frac{1}{2}$
1	6.0	Bright red.	87.00 of O = in Fe <sub>2</sub> O <sub>3</sub> , absorbed.	$\frac{3}{4}$

At a temperature of 417° C., CO<sub>2</sub> oxidises spongy iron, but in a mixture of equal vols. of CO and CO<sub>2</sub> this action is entirely arrested. At a red heat, however, the metal was rapidly oxidised.

From what has preceded it may be inferred that a position of stable equilibrium exists in which the oxide of iron ceases to lose oxygen, and the mixture of the two gases remains unchanged, the point of stability being affected by the temperature.

When trials were made at the blast furnace, the following information was obtained in experimenting with a furnace having a capacity of 25,500 cub. ft. The average temperature of the escaping gases was 312°. The quantity of CO<sub>2</sub> varied from 34 vols. to 45 vols. per 100 of CO, the average of 13 trials being about 37  $\frac{1}{2}$ . The following figures show the percentage of original oxygen removed:—

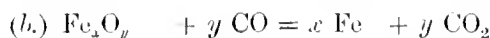
In 1 Hour.	In 2 Hours.	In 24 Hours.	In 48 Hours.
5.31	6.19	3.72	5.71

#### CHEMISTRY OF THE REDUCTION OF OXIDE OF IRON IN THE BLAST FURNACE.

It is now time that we should pass on to the concluding remarks I have to offer in reference to the action of the blast furnace. Reference was made in my opening remarks to the fact of this not being as simple in its character as has been supposed. Had the old views of the reduction of peroxide of iron to the state of metal been correct it would have been expressed by the following equations:—We must suppose Fe<sub>2</sub>O<sub>3</sub> by the action of CO brought down to a state of oxidation expressed by Fe<sub>x</sub>O<sub>y+1</sub>, when it encounters a fresh portion of CO, which will be designated in quantity as *y* CO. We have thus—



and then

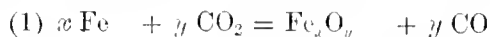


Of course, for reasons already stated, there is and must be an excess of CO present, it being considered impossible to convert all the CO into CO<sub>2</sub>. In or near the hearth, as formerly mentioned, the atmospheric moisture, silicon, phosphoric, and sulphuric acids part with their oxygen, the metal absorbing their bases along with a portion of carbon to form pig iron.

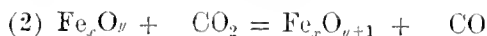
It had been observed by Ebelmen and others, and largely commented on by Percy, that there appeared an excess of oxygen at the tuyeres, *i.e.*, there was more than the quantity corresponding with the nitrogen and moisture of the atmospheric air which entered at the tuyeres. This observation has since been confirmed by my own observations and those of others. So far, however, as I have been able to discover, an accompanying increase of carbon in the gases had altogether escaped notice. Without any knowledge of what had been done by Schinz and others in connexion with the splitting up, in presence of iron and its oxides, of carbon monoxide into carbon and carbon dioxide, I became aware of the fact that in exposing iron ore to the escaping gases of a blast furnace, there was a copious deposition of a black substance which became as it were infiltrated in the ore, and which was found to be carbon. This discovery was followed up at a very great length of laboratory examination of the phenomena connected with this reaction, and in this I had the assistance of Dr. C. R. Alder Wright. These have been described elsewhere.\* I shall in consequence content myself with a simple enumeration of the reactions themselves of CO<sub>2</sub> and CO on metallic iron and its oxides:—

#### ACTION OF CO<sub>2</sub> ON SPONGY IRON.

This is the converse of that set forth in the previous equations, the metal being reoxidised with the reduction of the CO<sub>2</sub> to CO. The two equations are thus expressed:—



and then

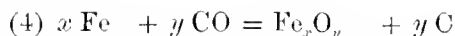


#### ACTION OF CO ON SPONGY IRON OR PARTIALLY REDUCED OXIDE OF IRON.

When the metal in the conditions named, is exposed to a current of CO at certain temperatures, pure C is precipitated with absorption of some O by the metal or its sub-oxide:—



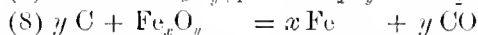
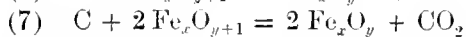
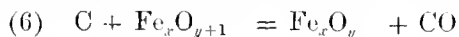
or



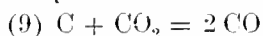
At other temperatures the additional oxygen thus taken up is removed and, as regards the carbon monoxide, the equations *a* and *b* express the change, *viz.*:—



It may also happen that at higher temperatures the liberated carbon may completely reduce the iron oxide as follows:—



At last, when the heat is raised, the carbon liberated may be dissolved by CO<sub>2</sub> formed in some of the previous reactions, and we get:—



\* Chemical Phenomena of Iron Smelting and Principles of the Manufacture of Iron and Steel.

It seems highly probable that the changes set forth in the preceding equations afford an explanation to the phenomena which excited so much surprise in the mind of my late friend Dr. Percy. The two following sets of analyses exhibit the composition of the gases of an 80 ft. furnace taken at different levels. The observations contained in No. 1 were made when raw limestone was being used, whereas in No. 2 the flux had been previously calcined; hence the difference in the quantities of O and C throughout. The figures given express cwt. per ton of iron produced:—

—	Temperature.	Oxygen, No. 1 Carbon.		Oxygen, No. 2 Carbon.	
Escape pipe .....	250° C.	33·40	20·96	29·00	20·28
Depth below surface of minerals 8½ feet	450° C.	26·18	18·52	25·00	17·23
„ „ 18 „	780°	24·12	18·14	24·70	17·32
„ „ 31 „	Bright red.	26·24	18·36	24·65	17·42
„ „ 41½ „	Very bright red, gradually increasing to white heat.	25·26	18·92	24·72	18·08
„ „ 57 „		25·16	18·62	24·14	17·98
„ „ 62½ „		25·01	18·78	24·74	17·80
Four feet above floor of hearth 68 „		27·56	20·26	26·96	19·34

After making allowance for the oxygen separated from the metalloids and atmospheric moisture, there remains about 2·04 cwt. of this gas for every ton of iron made. At the same time there is an increase between the same points (62½ ft. level and 68 ft. level) of about 1·65 cwt. of carbon.

Other examples gave per ton of iron:—

Increase at tuyeres of O cwt.....	1·45 — 1·81 — 2·22
„ „ C „ .....	1·55 — 2·68 — 1·91

The differences in oxygen were thus estimated:—

Quantity actually found in gases in first case given.....	Cwt. 27·56
Less corresponding to nitrogen present .....	22·65
Present in $P_2O_5$ ·39— $SO_3$ ·55— $SiO_2$ ·55 reduced ....	1·49
Separated from lime to form CaS in slag.....	·72
From moisture in blast.....	·66
	25·52
	<u>2·04</u>

As regards Cailletet's experiment quoted by Roscoe and Schorlemmer, from which the production of solid carbon in the hottest part of the furnace is inferred, this phenomenon was ascribed by the first named chemist, and confirmed by St. Claire Deville, as being due to the dissociation of carbon monoxide by the intense temperature in the crucible.

One of the analyses cited by Cailletet in support of this view will be made use of for the purpose of examination. The sample of gas was gathered from a furnace in which charcoal was used, and consisted, by weight, of:—

Free Oxygen.	Hydrogen.	Carbon Monoxide.	Carbon Dioxide.	Nitrogen.
17·03	0·12	2·08	4·61	76·16 = 100



The total oxygen, free and combined, almost exactly corresponds with the nitrogen, as the two gases exist in atmospheric air. Of carbon in the gaseous condition, there was only about one-tenth of the usual quantity found in the gases taken from the crucible of a blast furnace. Such carbon as Cailletet collected, lined the pipe used for withdrawing the gases for analysis. If the explanation of the French chemists be correct, the entire specimen of the gas, of which the composition is given above, must have had nine-tenths of its carbon precipitated from the carbon oxides, while being maintained at the necessary temperature by the combustion of the adjacent fuel. This is inferred from the fact that, in the specimen itself, the unburning, as it were, of 90 per cent. of the carbon it contained must have been accompanied by a great cooling of the gases immediately concerned in the change. There are, it seems to me, obvious difficulties in accepting this as a condition of things likely to occur in the hearth of any blast furnace.

Now I have observed when a furnace is blown out, that there is, not only in the zone of greatest heat, but far above it and in a comparatively cool position, a copious deposit of fine carbon. This I have hitherto ascribed to the dissociation of monoxide by iron or its suboxides, in the manner already mentioned. May not that noticed by Cailletet have been liberated from the ore under the circumstances just mentioned? permeated, as we know it becomes, with carbon. This separation I am supposing may take place as the reduced ore fuses at or near the tuyeres, and that the free oxygen in reality was obtained from a point, where the blast had not had time to combine with more carbon than that represented by the small quantity, mentioned in Cailletet's researches in the form of CO and CO<sub>2</sub>.

This dissent from the opinions of such distinguished chemists is expressed with much hesitation, but I venture to submit it as an alternative explanation which perhaps merits notice.

For reasons already adduced, the presence of carbon dioxide in the gases from a blast furnace constitutes an important feature in its action. Having regard to the thermal conditions of its interior, I see no grounds for hoping that the quantity 5.97 cwt. per ton of iron can be materially increased. This figure, however, is liable to fluctuation with differences in the composition of the pig iron, it being based on metal containing 93 per cent. of Fe. Percy mentions one of 96.274 per cent., for which the equivalent of C as CO<sub>2</sub> would be 6.18 instead of 5.97.

It may be useful to consider the obstacles to an increase of CO<sub>2</sub> beyond the points just named. It is possible, chemically speaking, to completely convert CO into CO<sub>2</sub> in reducing iron oxide, but practically in a blast furnace I regard it as impossible. Experimentally it has been proved, in the case of Cleveland ore, how slow the operation is when the reducing power of 2 vols. of CO is opposed by 1 vol. of CO<sub>2</sub> at temperatures below 480° C. An increase in the CO<sub>2</sub> would augment this inconvenience, and in that way the reduction of the ore might be delayed, until the mineral arrived at a level in the furnace where the heat would resolve any CO<sub>2</sub> which was formed into CO.

Let us, however, imagine an increase in the proportion of CO<sub>2</sub> to be practicable under circumstances where the work in the crucible permitted it. Suppose the coke to be reduced in quantity, and the deficiency in heat to be made up by an increase in CO<sub>2</sub>, generated in the reducing zone. The position of this zone is such that much of the heat generated therein would be carried off in the escaping gases. But, for the fusion of the slag and iron, &c., a certain amount of heat is required in the hearth, which has to be supplied by a given quantity of coke burnt with heated air. Diminish

this and disaster is sure to follow. It happens, however, that there are to be found ores so constituted that the work in the hearth is very much less than with a mineral resembling that of Cleveland and others. In the two tables an example is given under columns H., of the truth of this statement. In it grey forge iron is made in a very small furnace receiving its blast at only 200°, with 15·44 cwt. of fuel. It is true this fuel is charcoal, but I know from personal inquiry on Lake Superior, in Sweden, and in Carinthia that for like work, this form of fuel is not more effective than coke. The advantage of the position will be seen at once to be due to the diminished amount of duty involved in producing the ton of iron. This is apparent enough when we see that, instead of 80,000 to 85,000 calories being absorbed as in smelting Cleveland ore, 61,208 calories have sufficed for the Swedish furnace given under columns H. in the two tables.

Through the kindness of my friend Professor Åkerman, of Stockholm, I have the particulars of 27 Swedish furnaces, all using charcoal. Of these, 11 show a larger proportion of C as CO<sub>2</sub> than I have ever met with in Great Britain, where the proportion 50 vols. of CO<sub>2</sub> to 100 vols. of CO is, I suspect, never reached. Below, the actual vols. of CO<sub>2</sub> per 100 vols. of CO are given, and in some cases, along with the cwt. of C as CO<sub>2</sub> the weight of charcoal per ton of pig consumed, &c. are inserted:—

Vols. CO <sub>2</sub> per 100 vols. CO.	50·4	56·2	56·3	58·4	60·4	60·9	61·2	68·3	69·1	75·10	85·3
Cwt. C as CO <sub>2</sub> per ton pig.	4·24	4·99					4·89			5·47	5·83
„ charcoal „ „	19·36	19·02	18·2	19·60	20·4	17·01	17·26	15·70	15·66	17·50	15·44
„ ore „ „	36·36	41·40					39·71			39·13	36·44
„ limestone „ „	3·24	2·60					5·09			5·47	4·36
„ slag produced „ „	12·26	16·02					15·56			15·15	12·01
1 of C as CO <sub>2</sub> to 1 C as CO.	1·98	1·78	1·77	1·71	1·65	1·65	1·63	1·46		1·33	1·17
Temp. of blast C .....	200°	300°	200°	200°	170°	300°	200°	100°		230°	200°
„ gases C .....	400°	145°					182°			225°	222°
Height of furnace ... feet	54½	53½					48¾			50½	42
Weekly make per 1000 c. ft.											
of furnace capacity . tons.	40·7	41·3					47			41·6	41·1

The large production per 1,000 cubic feet of furnace space, twice that of the Cleveland furnaces, may be taken as chiefly due to the readiness with which these Swedish ores part with their oxygen.

In none of these, it will be seen, does the C as CO<sub>2</sub> in the gases amount to what is commonly found in the furnaces of our own country, reckoned upon the ton of iron made.

#### CYANOGEN IN THE BLAST FURNACE.

There is one chemical reaction in the blast furnace which merits some notice, viz., the generation of cyanogen. This is not from any importance the subject as yet possesses as a commercial question, because the quantity so produced is not large, and of this a great portion is decomposed by the oxide of iron it meets with in the upper or reducing zone. The formation of this compound of carbon and nitrogen is doubtless due to the presence of soda and potash in the materials used, and it seems highly probable that these alkalies, or their metallic bases in the form of sodium and potassium cyanides being volatilised and partially deprived of their cyanogen, are condensed in the upper regions of the furnace and then returned to the hearth over and over again. From some cause the amount of cyanogen present varies considerably at different times, the point of generation apparently being at the hearth. Below is inserted in grammes the weight found per cubic metre of gas on six

consecutive days, four feet above the tuyeres, and to this is added the quantity remaining in the gases after leaving the furnace:—

							Average.
Grammes per cubic metre . . . .	19·00	12·93	17·32	11·34	20·61	9·16	15·06
Grammes in escaping gases . . . .	4·00	6·60	3·57	2·91	1·79	3·77	3·77

Upon another occasion the gases were examined for cyanogen at different depths, with the following results:—

Height above the hearth . . . . .	8 feet.	24 feet.	52½ feet.	60 feet.	Escaping pipe.
Grammes per cubic metre . . . . .	49·06	15·76	7·67	5·94	4·73

Having regard to the value of cyanogen in the arts, it is questionable whether its production, in the way described, has received all the attention it has deserved.

#### AMMONIA IN THE BLAST FURNACE.

In furnaces which are fed with coke, small quantities of ammoniacal salts are detected in the gases. When raw coal is the fuel used, compounds of ammonia, chiefly sulphide, are given off in such a volume that the gases are passed through huge condensers, and the alkali so obtained is treated with sulphuric acid and delivered to commerce in the form of sulphate. The weight of this substance thus obtained amounts to about 50 lb. per ton of pig iron made.

#### FUME GIVEN OFF FROM BLAST FURNACES.

After the gases have been used as a fuel for raising steam and heating the air, there is emitted from the chimneys vast volumes of white smoke. Of this large quantities condense in the flues, &c., and its removal is a source of some expense. Naturally this condensed matter varies in composition and no doubt in quantity according to the nature of the ores employed. By means of an air-pump a certain volume of gas from a Cleveland furnace was drawn through water. The matter condensed was partly insoluble and partly soluble in water, about half of each. They had the following composition:—

	Insoluble in Water.	Soluble in Water.
Silica . . . . .	11·00	1·37
Alumina and ferric oxide . . . . .	10·76	12·20
Lime . . . . .	2·06	Trace
Magnesia . . . . .	Trace	Trace
Oxide of zinc . . . . .	13·28	4·58
Alkaline salts . . . . .	3·07	..
Carbon dioxide . . . . .	7·00	..
Chlorine . . . . .	..	0·57
Sulphuric acid . . . . .	..	0·59
Sodium and potassium carbonates . . . . .	..	22·90
Loss on heating . . . . .	..	10·46
	47·17	52·67
	Total . . . . . 99·84	

It has been attempted, in the present contribution to the work of your Society, to establish for the blast furnace a character for an economical use of fuel. At the same time it has been proved that the duty performed is by no means equivalent to the whole of the useful effect which coke is capable of affording. Taking a ton as being able to produce 20 cwt. of pig iron from Cleveland ironstone, this, after an allowance for ash, &c., represents, when burnt to carbon dioxide, 147,200 calories, whereas something like 90,000 calories has been the limit of the power obtained. This apparent sacrifice of nearly 40 per cent. is incurred by what is considered the physical impossibility of bringing, in this particular process, the whole of the carbon burnt to its highest point of saturation with oxygen.

Forty years ago this unexpended energy was not turned to any useful account. This loss is now avoided by burning, under the boilers and in the hot-air stoves, the carbon escaping from the furnace as combustible monoxide. By this beautiful application of what was a waste product, all the mechanical power needed at the works is obtained, at the same time that the blast is heated, and these advantages are accompanied by a considerable saving of labour. With the present improved form of furnace the coal consumed for these two purposes would be much less than formerly, but even now it would not be much short of 10 cwt. per ton of metal. This calculation is based on the steam engines being on the non-condensing principle, but assisted with condensers, steam enough is raised to do one-fourth more duty than that called for at the furnaces.

When fire-brick stoves supply the heat to the blast, the loss at the chimneys is about one-half of what it is with those of iron pipes. In point of fact at the former, the temperature of the products of consumption does not greatly exceed that required for maintaining a good draught.

There remains, however, as always happens, a considerable loss at the boiler chimneys, but this at the Clarence Works is utilised by applying the waste heat for the evaporation of brine. A similar use is about being tried, at the same place, with the heat contained in the molten slag before it is thrown over the spoil heap.

P.S.—The length to which this address grew prevented my going into the reasons for doubting the formation of any large quantity of  $\text{CO}_2$  at the tuyeres, as stated by Roseoe and Schorlemmer. My friend Ritter von Tunner has also, I think, expressed a similar opinion. As a question of heat evolution, within a very small area, the formation of  $\text{CO}_2$ , or the reverse, is immaterial, because as is stated by these chemists, any  $\text{CO}_2$  so produced is rapidly converted into  $\text{CO}$ .

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Table II.—At the foot of the page is an estimate of the heat evolved by the combustion of the fuel to which is added that carried into the furnace by the blast. The first line in this account shows the number of calories produced by the whole of the carbon of the fuel into CO less the quantity which has been carried off by the CO<sub>2</sub> of the limestone. The second line sets forth the additional heat afforded by the conversion of such a portion of the C in the CO as analysis indicates has been changed into CO<sub>2</sub>, and the third represents the heat in the blast.

The total carbon in the gases, in the tenth line of the page, is divided into the ascertained proportion of CO and CO<sub>2</sub>, and from these the quantity of oxygen present is calculated. This oxygen is contributed partly by the minerals and partly by the blast. That contained in the former, being calculated, is deducted from the whole, and the difference is considered as due to the atmospheric air of the blast. An estimate of the nitrogen is then made, and from all these factors the weight of the blast and of the escaping gases is easily obtained.

Following the estimate of the heat generated is a division showing the value of each unit of fuel used and from what source such value is derived, and lastly the relation in weights between C as CO and C as CO<sub>2</sub> is added: that in column A being 1 of C as CO<sub>2</sub> to 6.12 as CO; B, 1 of C as CO<sub>2</sub> to 3.74 of C as CO, and so on.

G represents a furnace working on English hematite ore, inserted as a comparative test of general correctness of mode in the other calculations.

TABLE II.

	A.		B.		C.		D.		E.		F.		G.		H.	
Height and cubic capacity .....	48 ft. 6,000 c. ft.		48 ft. 6,000 c. ft.		80 ft. 15,000 c. ft.		90 ft. 33,488 c. ft.		80 ft. 15,500 c. ft.		76 ft. 20,454 c. ft.		65 ft. 10,360 c. ft.		42 ft. 1,434 c. ft.	
Coke per ton, of No. 3 pig iron .....	40.76		28.50		22.50		19.69		19.99		19.49		21.06		15.44 charcoal	
Less ash, moisture, &c. ....	4.94		2.56		1.92		1.73		1.75		2.05		1.53		3.26	
Carbon in coke .....	35.82		25.94		20.58		17.96		18.24		17.44		19.52		12.00	
Carbon in limestone carrying off C of coke ..	2.19		1.92		1.32		1.26		1.32		0.22		0.91		7.52	
Leaving carbon to be burnt at tuyeres ..	33.63		24.02		19.26		16.70		16.92		17.22		18.61		11.56	
Weight of carbon delivered to furnace in coke and flux .....	38.01		27.86		21.90		19.22		19.56		17.66		20.13		12.00	
Less absorbed by iron .....	0.60		0.60		0.60		0.60		0.60		0.60		0.60		0.60	
Carbon in gases .....	37.41		27.26		21.30		18.62		18.96		17.06		19.53		12.00	
Carbon in gases as CO .....	32.16 = 0		21.51 = 0		15.33 = 0		12.96 = 0		12.99 = 0		12.73 = 0		15.26 = 0		6.17 = 0	
" " CO <sub>2</sub> .....	5.25 = 0		5.75 = 0		5.97 = 0		5.66 = 0		5.97 = 0		4.33 = 0		4.27 = 0		5.83 = 0	
C and O in gases .....	37.41		27.26 = 0		21.30		18.62		18.96 = 0		17.06 = 0		19.53		12.00 = 0	
Oxygen derived from materials :																
From Fe <sub>2</sub> O <sub>3</sub> in the ore .....	8.23		7.97		7.97		7.97		7.97		7.97		8.23		8.23	
" SiO <sub>2</sub> , SO <sub>3</sub> , P <sub>2</sub> O <sub>5</sub> , and Ca in slag ..	0.50		1.25		1.25		1.25		1.25		1.25		0.50		0.50	
" CO <sub>2</sub> in limestone .....	3.81		3.10		3.52		3.36		3.52		0.50		2.44		1.19	
" moisture in blast .....	1.40		0.91		0.66		0.60		0.60		0.60		0.63		0.50	
	15.97		15.23		13.40		13.18		13.34		10.41		11.50		10.42	
Difference being supplied by atm. air ...	40.91		28.78		22.96		19.19		19.90		18.11		19.94		13.86	
Weight of escaping gases and composition :																
Oxygen per ton of pig iron, as above ...	56.88		44.01		36.36		32.37		33.24		28.52		31.74		23.78	
Carbon per ton of pig iron, as above ...	37.41		27.26		21.30		18.62		18.96		17.06		19.53		12.00	
Hydrogen from blast .....	0.18		0.11		0.09		0.08		0.07		0.07		0.07		0.04	
Moisture in coke .....	2.49		0.74		0.58		0.58		0.58		0.53		0.54		0.50	
Nitrogen accompanying atmosp. O. ....	136.96		96.25		76.87		64.24		66.62		60.63		66.74		44.72	
	233.92		168.37		135.20		115.89		119.47		106.81		118.62		81.04	
Weight blast per ton of pig :																
Nitrogen, as above .....	136.96		96.25		76.87		64.24		66.62		60.63		66.74		44.72	
Oxygen, as above .....	40.91		28.78		22.96		19.19		19.90		18.11		19.94		13.86	
Moisture .....	1.62		1.02		0.58		0.58		0.63		0.63		0.63		0.50	
	179.49		126.05		100.41		84.01		87.15		79.36		87.81		59.38	
Summary of calories evolved per ton pig :																
Carbon burnt to CO at tuyeres .....	33.63 x 2,400 = 80,712		24.02 x 2,400 = 57,648		19.26 x 2,100 = 40,424		16.70 x 2,400 = 40,080		16.92 x 2,400 = 40,608		17.22 x 2,400 = 41,328		18.61 x 2,400 = 44,664		11.56 x 2,400 = 27,744	
Of this C as CO burnt to CO <sub>2</sub> .....	5.25 x 5,600 = 29,400		5.75 x 5,600 = 32,200		5.97 x 5,600 = 33,432		5.66 x 5,600 = 31,696		5.97 x 5,600 = 33,432		4.33 x 5,600 = 24,248		4.27 x 5,600 = 23,912		5.83 x 5,600 = 32,648	
Heat in blast .....	1,068		14,188		11,538		14,136		14,537		14,882		11,201		2,775	
	111,180		104,436		91,194		85,912		88,377		79,958		79,956		63,117	
Heat per unit of coke and units C as CO per unit C as CO <sub>2</sub> :																
Cal. Units C as CO	1,080		3,023		2,034		2,066		2,032		2,120		2,056		1,671	
By carbon burnt to CO in the gases ...	721		130		1,486		1,609		1,672		1,244		1,136		2,114	
Of this C as CO burnt to CO <sub>2</sub> .....	26		507		513		718		727		738		732		179	
Heat in blast .....	2,727		3,660		1,053		4,363		4,431		4,102		3,733		3,874	

\* 1,068 cal. is heat of combustion of 1 lb. of charcoal.



Mr. E. RIDER COOK, in moving a vote of thanks to the President, said that though the subject treated was strange to himself, he could see that it was of great interest to those who were concerned with or understood the subject. There was, however, one feature of the paper which had struck him, as it probably had also struck other members not intimately acquainted with the iron manufacture. It was that in the paper just read they had a striking illustration of the results of the application of science and accurate knowledge to a particular trade, and saw that the effect upon that trade was precisely similar to that which followed a similar course in the case of other industries. The general public would think as they saw the great blast furnaces at work that the smelting of iron could hardly involve a chemical operation. They would probably imagine, as they saw the heaps of coal and metal, and rough-and-ready beings tumbling them into the huge furnaces, that something was going on which could not have much connexion with the laboratory or with chemical science. But the paper had proved to him at any rate that where able men like the President had brought science and chemical knowledge to bear upon this rough operation, they had been rewarded by commercial prosperity in the saving of fuel. It had also shown him that where certain men without having chemical knowledge had yet been able to effect great improvements in the manufacture, subsequent investigation had nevertheless shown that unconsciously the improvers had been working on scientific lines. The lesson they ought to draw from the paper was that each member should seek to cultivate a stronger faith in the principles which the Society was intended to teach; that whether the substances they were dealing with were chemically combined or not, the laws of science might be brought to bear upon them with advantage; that the old system of rule of thumb might always be improved upon by those who acquainted themselves with the laws of nature and to the best of their ability brought them to bear upon the various industries with which they had to do.

Mr. LUDWIG MOND said that he rose with great pleasure to second the motion for a hearty vote of thanks to the President for the highly interesting and instructive address to which it had been the privilege of the members to listen. Sir Lowthian Bell had laid before them the work of his lifetime, together with his latest results and his latest opinions; and all those who were concerned in the manufacture of iron, or the treatment of masses of material by heat, or generally in the application of science to large industries and practical work, must have followed the address with the utmost interest. It was unnecessary for him to dwell upon the importance of the paper for the particular industry to which it referred; but he wished to point out the immense importance which the paper proved of investigating step by step all that happened in an industrial process and apparatus, however simple they might appear to be on paper (and nothing could be simpler than the reduction of iron by carbon as described in the olden times), and thus establish what the consecutive steps of the process really were. It was only by proceeding in that manner, and thus gaining an accurate knowledge of the complications really underlying what was apparently simple, that the best possible industrial results could be attained.

The motion having been put and carried by acclamation,

The PRESIDENT said in reply that his friends the mover and seconder of the vote had each endeavoured to commend themselves to the gratitude of the meeting by making a short speech, and he would endeavour to earn even deeper gratitude by making a still shorter one. He would merely say, therefore, that he had been agreeably impressed by the kind attention which the members had paid to the address which he had prepared for the occasion. He had certainly learned one good lesson from the Latin studies of his early days. It was expressed in the line: *Rerum cognoscere causas*, and he had ever since thought it desirable to endeavour to ascertain at the time the cause of any phenomena in which he was interested. He would only say in conclusion that he felt very grateful to the members

for their vote of thanks; for if he talked for half an hour he could not add to, and might weaken, that expression of his feelings.

The SECRETARY then read the report of the scrutators, which showed that 176 ballot papers had been sent in, and that the following had been elected to fill six vacancies among the ordinary members of Council:—Mr. A. H. Allen, Mr. H. Grimshaw, Professor R. Meldola, F.R.S., Mr. E. K. Muspratt, Mr. T. L. Patterson, and Mr. T. W. Stuart.

The following is a list of the Council for 1890–91:—

*President and Treasurer.*

E. Rider Cook.

*Vice-Presidents.*

Sir Lowthian Bell, Bart, F.R.S.	Ludwig Mond.
R. Forbes Carpenter.	Dr. Hugo Muller, F.R.S.
Prof. James Dewar, F.R.S.	B. E. R. Newlands.
James Duncan.	Dr. W. H. Perkin, F.R.S.
David Howard.	Dr. Edw. Schunck, F.R.S.
S. H. Johnson.	J. C. Stevenson, M.P.

*Ordinary Members of Council.*

A. H. Allen.	Prof. R. Meldola, F.R.S.
L. Archbutt.	E. K. Muspratt.
S. B. Boulton.	T. L. Patterson.
Jno. Brock.	Jno. Pattinson.
Jno. Calderwood.	Jno. Spiller.
H. Grimshaw.	T. W. Stuart.

*Foreign Secretary.*

Dr. Ferdinand Hutter.

*ELECTION OF AUDITORS.*

Mr. S. H. JOHNSON moved that Messrs. Theobald Bros. and Miall, Chartered Accountants, be re-elected auditors for the ensuing year. The motion was seconded by Mr. T. TYLER and carried unanimously.

*ANNUAL GENERAL MEETING FOR 1891.*

Mr. T. TYLER moved that the Society meet at Birmingham in 1891. Letters received from the Chairman of the Birmingham Section (who was prevented by indisposition from attending the present meeting) and from various gentlemen connected with the town made him feel quite sure that the Society would receive such a welcome there as the reputation of the town for hospitality would lead one to expect.

Mr. R. B. BATTY seconded the motion, which was carried unanimously.

The PRESIDENT then called upon the meeting for a hearty vote of thanks to the Senate of University College. Although such a vote was generally regarded as mere matter of form, it was only proper to express to the gentlemen connected with that magnificent establishment the assurance of the members' complete appreciation of what they had done towards the reception. Judging of their sentiments in that direction by what they had done for the cause of education by the construction and maintenance of that splendid building, he felt sure that those gentlemen must take a sufficient interest in the visit of the Society.

Mr. NORMAN TATE had much pleasure in seconding the vote proposed by the President. He felt sure that all the members present must feel pleasure in noting what had been done at Nottingham in the way of giving scientific instruction which would help forward the progress of that industrial chemistry in which they all felt so deep an interest.

The vote of thanks having been passed unanimously,

The MAYOR said that on behalf of the committee entrusted with the management of the College he thanked the Society for the way in which their services had been appreciated. As representing the Corporation, he felt proud that they were in a position to receive the Society in an establishment like that in which they were then assembled.



He believed that he might say without fear of contradiction that the Corporation of Nottingham was the only one that had had the courage to do what they had done, namely, to work a University College. There were other colleges in existence which no doubt far exceeded theirs in magnitude and usefulness, but none of them had been built by the town and for the town. Other similar institutions elsewhere were built and worked by private enterprise or contributions; but that in which they were now assembled was built by the town's money, and would, he hoped, do good service to the town and to the Society represented there that day.

### VISITS TO WORKS.

#### VISIT TO THE MIDLAND RAILWAY WORKS, DERBY.

About 120 members proceeded by train to Derby to visit the works of the Midland Railway Company, travelling in saloon carriages provided by the Nottingham Section. On arrival at Derby, the party proceeded to the Midland Hotel, and, by invitation of the Nottingham Section, partook of luncheon. After luncheon, the President and a party of about 100 members proceeded to the locomotive works, where they were met by Mr. S. W. Johnson, the locomotive superintendent, Mr. Holt, the works manager, and other officials. Time did not permit of more than a hurried inspection of the extensive works, which cover nearly 12 acres of ground adjoining the Midland Railway station, and give employment to more than 3,000 men. Commencing with a visit to the laboratory, and walking through the millwrights' shop, the party proceeded to the mess rooms, capable of accommodating 700, 500, and 300 men respectively, in which a staff of cooks find constant employment in the preparation of the meals of those workmen who live at a distance. The party thence proceeded to the spring shop, and through the wheel and tyre turning shops, and the smithy, to the boiler shop, where the steam rivetting machine, the powerful shearing machine, and the machine for drilling the holes for boiler stays evoked considerable interest. Escaping from the deafening din of the boiler-smiths' hammers, the route was continued through the smaller erecting shops, the press shop, the coppersmiths' shop, and the brass foundry, to the iron foundry, a lofty building 180 ft. long by 90 ft. wide, where about 310 tons of cylinders, chairs, and all kinds of general castings are turned out every week. Adjoining the iron foundry were seen the gas works which supply the various shops, as well as the offices and station, with gas, the quantity made averaging about  $1\frac{1}{2}$  million cubic feet per week. The next shop visited was the large erecting shop, 150 ft. broad by 450 ft. long, covered by a roof of three spans, and capable of accommodating 90 engines in progress of construction and undergoing repairs. The six powerful travelling cranes, each capable of lifting 25 tons, and worked by endless cords, were objects of interest. The adjoining fitting, turning, and brass-finishing shop was much admired. This shop has four spans, each 40 ft. wide by 450 ft. long, and contains no less than 460 separate machines driven by a quite bewildering mass of belting. The many interesting machines in this shop would have repaid a more lengthened inspection, but as time pressed the party proceeded to the painting shop, where an opportunity was afforded the members of viewing and examining a newly-finished engine and tender similar to that which gained the "Grand Prix" at the recent Paris Exhibition, and which was very much admired. A full description of this engine appeared in "Engineering" on March 2nd and 16th, 1888. A walk through the new engine shed, where accommodation is provided for 48 engines to get up steam and stand in readiness for their respective trains, brought the visit to a close.

A smaller party of about 20 members had in the meantime proceeded to the carriage works, where the manager, Mr. Osborne, and other officials took charge of the party and conducted them through the various shops. The carriage works of the Midland Railway Company are situated about half a mile south of Derby station, and cover  $14\frac{1}{2}$  acres of ground with  $10\frac{1}{2}$  miles of sidings and

rails. They are the largest of their kind in the kingdom, and consist of seven large shops, four on the west side devoted entirely to the preparation of timber, putting together of material, and finishing off of vehicles, and three on the east side devoted entirely to metal work; all the shops being conveniently arranged for the direct transmission of work from one to another by means of steam traversers, so as to avoid all unnecessary labour in passing backwards and forwards. The sawing and wood-working mill is 320 ft. long by 200 ft. broad. The floor is supported on more than 500 cast-iron columns, and all the main shafting, pulleys, belting, &c., are situated in the cellar below. This mill contains about 100 machines specially designed for the various operations of wood working. Next in order is the wagon shop, of the same dimensions as the saw mill, and here the prepared timber from the mill and the prepared metal-work from the machine shop are put together in the form of waggon. The demand for waggon by the Midland Railway Company, 66 per cent. of whose traffic is goods, is very great, and has so much increased of late years that it has been found necessary to build a new shop, considerably larger than any of the older shops, which is entirely devoted to wagon building, and some idea of the facilities afforded for this kind of work may be gathered from the fact that on an average one new wagon is completed every 20 minutes. In the next shop to the old wagon shop the building of carriages is carried on, and the finished carriages pass thence to the painting and trimming shop where they undergo no fewer than 25 different operations of priming, filling-up, rubbing down, painting, varnishing, &c., to enable them to withstand the weather and present a satisfactory appearance. The lighter part of the upholstery work and the French polishing connected with the vehicles is carried out by women and girls, who work in a separate portion of this building with separate mess-rooms and offices. The iron foundry, smithy, bolt and spring shop, wheel-tying shop, and machine and fitting shop were all inspected, and after a most interesting afternoon the members joined the larger section of the party from the locomotive works and returned to Nottingham in the saloon carriages in which they came.

#### MESSRS. J. AND R. MORLEY AND CO.

Visits were also paid by some of the members to the hosiery factories in Manvers Street and Handel Street belonging to the well-known firm of Messrs. J. and R. Morley, whose manufactories have been fixed in Nottingham and its neighbourhood for almost a century.

In these factories the various processes of manufacture were watched with much interest, special attention being given to the new patent self-acting fashioning frames, of which this firm, in their various factories, are said to have the largest plant in the world.

Machines for making the well-known Derby-ribbed work, and also the numerous kinds of natural wool underclothing now so largely used, were closely inspected by the visitors, who expressed much pleasure at what was shown to them.

In the Handel Street factory, in addition to the making of hosiery goods, the process of cotton spinning is also carried on, and this added to the interest of the visit.

#### TRENT BRIDGE LEATHER WORKS.

A party of about 25 members assembled at the works of Messrs. Turney Brothers, Limited, leather manufacturers, Trent Bridge, and were shown over by Mr. J. T. Wood.

Commencing with skins as they entered the works from the fellmongers' yards, the party followed them through the various processes—the liming, fleshing, splitting, bating, and tanning (both by sun and bark).

They then inspected the manufacture of chamois or wash-leather by means of fish oil, the felling mills, hydraulic presses, and the scouring of the leather, and saw the finished article being packed in the warehouses for export.

The dyeing and finishing of the leather for bookbinding, purse-making, boot uppers, boot linings, and hat leathers was next inspected, and a new product which the firm are manufacturing, the glazed Dongola leather for the upper leathers of high class boots, attracted considerable attention.

The salting or pickling of skins for the American market was next examined, and after looking at the laboratory the party went away expressing themselves very pleased with their visit.

The following works were also open for inspection:—

Messrs. Blackburn's Hosiery Machine Works, and Messrs. Lindley, Wright, and Cox's Lace and Hosiery Finishing Works.

#### CONVERSAZIONE AT THE CASTLE.

On Wednesday evening the Mayor of Nottingham (Alderman Goldschmidt) recognised the presence of the Society in the town by giving a conversazione in the Castle Museum and Art Gallery. Upwards of 850 invitations were issued, and of this number about 800, including 200 members of the Society, accepted the kind hospitality of the Mayor. Eight o'clock was the hour fixed for the commencement. The arrangements in every respect were admirable, both inside and outside the Castle. The guests were received in the Long Gallery at the north end by the Mayor and Miss Goldschmidt, in the absence of the Mayoress, who was unfortunately too indisposed to be present. The Mayor's party consisted of Lady Turney, the Sheriff (Councillor Pullman) and Mrs. Pullman, Mrs. S. G. Johnson, and Mrs. Lewis. The gallery was brilliantly lighted, and soon the scene was a very pleasing one. Behind the Mayor and party were a number of palms, plants, and flowers, and throughout the rooms and on the steps were similar banks of colour. From the Long Gallery the guests could promenade at will through the Water Colour Gallery, and into the Dawson Gallery, where light refreshments were served. The guests could wander still further, either into the well-lighted museum to admire the many interesting curiosities, or into the second refreshment room on the ground floor, and so on to the East Terrace. From thence gentlemen and their more timid companions, who cared to venture under ground, could be conducted by an attendant to "Mortimer's Hole." Another opportunity was also given to those who chose to seek the refreshing, but somewhat cold breezes of the night, the entrance to the Castle roof being thrown open. At no period of the evening were the galleries uncomfortably crowded, and the absence of this almost inevitable feature of all such functions was doubtless matter of congratulation on the part of the assembled guests. Mr. Watson's band played capital selections of music in the Water Colour Gallery. From the Castle green, during the early portion of the evening, there also floated the sweet strains of a band, which at dusk played inside the Castle. It was not until 10 o'clock that the loud hum of conversation noticeably lessened, and the pleasant lounges began to be forsaken; but after that hour the guests gradually dispersed, the conversazione concluding at 11 o'clock.

#### SECOND DAY.

##### VISIT TO THE BURTON BREWERIES.

On Thursday morning a party of about 100 members proceeded to Burton, where they were met by Messrs. Matthews and Lott, secretaries of the reception committee. From the station the party proceeded in sections of about 20 to the breweries of Messrs. S. Allsopp and Sons, Lim., Messrs. Bass, Ratcliffe, and Gretton, Lim., Messrs. Ind, Coope, & Co., Lim., Messrs. Salt & Co., and Messrs. Worthington & Co., Lim. At the several breweries they were received by Dr. Harrow, Mr. T. C. Martin, and Mr. Strachan (Allsopp's); Mr. C. O'Sullivan, Mr. J. O'Sullivan, Mr. Walter Odling, and Mr. Jas. McGregor (Bass's); Mr. Skilton and Mr. Lloyd (Ind, Coope's); Mr. Adrian J. Brown, Mr. H. Daniel, and Mr. W. Newton (Salt's); and Dr. G. Harris Morris, Mr. W. Swinnerton, and Mr. C. Fletcher (Worthington's). A thorough inspection from the malt-house to the tasting-room was afforded in each case, and the familiar process of brewing was exhibited under the most favourable condition that a perfect water and the highest technical skill can supply.

After two o'clock the various parties met at the St. Paul's Institute where, by the hospitality of the five firms above

mentioned, members partook of a sumptuous luncheon. At the conclusion of the repast Mr. Ludwig Mond and Mr. Muspratt proposed and seconded in brief speeches a warm vote of thanks to the five firms, their hosts. Mr. David Howard asked leave to include in this the names of those who had received and escorted the various parties. The votes of thanks were carried by acclamation. Mr. C. O'Sullivan (Messrs. Bass & Co.), Mr. Martin (Messrs. Allsopp & Co.), and Mr. Lott responded.

##### MESSRS. SAMUEL ALLSOPP AND SONS' BREWERIES.

Those members visiting the breweries of this firm were first conducted to the new brewery. Besides the fine offices and the huge storeroom, which is capable of holding over 30,000 casks, the capacious well, the largest in Burton, and having a diameter of 46 ft., formed an object of interest. The party was now taken by a special train to the middle yard, where, after seeing the stables, it was shown through the general stores. The train then steamed across to the old brewery, and here Mr. Martin was able to explain in detail the brewing process, commencing with a mash which was carried out whilst the party looked on. Several of the members were much interested in the special washing treatment this firm subjects the yeast to which is not utilised on the premises. It is thus possible to obtain a higher price for this material than could otherwise be obtained. In connexion with this Mr. Martin said he was glad to know that some members of the Society were devoting their attention to the more advantageous disposal of brewery by-products, and hoped they might be successful in finding a means of replacing German yeast by a similar article made in this country from brewer's yeast, and also make spent hops and grains of more service to the farmers.

Returning to the new brewery, the party visited the two large union rooms, holding respectively 1,624 and 1,376 casks, and capable of cleansing nearly 500,000 gallons of ale at one time; and the enormous racking hall, 400 ft. long by 100 ft. wide.

Before leaving the premises, a visit was paid to the laboratory, where Dr. Harrow, successor to the late Dr. Griess, with whom he had for many years been working, pointed out many of the more peculiar pieces of apparatus and contrivances for analysing beer and the materials necessary for its production.

##### MESSRS. BASS, RATCLIFF, AND GRETTON, LIMITED, BREWERIES.

Messrs. Bass and Co. have three breweries in Burton, the Old, the Middle, and the New—in each of which the process of brewing is carried on independently and completely. The capacity of the three together is at present somewhat over 30,000 barrels weekly.

The first department visited was the cooperage, adjoining the Middle brewery. This was seen in full work, about 40,000 casks being required annually for the export trade alone. There was more machinery in this department than is usually seen in connexion with this industry, and the method of closing the staves, after one end of the cask had been secured, by surrounding it with a powerful chain and tightening this by hydraulic power, was watched with some interest. The party then proceeded to the extensive hop-stores, with about 800 tons of hops in stock, kept in the dark to preserve the flavour.

Underneath these were the ale stores, where samples were produced for examination by the visitors. From this point the party was conveyed in waggons by a private railway line round the town to the Old brewery.

Here the pumping machinery for the water supply was first inspected; there being separate sets of pumping machinery for the brewing, the cooling, and the washing water. The brewing water is raised from wells, adjoining the Shobnall Maltings, about a mile and a half away; this is pumped into the brewing tanks, while the cooling and washing waters are stored in large service tanks at the top of a tower. Burton water is drawn from the new red sandstone foundation. This sandstone contains mud and gypsum, from which the water dissolves calcium sulphate to the extent of about 200 grains per gallon, making it extremely hard. It is also free from organic matter, a

most important fact as affecting the fermentation. It was formerly supposed that the presence of this calcium sulphate was advantageous, as hindering the solution of the albuminous portions of the malt; but experiments have since shown that this is not so, and that the chief merit of the calcium sulphate lies rather in its antiseptic properties, which serve to keep the fermentation under better control. Near the pumping engines are situated the boilers to supply the hot water required for the mashing process.

In the top storey of the Old brewery the crushed malt is passed in thin streams into the mash tub, where it is treated with hot water, the mixture being kept in motion by an agitator revolving on a vertical axis, on the arms of which again revolve paddle-wheels. The room visited contained seven mash tubs, each capable of mashing 60 quarters of malt. The "wort" flows thence through a perforated bottom to the "underback," a species of storage reservoir, and thence to the "coppers," where it is boiled with hops. These coppers are heated by an open fire. Thence the boiled wort flows to the hop-back, in which the hops are retained by a perforated false bottom; it is pumped on to the "coolers," large shallow trays of wood; it then flows into the "refrigerators," consisting of tanks containing great lengths of copper tubing through which a current of cold water flows. The wort having reached a proper temperature now passes into the "fermenting squares," where yeast is added, and where the evolution of carbonic acid is very marked. There are on one floor 160 of these "squares," each of 50 barrels capacity—a very large plant. From the squares the beer is allowed to flow, at the proper time, into the "union room," a feature peculiar to Burton. This "union room" contains rows of large casks communicating in "sets," and when in work kept constantly full of beer. From the top of each cask issues a pipe which rises a few feet and then dips over into a trough. In these casks fermentation is completed, and the yeast is gradually forced up the ascension pipe from the full cask and is collected and retained in the troughs. The temperature of both casks and troughs is regulated by a current of cold water passing through tubes inserted in them. From these the beer is drawn off by a moveable cock which so projects upwards into the cask that the sediment is not disturbed. The union room is 125 yards square, and can contain 2,500 casks, each of 160 gallons capacity. From this room the beer passes into the "racking room," where the process of brewing ends, the finished beer being here "racked" from large tanks into casks of various sizes for use.

The party next proceeded to view the private laboratory of Mr. C. O'Sullivan, F.R.S., who manages this brewery and directs the scientific department connected with the whole concern, and then resumed their places in the waggons and were conveyed to Shobnall. The firm own 32 maltings at Burton, of which seven are at Shobnall—all connected—and they own and work many other maltings at Lincoln and elsewhere. Here barley is steeped in warm water, then allowed to germinate on large floors, and finally kiln-dried at a temperature increasing from 110° to 160° F. The capacity of the Shobnall Maltings is 60,000 quarters.

#### •MESSRS. IND, COOPE, AND CO.'S BREWERY.

The party which visited Messrs. Ind, Coope, and Company's brewery were ably conducted over the establishment by Mr. C. F. E. Skilton. The Burton work is, we believe, not so large as the same firm's brewery at Romford, but it struck us as being singularly compact and well arranged, and every department was a pattern of neatness and good order.

The visitors were taken first to the mashing rooms, of which there are two, containing four mashing tuns each, the total working capacity of which was stated to be about 360 quarters of malt. From these they proceeded to the rooms in which the wort from the mashing tuns was being boiled with its complement of hops in six huge coppers, each of about 110-barrel capacity. On the way to the next department the visitors examined the mill in which the malt is crushed by steel rollers, and from which it is distributed by means of hoppers to such of the mashing tuns as require feeding. Next were seen the two large cooling tanks, which are used for surface cooling only, a very simple floating arrangement being employed by means of which

the pipe that carries off the liquor to the refrigerators is kept just below the cooled surface. The new system of refrigerators adopted by the firm appear to be very ingenious and effective. Each apparatus (of which there were two of a series of four in work) consists of a stack of horizontal pipes through which cold water continually passes, the warm liquor trickling down from the top and passing over the edges of the series of pipes, finally leaving by a trough at the bottom cooled down to 58° F. From here the party was conducted to the hop store, passing on the way the new tanks for supplying hot water to the mash tuns. Of these there are nine, each of 3,400 gallons. Traversing the larger of the two fermenting rooms (containing 32 tanks) the visitors proceeded to the "union room," wherein is conducted the process of final fermentation and cleansing by the Burton method, details of which are given in the paragraph relating to the visit to Messrs. Bass and Co. It should be mentioned, however, that in one "union room" Messrs. Ind, Coope, and Co. have adopted the plan of cooling by means of pipes running along above the barrels; but it was stated that the method does not work so well as the old system of passing cold water through pipes traversing the barrels. The finished beer is drawn off from these barrels and run through troughs into the "racking vaults" beneath, where it is casked.

After inspecting the Reece's freezing apparatus, the yeast pressing room, and the laboratory, the visitors retired to the stores, where they tasted samples of the various ales produced by the firm, finishing with a bumper of the special "Jubilee brew."

#### THOMAS SALT AND CO.'S BREWERY.

Those visiting Messrs. Thos. Salt and Co.'s brewery first drove to the storerooms in Wetmore Road. Thence the party was conducted to the cooperage, where, after inspecting the unheading, washing and steaming of casks, they examined numerous machines for cutting and preparing the oak staves so that they might be put together into casks of various sizes. The malt-houses were next visited, and although in summer time malting is not carried on, the process was thoroughly explained. After this, the members were rapidly conveyed in a railway waggon to the brewery proper—about half a mile away. In order were seen first the crnshing of the malt, attention being specially directed to the grooved or fluted steel rollers used by this firm in preference to the smooth ones elsewhere in vogue. Next the raising of the malt by automatic elevators. Then the mash tubs, five in number, each capable of holding 60 quarters of malt and 120 barrels of water. Here attention was called to the device by which the brewer in charge could control both temperature and volume of water used. Then came the copper-house containing eight large coppers, whence the boiled wort is pumped to the top of the building and run into large shallow copper coolers and subsequently over refrigerating pipes, and so down to the "square" room. As elsewhere in Burton fermentation is carried on here first in "squares" or open vats, and subsequently in casks or "unions," where the finished ale is cleared of its yeast. Messrs. Salt's "union" room is capable of holding as much as 3,600 barrels of ale at one time. Below is the racking-room, one of the largest of its kind in Burton, where the finished ale is run into wooden rounds, holding 100 barrels each, prior to being put into casks. After inspecting the yeast presses the party proceeded to the St. Paul's Institute for lunch.

#### WORTHINGTON AND CO.'S BREWERY.

The party for Messrs. Worthington and Co., Limited, were conveyed in breaks to the brewery premises in High Street. Here the stores were first visited. From the stores the party proceeded through the maltings. Owing to the time of year no malting operations were in progress, but the visitors had an opportunity of examining the malt-houses, the largest of which, with a cistern of 180 quarters capacity, and floors capable of receiving a steep of this quantity every four days, was much admired. The machinery for screening, cleaning, and grading the barley was also inspected, and after passing through the barley and malt stores, capable of holding 13,000 quarters, and from which the barley is

distributed to the different malt-houses by means of a travelling india-rubber endless band, the party visited the laboratory. Here the members were naturally much interested in the complete arrangements for the examination and analysis of wort, beer, malt, &c. The room fitted up specially for the pure cultivation of yeast by Dr. Hansen's method attracted great attention, and it was mentioned that the company had brewed as many as 6,000 barrels of beer with different varieties of pure yeast, each cultivated from a single cell. The results, however, were not sufficiently satisfactory to encourage a more extended use of pure cultivated yeast. Leaving the laboratory, the party passed on to the brewery on the opposite side of High Street, and, starting with the malt-mill, which contains a pair of east-iron rollers capable of crushing 50 quarters of malt per hour, the members followed the grist into the mash-room, containing three mash tuns, each fitted with a Steel's mashing machine capable of mashing 80 quarters in from 10 to 12 minutes. From the mash tuns the wort was followed to the coppers, the largest of which holds 160 barrels, and where it is boiled with the necessary amount of hops. From the coppers the party proceeded to the hop-back, where the hops are strained from the wort, thence to the cooling room, and on to the refrigerators and fermenting room. Here the various devices to prevent, as far as possible, contamination of the wort at this point were explained to the party, and it was stated that the fermenting room was the most complete and best fitted in England. From the fermenting room the partially fermented wort passes to the "union casks," in which the primary fermentation is completed. The "union cask" system is peculiar to Burton and its immediate neighbourhood, and it is said that the character of the beers for which Burton is famed is largely due to this system. From the "unions" the beer is run into racking-squares and thence into the casks ready for consumption or storage, as the case may be. The hop- and barm-presses and other adjuncts to the main brewing process having been inspected the party proceeded to the cooperage and to the washing-out sheds, where all casks are thoroughly washed by several processes before being used in the brewery. The very complete arrangements for testing, washing, and drying casks excited great interest among the members, and after examination of these as time would allow, the party entered railway trucks attached to one of the company's locomotives, and after passing through the entire brewery premises made a complete circuit of the town, and by passing the premises of the other Burton firms were enabled to form an idea of how completely the town is devoted to the manufacture of beer.

#### VISIT TO THE BUTTERLEY IRON WORKS.

This alternative visit was participated in by about 30 members on Thursday afternoon, July 10th.

They were conducted round the works by the manager and others of the firm, and the arrangements were such that the critical or finishing stages of several important processes could be seen to advantage by the visitors. Gaseous fuel is largely consumed in the works and is generated in a series of Wilson gas-producers. Near to and fired by these producers were several Martin-Siemens steel furnaces, and the pouring of the molten steel into moulds to form ingot-steel excited much interest. Perhaps the most beautiful sight was that of the intensely-heated molten metal in the furnaces themselves, as looked at through blue cobalt glass, for the eyes of the visitors could not endure the intense glare of white light without such aid.

A great deal of interest was also evinced in the processes of manufacture of bar and rod iron of various dimensions and grades, &c., as well as in the powerful and ingenious machinery for rolling, drawing, and other purposes. Several gigantic steam hammers were in operation, and as the evidence of enormous economy both of time and labour by the use of this apparatus was witnessed, it was difficult to regard these giants of force, as well as of skill and ingenuity, without a mingled feeling of admiration and reverence for the master mind, of which we have been so recently bereft, and who invented and brought the steam hammer to perfection, and when Watt, of Glasgow, and Stevenson, of

Tyneside, are henceforth spoken of, the association of British engineering genius will not be complete without the addition of the name of Nasmyth, of Manchester.

Every attention was shown to the visitors, and much pains taken to answer the numerous questions asked.

#### MESSES. MAXLOVE, ALLIOTT, FRYER, & CO.

The engineering works of this firm in Nottingham were visited on Thursday afternoon. The visitors were first received in the drawing office, where were exhibited a number of elaborate arrangements of the complete plants necessary for the prosecution of various industries, including the most modern sugar factories, oil mill installations, steam laundries, &c.

Arrangements of machinery for the treatment of precipitated sewage sludge were also shown, and members had an opportunity of inspecting models of the most recent type of gas engine, known as the "Cycle."

A conspicuous object on first entering the erecting shops was the Passburg grains dryer, for drying brewers' grains. A factory is now being fitted up by the firm at Burton-on-Trent, in which four of these large machines are to be placed. A number of washing machines of various sorts, including rotary steam washers, Découdan steam ironers, &c., and some large filter presses for the Wolverhampton Corporation were also shown. These latter will yield one ton of pressed cake per hour from sewage sludge after lime treatment.

In a shop adjoining several centrifugal machines of various types were to be seen in motion, the cages being directly driven by small engines attached to the iron casing or pan, the whole being either suspended from small pillars secured to the foundations, or else allowed to slide about on "slippers" on small carefully faced plates attached to the floor. By these means the inevitable vibrations caused by the great speed of the engine are reduced to a minimum.

An apparatus which deserved some attention from those engaged in chemical pursuits was next inspected. The "inspissator," as it is called, is an invention of Mr. Fryer, and with it solutions readily injured by heat can be rapidly concentrated without the necessity of boiling in vacuo. To illustrate the process to the visitors some water was rapidly evaporated by direct fire at a temperature of 120° F. This particular apparatus was of small size, capable of being worked by hand, and heated by small gas jets, being, in fact, the size suitable for laboratories.

Newhall's conveyer dryers, for the extraction of the last remnant of moisture from sugar crystals, also attracted some considerable amount of attention, more especially as the arrangement can be used in conjunction with the ordinary granulators of the Newhall or Hersey type.

#### VISIT TO THE NOTTINGHAM MANUFACTURING COMPANY'S WORKS, STATION STREET.

The party was received by Mr. Heslop, the Managing Director, and he with the assistance of Mr. Taylor, the Manager of the Manufacturing Department, and other heads of the different departments, explained very fully the various processes by which stockings and underwear are produced.

A model of the machine principally in use in the first quarter of this century was first shown; and Mr. Heslop very briefly explained how hosiery machinery was originally let out to cottagers in the surrounding district, and how the rents were received as regularly as that of the cottage they occupied, and how, as a matter of fact, hosiery machinery was heritable property, and was often transferred to the daughters of the family as a portion of their dower.

Later on, as was the case with other hand-loom when steam was introduced as a motive power, heavier and faster machinery was invented, and the manufacture of hosiery was transferred to factories in the towns, the effect of which was graphically described in the "Times" of January 19th, 1852, an extract from which Mr. Heslop produced. When hosiery machinery was let out to villagers a man would produce on the average 12 pairs per week; from this a gradual improvement of machinery developed upon the same lines until a new departure was made by Cotton's

patent of 1864, when a machine was invented which eventually revolutionised the trade, notwithstanding the prejudices of manufacturers, which prejudices were strengthened by the knowledge that this machine must render almost useless all the expensive plants in the various factories in the town. The patent was purchased by the Nottingham Manufacturing Company, who afterwards disposed of a share of it to the firm of J. and R. Morley, of London and Nottingham, and the success the two firms had with the production from this machinery obliged outsiders to seek for licenses to work the patent. Year by year improvements were made, and at this factory a man with two apprentices now makes both plain and ribbed work of similar properly fashioned, or as ladies understand in knitting, narrowed make, at the rate of 1,000 pairs per week.

After the factory the party passed through the rough stock room, and were informed that the fearful competition in the manufacture of hosiery made it somewhat difficult to satisfy the requirements of the shareholders, and that the demands of labour rendered it probable that in the future capital would have to be satisfied with a smaller return.

After a walk through the warehouse, where the various articles were finished, folded, and put up, the party visited the finishing shop, engine house, and yarn cellars, and departed much gratified by the visit.

The following works were also open for inspection:—

The Birks Co.'s Embroidery Works, Messrs. Dobson's Lace Factory, Messrs. Lambert's Lace Dressing Rooms, the Midland Lace Co.'s Frilling Factory, and Messrs. Doughty's Lace Factory.

#### ANNUAL DINNER.

At 7.30 about 150 members assembled in the Exchange Hall—most generously granted for the occasion by the Mayor and Corporation of Nottingham—for the annual dinner.

The President, Sir Lowthian Bell, Bart., F.R.S., occupied the chair, supported by the Mayor of Nottingham (Mr. Alderman Goldschmidt), Mr. E. Rider Cook (President-elect), Messrs. Ludwig Mond, David Howard, and E. K. Mnspratt (past Presidents), Alderman Sir John Turney (Chairman of the Nottingham Section), Professor Clowes, D.Sc., and many other members of Council and the local committee.

The PRESIDENT, in proposing the first toast: "The Queen, the Prince of Wales, and the rest of the Royal Family," said that the most exalted personage in the realm had so endeared herself to the hearts of her subjects, and had so entirely gained the respect of the rest of the civilised world, that he need do no more to recommend her to the affectionate consideration of the company than simply pronounce the name of Her Majesty the Queen. He had had the honour of serving under the Prince of Wales on the occasion when the object in view was the progress of that knowledge which it was the aim and duty of the Society of Chemical Industry to cultivate and advance. He was sure that it was unnecessary for him to say anything further.

The toast having been drunk with musical honours,

The PRESIDENT next gave "The Town and Industry of Nottingham." It was, he said, about 25 years since he had last visited the town, and he had endeavoured during his short stay to acquaint himself as far as possible with its claims as regarded the prosperity of its industries. He must confess that if Nottingham's claims were judged by the progress which it had made during that 25 years, no amount of prosperity which it might receive would be in excess of its deserts. He had seen enormous progress in its public monuments; streets where there were no streets before; and roads which were a pattern of excellence to all the world. He had endeavoured to account for this by the fact that the town had at its head a public spirited and intellectual municipal body watching over its interests. But that would amount to very little if the endeavours of such a body were not backed up by the intelligence and goodwill of the inhabitants.

With regard to the particular branch of science with which they were specially concerned, no words of his could better recommend Nottingham to the favourable consideration of those engaged in chemical industry than did the town's own action in promoting the cause of science by the erection and endowment of one of the best appointed colleges of science in general, as well as chemistry, that he had ever seen. Some years ago, he had paid the highest compliment to the College of Science in Nottingham that could be paid by any one man. For when at Newcastle they wanted a principal for their college, they, on his advice, sought him in Nottingham, and took him away from there; and, in the absence of his friend Professor Garnett, he might venture to say that no man had better deserved the confidence of his council and the town of Newcastle than he had. But the toast assigned to him related more particularly perhaps to the industries of Nottingham. He was sorry to learn from sources which he could not possibly have misunderstood that one of the staple trades of the town was not in that commercial position which they and the country at large would like to see it enjoy. He, however, was one of those who were far from thinking that occasional periods of depression in any branch of industry were an unmixed evil. Those were the occasions which intelligent and brave men used for the purpose of advancing that branch of industry to which they devoted their lives by a steadfast and intelligent study of its special requirements. If one looked back for a few years into the history of any particular branch of industry one was astonished at the progress which had been made. Confining himself for the moment to the iron trade, he might mention that when he first entered it they thought it very clever to make one ton of iron with four tons of coal, and to produce 70 tons of iron a week. They were now able, using a refractory ore, to make a ton of iron with half the quantity of coal once used; and instead of making only 70 tons a week, they thought themselves badly used by their furnaces if they did not make more than 70 tons a day. With respect to the lace trade and the other industries of Nottingham, he, of course, could not speak with authority. He had for the first time in his life been that day permitted to inspect a manufactory devoted to the production of that useful article called leather, and carried on under the scientific guidance of his friend Sir John Turney. He never was more agreeably impressed with a branch of industry with which he was unfamiliar than on that occasion. In visiting the Midland Railway Works under the guidance of his friend Mr. Johnson, he had enjoyed a great treat, for it happened that he was a director of a railway in the North of England, and was in charge of what was known on that line as the locomotive committee. He had found at Derby an excellent laboratory, with a skilful chemist engaged in examining with the utmost care almost every article which entered into consumption on the Midland line. His friend Mr. Johnson would be grieved beyond measure if at any time he received a report showing that the metal out of which his tyres were made contained the 1,000th part of its weight more of phosphorus than he considered it prudent to have. He confidently asked the members to join him in drinking to the return and continuation of prosperity to the town of Nottingham, and with the toast he would couple the name of his friend the Mayor.

The MAYOR, in responding, said that he and his colleagues were extremely pleased to see the Society in Nottingham, and anything they had done to make the visit a pleasant one had been done in appreciation of the science which the members pursued. That science had, he took it—excepting perhaps engineering—done more than any other to develop the commerce of the country and to contribute to the comfort of the inhabitants. The President had told him that he would go even further, and say that even engineering could not get on without the aid of chemistry, and he fully believed that the Forth Bridge could not have been erected without the assistance of that science of which the company present were the votaries. As regarded the Corporation which he had the honour of representing, the Society had seen what had been done for science in the college in which they were received. They had seen also in that beautiful temple of



art on the Castle Rock what had been done for higher culture. Here rejoiced that the Corporation excelled all others in this, that they were not satisfied with only looking after the material welfare of the town, with providing good, well-paved, clean streets and capacious sewers, but their aims went higher, and they took care of the intellectual progress of the inhabitants. With respect to the commerce to which Sir Lowthian Bell had so pathetically referred, it was quite true that for the time being one of the principal industries of the town was under a cloud. But they did not despair. They had seen similar periods of depression before, and had overcome them; and he had no doubt whatever that they would ride over the storm on which they were tossed about at the present moment, and safely arrive in a well-sheltered harbour. That particular industry was to some extent connected with chemistry, since lace, like all textile manufactures, depended a good deal upon the art of dyeing. Formerly, dyeing operations used to be done by rule of thumb; but that period had passed away and everything now depended upon science. He sincerely hoped that the visit of the Society to Nottingham would stimulate those who were engaged in dyeing, and that henceforth they would not be satisfied with merely being abreast with their foreign competitors, but by dint of science surpass them in the race.

The Town Clerk (Mr. S. G. JOHNSON), in proposing the toast of "The Society of Chemical Industry," said that he wished that the Society could have visited the town when it was at the flood of prosperity rather than in the ebb of adversity. But an old divine had said that where one would be found who could bear prosperity well, ten would be found who could bear adversity well. That saying was recalled to his memory by the President's remarks; and he quite believed that times of adversity had been the times when Nottingham men had renewed their strength and risen against adverse circumstances to new prosperity. It might, indeed, be fortunate that the Society had come at such a period, because they could commend to the members many circumstances in which they might find means to encourage them. They had there, in the centre of England, a very fine town, hardly to be surpassed in the manufacturing centres of the country; a very healthy town; a town which they had endeavoured to beautify, and in which they had done all they could for the intellectual growth of the inhabitants. They had there all the chief materials of England's prosperity: iron, coal, railway and water communication, and above all a people who were hard headed, strong handed, and willing to work with intelligence, industry, and perseverance. He trusted that some one might find the means of utilising all the material he had named, and thus make a good thing for the town and for himself. He fully endorsed what the Mayor had said respecting the value of the Society's work, and was glad to find that the members could sink all small differences and jealousies, and meet on common ground to interchange ideas and discoveries, instead of bottling them up in their own breasts. He felt that he was addressing men who had gone out to wrest from nature her great secrets, and who had struggled hard in their laboratories, for it could not be supposed that they were usually occupied as comfortably and pleasantly as he saw them at that moment. But he thought of them in their workshops, smithies, and laboratories, working day by day to acquire further knowledge for the benefit of that great humanity of which they all formed a part, and he knew that what they had done was only an earnest of what they would yet do. He need not use many words in commending a toast which practically meant themselves; but he had the pleasure of coupling with that toast the name of Mr. Rider Cook, who had been their Hon. Treasurer for 10 years, who had helped to establish the Society, and whom they had that day chosen as their next President. He was sure that under his guidance the Society would continue to prosper.

Mr. E. RIDER COOK said that he felt it a very high honour to be called upon to respond to the toast so ably proposed by the Town Clerk of Nottingham, a gentleman whose reputation was not confined to that very energetic

town, but was well known throughout England. He felt that he had a great deal more to thank Nottingham for than the mere drinking of that toast. Ever since he had set foot in the town, his mind had been full of gratitude for the princely and magnificent manner in which the Society had been entertained. When he received the papers relating to the meeting, he felt that the committee who had the matter in hand, had not only bestowed an immense amount of thought on the task of making the gathering a success, but had translated that thought into very efficient action. But when he arrived in the town and found that the University College, the Museum, the Arboretum, the Public Library, and the other institutions open to them, were all the work of the Corporation, he felt that they were going through an entirely new experience in receiving the hospitality of a municipal body, and that the example set by Nottingham was one which might well be followed by other municipalities throughout the country. He did not believe in centralisation. He believed in the people of a place managing their own affairs for themselves; and when he found that the Mayor and Corporation of Nottingham had been actuated by a desire not only to make their city beautiful and healthy, but to bring into operation those great institutions for developing the taste, thought, and education of the people, he felt sure that, though the ratepayers might have grumbled at first, they were now proud of what had been done. Moreover, the Mayor and Corporation had not confined themselves to general arrangements and the loan of rooms, &c., but had even granted the use of their plate and table requisites—a kindness which, though apparently slight in itself, called, he thought, for their especial gratitude. On behalf of the Society, he desired to thank also the various manufacturers in the neighbourhood who had so freely thrown open their works to the visitors. There had been no limitation whatever, the owners of the various works had trusted to their visitors, while learning lessons for themselves, taking no undue advantage of the privileges so freely accorded to them. He himself thought that very little harm was done in that way. What even technologists learnt in going over works similar to their own was not the particular little secrets which the owners relied upon for their own success. They learnt, rather, that general arrangement of plant, which, while it was useful to those who saw it, did no harm to those who arranged it. He would have felt it impossible to have accepted the position which their kindness had placed him in if the Society had been the Chemical Society. There was a time when he thought that he knew something about chemistry, but the rapid advance of the science had long left him behind. But the Society he was addressing was not the Chemical Society, and it was well that that should be borne in mind. Neither was it the Institute of Chemistry, a body which had to do with the conduct of practical analyses by skilled chemists. The body he was addressing was the Society of Chemical Industry, and members of that Society required not only chemical knowledge, but that knowledge which manufacturers gained in carrying on works and plant on a large scale. The name originally proposed was the Society of Chemical Engineers; and though the present title was preferred, it would be wise for them to try to ensure that their Council and the committees of their local Sections should always include, not only professional chemists and workers in pure science, but also men who had been successful in arranging plant and processes under the guidance of chemical knowledge. They should always seek to guard the Society from being regarded as a chemical society pure and simple. Chemists were wanted, but the chemists required the aid of manufacturers with knowledge and experience; and it was by these two classes of members working together that the Society could best serve the trade of the country. Under those circumstances, he hoped that his experience as a man of business, and his knowledge as a manufacturer, might enable him, to some extent, to justify the confidence which the members had placed in him in electing him to the high office of President. He thanked the worthy people of Nottingham, and especially the Mayor and his colleagues, for receiving the Society so kindly, and the present company for drinking the toast so heartily.

Sir JOHN TURNER, in giving the next toast, "Our Guests," said that he proposed the toast of "Our Guests" with great pleasure, and he felt that in doing so he had the sympathy and support of all the people of the town. They felt it a distinguished honour to have an opportunity of entertaining gentlemen who held so high a position in the scientific and manufacturing work of the whole world—men who had rendered immense services to science, and who had as manufacturers overcome difficulties which at one time seemed to be insuperable. Looking at the names connected with the Society,—Sir Henry Roscoe, Sir F. Abel, Walter Weldon, Dr. Perkin, David Howard, Professor Dewar, Ludwig Mond, Sir Lowthian Bell, and the new President, Mr. E. Rider Cook, any town might feel proud of having an opportunity to pay such men a compliment. If they had done anything to make the Society's visit enjoyable, they were amply repaid. He had great pleasure in coupling with the toast the names of Mr. W. E. Dobson, Mr. Councillor Fitzhugh, and Mr. Frank Lott.

Mr. J. E. DOBSON found it difficult after the able speeches of the Town Clerk and Sir John Turner to do more than reiterate their words of welcome, and to wish the members of the Society God-speed in their good work.

Councillor FITZHUGH had great pleasure in responding to the toast. He was present as President of the Nottingham and Notts Chemists' Association, an institution which was engaged in training youths whom, he hoped, would in due course become members either of the Society of Chemical Industry or of some kindred body. They had turned out many good men, and he hoped that they would make their mark in the world. It was their constant endeavour, as the Mayor and Town Clerk had said, to do everything in their power for the education of the town, and particularly, if he might say so, in his own department, which was concerned with chemical science. He would conclude by thanking the Society for the honour it had done the town by holding its meeting there on the present occasion.

Mr. FRANK LOTT, in responding, said that all the members present, with the exception of local men, were practically guests of the Nottingham Section, and the numbers present was the best proof they could have of the cordiality with which the invitation had been received. Speaking both as a member and as a guest he could only say that in whatever town the Society might next meet he hoped that they might receive as cordial a welcome and hospitality as full as that which they had enjoyed in Nottingham.

Mr. E. K. MUSPRATT, in rising to propose the toast of "The President," said that Sir Lowthian Bell had been connected with chemical industry from his infancy. His father was, he (Mr. Muspratt) believed, a partner with Mr. Losh, who had studied chemistry in Paris under the great Lavoisier. That carried one back to the birth of modern chemistry, the channel through which the great progress in industry had been brought about. Sir Lowthian Bell had been intimately connected therefore with that branch of applied chemistry with which he (Mr. Muspratt) was concerned, namely the alkali manufacture, though he had made his name by the application of chemical science to the iron manufacture of this country. He could remember the time when no iron works in England—perhaps in the world—possessed a chemical laboratory. Now, he presumed, there was not one without such an adjunct; and that in itself marked a great amount of progress. He thought, indeed, that he might justly say that all the progress made in the production of iron during the last 30 or 40 years was due to the application of chemistry. The address of the President showed what part he had taken in that application, and showed, too, that they owed a debt of gratitude to him for the position which the iron manufacture still retained in England. For with respect to that manufacture England had to a great extent been deprived of the supremacy which the contiguity of the raw materials used to give her. Other countries were now found to be as well provided and better situated than she was. But so long as England possessed men like Sir Lowthian Bell to lead the way in the application

of science to that industry, she need not fear competition with all the world. He had much pleasure therefore in proposing the toast of "The President," Sir Lowthian Bell.

Mr. DAVID HOWARD desired to express on behalf of the London Section an entire concurrence in the remarks made by Mr. Muspratt. They were proud to have as the President a man like Sir Lowthian Bell, the most eminent manufacturer of that product which, more than any other, tended to the prosperity not only of chemical industry but of all the industries of Great Britain.

The toast having been drunk with enthusiasm,

Sir LOWTHIAN BELL, in response, said that whatever might be their custom when they met together to discuss questions of a purely scientific character, they were bound, when they met round the hospitable board, to carefully abstain from debatable matter. Nevertheless, he could not allow the kind language used by his friends who had last spoken to pass unchallenged. He was sorry, on his own account, that the kind words which they had used were far in excess of his merits. He admitted that he had done a little—at any rate, he had done his best—to advance that industry with which he had been connected since his birth; but, to say that the iron industry of the world owed him a great debt of gratitude was much in excess of the truth. He was glad, on one account, to be permitted to say a few more words, and that was to express his entire concurrence in what had been said as to their reception. The machinery had worked so easily that they were apt to forget that any trouble had been encountered in making the arrangements for their entertainment. But everything had gone as smoothly as a well-balanced and carefully designed machine; and so they seemed to have been dwelling in a paradise of ease and comfort. He thanked Mr. Muspratt and Mr. Howard for their kind words in proposing his health, and the members of the Society for the cordial manner in which it had been received; and he hoped that those present would live to enjoy many more such happy meetings.

Mr. T. TYLER said that there was one more item to be brought forward, an item which the modesty of the hosts would not allow to appear on the official programme, and the charge of which had fallen upon him. He had had a great deal to do with the organisation of the meetings of the Society, but he had never felt prouder of it than he did that night. It had begun life well; it had gone on to better things; but on the present occasion it had excelled itself. To mention only a small matter—though business men would agree with him that it was on such matters that success often depended—he would refer to the fact that, following the usual precedent, a circular was issued with the last number of the Journal containing a list of the visits and entertainments, and with a request to members to initial those which they wished to attend. The highest type of bureau and the most systematic arrangement he could conceive of was the English railway booking office, where one was supplied with what he wanted with automatic precision no matter what the crowd or the pressure of business. But those who applied at University College on Wednesday morning were merely required to state their name and town, and were then immediately handed an envelope from a neatly arranged packet, containing everything that was necessary to convey them to the places they wished to visit, without trouble or price, and with the assurance of a hearty welcome. He said, therefore, that the Nottingham Section had excelled all that had been done before, and had set an example to other Sections which he hoped might be followed, but which could scarcely be surpassed. To whom did they owe all this? They owed it in part to the loyal co-operation of the Burton members of the Birmingham Section, but chiefly to the organising committee of the Nottingham Section, Sir John Turner, Professor Clowes, Mr. Archbutt, Mr. Lloyd Whiteley, Mr. S. V. Holgate, and the late Secretary, Mr. Ashwell, and to the fact that these gentlemen had secured the invaluable co-operation of the Mayor. He had the greatest pleasure, therefore, in proposing the health of the members of the



Nottingham Committee, coupled with the names of Professor Clowes, Mr. Archbutt, and Mr. Lloyd Whiteley.

The toast having been cordially drunk,

Professor CLOWES said that he had been taken by surprise by Mr. Tyrer, and scarcely knew how to thank him for his kind words respecting the Nottingham Section. It was, he thought, very fortunate that the Society of Chemical Industry ventured to "perform out of London," and did not, like a certain other Society, selfishly confine itself to the metropolis. It was an advantage to the Society at large and to the local Sections that it should be peripatetic, at least in its annual meetings. They had been profanely called "the industrious chemists," a title to which they were not likely to demur. The name certainly fitted the committee which had been working with him for months to provide entertainment for their visitors. They had been industrious, and their industry had met with its hoped-for reward.

Mr. L. ARCHBUTT wished to thank the company heartily on behalf of the Nottingham Section for the cordial manner in which their efforts had been received. There had undoubtedly been a great deal of time and labour expended; but it had been amply repaid by the success of the meeting and the numerous expressions of appreciation which had fallen from the various speakers that evening. He was only expressing the feelings of the committee when he said that they would not have succeeded as they had if it had not been for the kind co-operation of the Mayor of Nottingham and their friends of Burton. This was their first experience, and he looked forward with pleasure to the next visit of the Society after it had gone the round of the other Sections.

In the intervals between the toasts a selection of songs and instrumental pieces was admirably rendered by Madame Fannie Lynn, Mr. Bingley Shaw, Mr. Charles Young, Mr. Alfred R. Watson, and a select band under the direction of the latter gentleman.

### THIRD DAY.

On Friday morning a party of about 140 members left the Great Northern station at Nottingham, at 8.30, by special train for Worksop. The weather was, unfortunately, cold, damp, and gloomy, but little rain actually fell. This was of some importance, as at Worksop some dozen or more open breaks met the party—half of whom proceeded to Welbeck Abbey, the seat of the Duke of Portland, and the remainder to Clumber, the seat of the Duke of Newcastle. Both parties then proceeded to Edwinstowe, where an excellent lunch was provided by the Section. After lunch the Welbeck party returned *via* Clumber, and the Clumber party *via* Welbeck, reaching Worksop at 6 o'clock and Nottingham at half-past eight, after a very enjoyable day.

## London Section.

CHEMICAL SOCIETY'S ROOMS, BURLINGTON HOUSE.

*Chairman:* T. Tyrer.

*Committee:*

W. Crowder.	B. E. R. Newlands.
J. Dewar.	B. Redwood.
A. G. Green.	John Spiller.
S. Hall.	W. S. Squire.
C. W. Heaton.	Wm. Thorp.
D. Howard.	T. E. Thorpe.
C. C. Hutchinson.	C. R. Alder Wright.
R. Messel.	

*Hon. Local Sec. and Treasurer:*

T. W. B. Mumford, 1, Glendale Villas, Sylvan Road, Wanstead, E.

## Liverpool Section.

UNIVERSITY COLLEGE, BROWNLOW STREET.

*Chairman:* A. Norman Tate.

*Vice-Chairman:* F. Hurter.

*Committee:*

J. Campbell Brown.	C. L. Higgins.
H. Brunner.	A. H. Knight.
T. Fletcher.	D. McKechnie.
H. Gaskell, jun.	F. K. Muspratt.
F. Gossage.	Henry Tate.
W. D. Heriman.	A. Watt.

*Hon. Treasurer:* W. P. Thompson.

*Hon. Local Secretary:*

Dr. Chas. A. Kohn, University College, Liverpool.

Notices of Papers and Communications for the Meetings to be sent to the Local Secretary.

## Newcastle Section.

*Chairman:* John Pattinson.

*Vice-Chairman:* T. W. Stuart.

*Committee:*

A. Allhusen.	T. W. Lovibond.
P. P. Bedson.	H. R. Procter.
G. T. France.	B. S. Procter.
G. Gatheral.	W. W. Procter.
C. H. Hills.	W. L. Remondson.
T. W. Hogg.	J. E. Stead.

*Hon. Local Secretary and Treasurer:*

Dr. J. T. Dunn, The School, Gateshead.

Notices of Papers and Communications for the Meetings to be sent to the Local Secretary.

## Manchester Section.

*Chairman:* Ivan Levinstein.  
*Vice-Chairman:* Edw. Schunck.

*Committee:*

J. Angell.	J. Grossmann.
G. H. Bailey.	P. Hart.
R. F. Carpenter.	A. Liebmann.
G. E. Davis.	Sir H. E. Roscoe, M.P.
H. Grimsshaw.	C. Truby.
Harold B. Dixon.	D. Watson.

*Hon. Local Secretary:*

J. Carter Bell,  
Bank House, The Cliff, Higher Broughton, Manchester.

Notices of Papers and Communications for the Meetings to be sent to the Local Secretary.

## Nottingham Section.

UNIVERSITY COLLEGE, NOTTINGHAM.

*Chairman:* Sir John Turney.  
*Vice-Chairman:* L. Archbutt.

*Committee:*

Jno. R. Ashwell.	J. M. C. Paton.
F. Clowes.	S. J. Pentecost.
J. B. Coleman.	C. Spackman.
W. A. Curry.	H. J. Staples.
H. Forth.	J. T. Wood.
E. Francis.	

*Treasurer:* S. V. Holgate.

*Hon. Local Secretary:*

R. L. Whiteley, University College, Nottingham.

Notices of Papers and Communications for the Meetings to be sent to the Local Secretary.

## Glasgow and Scottish Section.

*Chairman:* E. C. C. Stanford.  
*Vice-Chairman:* A. Crum Brown.

*Committee:*

G. Beilby.	J. Falconer King.
Wm. Boyd.	G. McRoberts.
J. Christie.	T. P. Miller.
W. J. A. Donald.	E. J. Mills.
D. B. Dott.	T. L. Patterson.
C. A. Fawsitt.	J. Pattison.
Wm. Foulis.	E. Smith.
J. Gibson.	R. R. Tatlock.

*Hon. Treasurer:* W. J. Chrystal.

*Hon. Local Secretary:*

Dr. G. G. Henderson, Chemical Laboratory, University of Glasgow.

Notices of Papers and Communications for the Meetings to be sent to the Local Secretary.

## Communications.

### ON THE ACCURACY OF THE GREASE SPOT PHOTOMETER FOR MEASURING THE DENSITY OF PHOTOGRAPHIC PLATES, AND A NOTE ON THE SECTOR PHOTOMETER.

BY CAPTAIN W. M. W. ABNEY, C.B., R.E., D.C.L., F.R.S.

THE publication of a paper by Messrs. Hurter and Driffield in the Journal of Chemical Industry, on "Photo-Chemical Investigation, and a New Method of Determination of the Sensitiveness of Photographic Plates" has induced me to offer a few criticisms on the subject.

I have two reasons for doing this; one is that I have written a good deal regarding part of it, and the other that some doubt is thrown in the paper on the accuracy of the results I have obtained, owing, as it is asserted, to the photometer I use being faulty.

I should have been unwilling to enter into a criticism of the paper now the session is over were it not that it will no doubt be quoted largely at home and abroad as giving an authoritative exposition of the law of density, and that no opportunity of having a say in the matter would occur before November.

We may take the paper as practically divided into four parts: Definitions of terms employed; instrument for measuring and method of measuring the density of deposit in photographic plates; methods of producing density; and a mathematical investigation of the subject.

Under the first heading I may have one or two criticisms to make subsequently—such as on the law of absorption applied to a film in which opaque particles are embedded—but for the present I wish to confine myself to the second division, viz., the instrument and the mode of measuring densities, for if these are in fault, the measured densities and the application of theory to them will require revision.

To begin with, the instrument itself as used by the authors. There is a statement made regarding the method of photometry employed to which I must take exception, and that is that the Bunsen disc is more sensitive than the (Rumford) method of shadows. The leading authorities on photometry in London, viz., the Gas Referees, have adopted the shadow test, and certainly the measurements made by Mr. Vernon Harecourt are not wanting in delicacy. The well-known drawback to the grease spot method is the necessity of excluding all reflected light from the grease spot, and when it is recollected that the blackest lamp-black reflects more than 2 per cent. of white light, the exclusion of all such reflected light becomes a problem very hard to solve, more especially in an instrument  $12 \times 4 \times 6$  in. such as is used by Messrs. Hurter and Driffield. I am not saying that they have not overcome this difficulty, but I bring forward a possible objection to this instrument. This may be passed over, however; but unless I am very much mistaken a much more serious, if not fatal, objection must be taken to it when measuring the density of photographic deposits, and is one I have long been practically acquainted with, and which I have again investigated recently. Let any one, after focussing a view, replace the ground glass of a camera by a photographic negative, and place his head under the focussing cloth. He will see two objects, the lens through the plate, and the view, more or less dim, on the negative itself. In other words, the film acts like ground glass and is partially translucent. Now if the negative be replaced by a glass of apparently the same darkness in which the colouring matter is part of the glass itself, the view is no longer visible, or, if visible at all, very faintly so, but the lens will be clearly seen. Now ground glass, if illuminated, becomes practically a source of light, the light being scattered in every direction, but it naturally illuminates more strongly in the direction from which the original light falls on it, if the glass be perpendicular to that direction. Thus, when a piece of ground glass is placed at the end of a narrow tube which is inserted in the walls of a dark, and black

painted, room (the ground surface being inside the room), it will be found that if a beam of light be sent through the tube, not only is the room illuminated exactly opposite the tube, as it would be were the ground glass removed, but also at every angle with the axis of the tube. The illumination of a white surface gave the following results in one case, the surface being always at the same distance from the aperture.

TABLE I.

Angle from the Axis.	Relative Illumination.
0°	100
10	64
20	21
30	14
40	8.2
50	7.2
60	6.0

Evidently the amount of scattered light would here have to be taken into account, as, if not, it would be immaterial if the ground glass were in front of the tube or not, except for the small amount of light which would be reflected back towards the source of light; and further, the brightness of the light which illuminated the grease spot would not vary exactly inversely as the square of the distance since the light is not symmetrically scattered, if we may use such a phrase.

If the ground glass in the above experiment be replaced by a piece of a negative the same scattering of light will be readily seen by holding a piece of white card about 1 ft. from the orifice.

Now let us see how the results obtained by the instrument under consideration would be affected by the "ground glass" effect of the negative. Suppose we have two lights properly enclosed in a darkened room, one stationary with an aperture in front of it, the flame being of such dimensions that it more than fills the aperture (in fact, just as the authors arrange both their lights) and the other moveable. Let a moveable Bunsen disc be placed at some fixed distance from the aperture, and the other light be moved till equality of illumination of the grease spot is secured, and let the distances of the aperture and of the moveable light from the grease spot be noted. Next, let the Bunsen disc be moved to a different distance from the aperture and the moveable light be again moved till equality of illumination is secured, and let the two distances be again measured. The distances of the aperture and of the moveable light from the grease spot in the first case should be proportional to the two distances in the second case. Suppose, in the first instance, the distance of the aperture from the grease spot was 6 in. and that of the moveable light 3 ft., then if the grease spot be moved to 12 in. from the aperture the distance of the moveable light should be 6 ft., and so on. Now if the measurement of density of a plate by the grease spot method be correct when a film is placed in contact with the aperture, the same should hold good, but if the scattering of light by the film affects the result it should not. To test this I have made a good many very careful experiments, and I cite one here, which is a sample of the results obtained in all cases. The plan adopted was to make measures of a lighted aperture as stated, and then without any alteration of the general arrangement to place a portion of a plate with a photographic deposit upon it against the aperture, and measure the light coming through it by placing the grease spot at different distances from the aperture, and then to get equality of grease spot illumination by moving the second light. Table II. gives the measures of the unmasked aperture, which was about  $\frac{1}{4}$  in. in diameter as taken.

TABLE II.

Distances of Aperture from Grease Spot.	Distance of Moveable Light from Grease Spot.		
	Readings.	Mean.	Adopted.
Cm. 3.5	1.3, 75, 1.2	4	4
7	8, 8, 8,	8	8
14	16, 16	16	16
28	31½, 31½, 32	31.7	32
42	47½, 48½, 48½	48.2	48
56	63, 63, 63	63.3	64
70	80, 78, 79, 79	79	80

The readings are sufficiently close to show that for naked lights the results are concordant. Table III. gives the measures of the light passing through a negative, and at different distances reaching the grease spot.

TABLE III.

Distance of Aperture from Grease Spot Negative in Front.	Distance of Moveable Light from Grease Spot.			Ratio of Distance of Aperture to Moveable Light from Grease Spot.
	Readings.	Mean.	Adopted.	
Cm. 3	42½, 42, 42½	42.7	42.7	14.2
4	48, 49, 49	48.7	48.7	12.2
5	57, 58, 56½	57.2	57.2	11.4
6	63, 62, 64	63	63	10.5
8	84, 85, 84	84.3	84.3	10.5
9	93, 95, 95	93.7	93.7	10.4
10	102, 99, 100	100.3	100.3	10.03

If we critically examine this table we shall at once notice that large discrepancies appear. For instance, at 6 in the first 1st column, the distance of the moveable lamp should be double that at 3, or should be 85.4, but it is only 63; so at 8, if it were double of that at 4, it should be 97.4, whereas it is 84.3; at 10 it should double that at 5, or 114.4, whereas it is 100.3. The last column gives the ratios of the distances.

The squares of the ratios of the first and last measures, viz., at 3 and 10, the distances of the aperture from the source, are as 201.6 to 100.6, or nearly as 2 to 1. The nearer distance of the screen to the aperture therefore gives a lower value of the transmitted light than the further one. The question is which is, if either is, correct, and looking at it from a theoretical point of view, it appears that neither is. According to these two measures the light transmitted through the negative was about  $\frac{1}{1.5}$ th and  $\frac{1}{2.5}$ th, whereas by a correct means of measurement the amount transmitted was found close upon  $\frac{1}{1.5}$ th.

The trial of their instrument by the authors with Indian ink and indigo solutions is totally different to its trial with films. In a minor degree Indian ink scatters, but the indigo solution is transparent. Now, from the published description\* of the instrument as used by Messrs. Hurter

\* A rough description of their instrument is as follows:—A box 12 in. x 6 in. x 4 in. with an aperture of small dimensions at each end. A broad light behind each aperture so that all distances to the disc carrying the grease spot can be measured from the apertures. The grease spot disc is in a box two inches cube, travelling inside the bigger tube, apertures being cut to allow the light to pass. In front of one aperture the negative whose density has to be measured is placed, and equality of illumination is secured by cutting off light from the other aperture and by moving the grease-spot disc.

and Driffield it is probably impossible for the grease spot to be nearer to the aperture than  $1\frac{1}{2}$  in., or about 3 cm., seeing that the inner box is 2 in. cube, and that we have to add for the thickness of the glass of the negative and the thickness of the box itself. We may take it that the distance of the grease spot in the measures made varies between 6 in. and  $1\frac{1}{2}$  (15 and 3 cm.) inches, and in this range there can be a remarkable variation of densities to be obtained from the different readings. It may be said that the measures obtained are concordant one with another. This may be so, since it will be seen from Table III. that after a certain range is passed the ratios of the two distances become almost the same, but they will not be true measures of the light transmitted. Taking all things into account, it seems not unreasonable to conclude that the measures of light given as transmitted through deposits are too small; and as has been shown, when the densities are great, and the grease spot has to be moved close to the aperture, the variation may be as much as 100 per cent. less than would have been obtained if a longer box had been employed and the disc were further from the aperture.

In this part of the paper to which I am confining myself, the authors allude to the photometer which I use, and which works on the principle of cutting off light by means of rotating sectors of varying apertures, but a remark is made which more than suggests a want of accuracy in its measure. The authors say that with it the density measured "rises to over 100 per cent. (of error) with plates of high densities, which renders it utterly untrustworthy." From the formula of light transmitted through my photometer given by the authors the 100 per cent. evidently means making the density too great. In taking exception to the results obtained by their grease spot method, I have indicated experiments which anyone interested can try, and I shall now give some made with my own photometer. I might, however, stop here and use an "*argumentum ad hominem*" which would be thus. The grease spot photometer having been proved capable of giving results *inter se* 100 per cent. in error, and mine having in the same hands given results 100 per cent. in error when compared with these, may it not be assumed that my photometer gives correct results, or at all events more nearly correct results than the grease spot method gives? Setting this kind of argument on one side I will for a brief space examine the statements that have been made regarding my photometer. They say the light transmitted is more correctly represented by  $I_x = I \cdot \frac{\phi}{360} + C$

(where  $I_x$  is the light transmitted,  $I$  the light falling on the sector,  $\phi$  the aperture of the sectors, and  $C$  a constant), than by  $I_x = I \cdot \frac{\phi}{360}$ , which I have always taken as the true transmission. Their formula of course, if pushed to extremes, results in an absurdity, for if the aperture be  $0^\circ$ , that is if the sectors be closed, still  $C$  light will be transmitted. The reason assigned for adopting this formula is said to "be due to the semi-shadow on both edges of the sector openings." Evidently then, if we increase the number of sector openings, keeping the total aperture the same, the magnitude of  $C$  will increase. If with 2 sectors it is  $C$  then with 4 sectors it would be  $2C$ , with 8 sectors  $4C$ , and so on, till we arrive at a point when  $nC = I$ , which is the total intensity, leaving no light to pass through the apertures.

Had the formula been  $I_x = (I \cdot \frac{\phi}{360} - C)$  there might have been something to say in its favour, for if the light be close to the sectors the thickness of the metal of which the sectors are made might possibly cut off a fraction of a degree, but but as I never use the photometer in such a manner any error caused by this is quite negligible, more especially as the photometer, for other reasons, is rarely read below  $7^\circ$  or  $8^\circ$ , the light being adjusted to make higher readings when low readings with one position of light would be obtained.

I may as well here give some experimental results of the correctness of the photometer.

Two incandescence lights approximately similar in dimensions were used and kept glowing with a constant current. One lamp was fixed at a distance of about 18 inches from the screen, and the other placed on a block sliding on a bar,

and the illuminated shadows cast by a rod fell on a white patch and were equalised in brightness. The rotating sectors were introduced between the first light and the screen and were fixed at different apertures. Equality of illumination was obtained by sliding one light along the bar.

The following are the results:—

TABLE IV.

Sectors set at	Mean Reading of the Distance of the Movable Lamp.	Calculated Value.
°		°
180	16.25	180
90	23	90
45	32	46.5
20.5	48.5	20.2
10	69	10
4.8	101	4.7

In the next two tables we have a large luminous source, viz., an Argand burner  $\frac{3}{4}$  in. in diameter.

The first shows the worst measures made, and the second, one usually obtained.

TABLE V.

Sector Set at	Mean Readings of Lamp from Source.	Calculated Intensity.
°		
180	13.75	176.8
88	19.5	88
60	23.5	60.5
45	27.25	44.5
30	33	30.8
10	59	9.6
5	83	4.8

TABLE VI.

Sector Set at	Mean Reading of Lamp from Source.	Calculated Intensity.
°		
180	13.25	180
90	18.75	90.3
44.5	24.75	44.6
10	57.25	9.6
5.2	79.0	5.1
3	106.5	2.8

Equally good results were obtained when the sectors were placed in the beam of light which was focussed on the screen by means of a lens.

In the foregoing remarks I have given my criticisms on the accuracy of the grease spot photometer for measuring densities, and it has seemed to me that the whole paper more or less hangs on this. I have also endeavoured to vindicate the sector photometer from the slur which has been placed upon it. All the experiments I have given on both subjects are easy to repeat, and I trust that some independent physicist will take the matter up, and judge between myself and the authors.

It would be premature to go into the proofs which can be adduced that the method employed by myself for measuring the densities is correct. If necessary, such proof can be given. Doubt has only been thrown upon the instrument I use, and not on the manner of using it in its latest development. My admiration of the paper is not diminished by any fault I have found in the methods employed by Messrs Hurter and Driffield, but I thought it right, in the interests of photographic science, to point out what I believe to be a weak spot in the proofs of their theory.

## REPLY TO THE PRECEDING COMMUNICATION OF CAPTAIN ABNEY "ON THE ACCURACY OF THE GREASE SPOT PHOTOMETER, Etc.\*"

BY FERDINAND HURTER, F.R.S., AND V. C. DRIFFIELD.

CAPTAIN ABNEY has conferred a great honour upon us by his kind criticism of part of our paper.

The objections which Captain Abney brings against our photometer are two. In the first place he objects to the light reflected from the walls of the blackened large chamber as likely to interfere with the readings of our instrument. But we placed the photometer disc within a second chamber for this very reason, and the light reflected from the walls of the second chamber are equal on both sides when the disc is at point of equality.

The second objection is of greater importance. The phenomenon of "scattering" represents one of the great difficulties to contend with. It is more readily observed with white opaque substances, such as ground glass or emulsions, but it also undoubtedly exists, though to a small extent, in negatives. Fortunately, it interferes only in plates of very high densities.

The experimental results given by Captain Abney agree fairly with our own, though they are rather worse. We have, however, guarded against serious errors by showing in Table I. the extreme limits to which we use the instrument. It will there be seen that the last figure we use is 1.7, corresponding to  $l \times 0.75$ , which in our instrument means a distance of 1.5 inches between the Bunsen disc and the plate to be measured. If we accept Captain Abney's results, given in Table III., as correct, the relative densities shown for this distance of 4 cm., and one of 10 cm., would be proportional to the logarithms of the numbers 12.2 and 10.03 respectively, i.e., to 1.086 and 1.00, and the error made in this extreme case would be 8 per cent. only, and not 100 per cent., as stated by Captain Abney. But it is very seldom indeed that the disc has to be moved so close to the plate as is assumed in this example, and we have clearly and carefully stated that we do not claim any greater accuracy than 5 per cent.

With regard to the angle subtended by the spot of the Bunsen disc and the diameter of the diaphragm, it is even in this extreme case less than  $10^\circ$ .

Respecting the objection we made to the revolving sector, we asserted that the light transmitted by the sector was not exactly the half or the quarter of that which the lamp gives directly, when the sector is open to  $180^\circ$  and to  $90^\circ$  respectively, and the experiments quoted by Captain Abney are no answer to this objection. We also stated that the error was small with plates of low density. The formula we gave was the result of experiment and not of any theoretical considerations.

Like other empirical formulæ it has the property of leading to absurdities when pushed to extremes. We may have occasion to quote an important formula published by

Captain Abney which has the same disagreeable property, but it is interesting perhaps to state that we assured ourselves of the existence of the error by the very experiment Captain Abney mentions, viz., by multiplying the sector openings.

We have to apologise to Captain Abney for the omission in our paper of an important statement which was made when the paper was read, viz., that this error does not in any way affect the results of his researches on the sensitiveness of the silver compounds to the various parts of the spectrum. We thank Captain Abney for his expression of admiration of our paper, and we can assure him that we do not undervalue or wish to detract from the importance of his classical researches.

## Journal and Patent\* Literature.

### I.—GENERAL PLANT, APPARATUS, AND MACHINERY.

*Tannin Matters as Anti-incrustators for Steam Boilers.*

L. Vignon. Bull. Soc. Chim. 1890, 3, 410—413.

See under XXIII., page 767.

#### PATENTS.

*Improvements in or relating to Glass-lined Fittings for Glass-lined Tubing.* D. Rylands, J. Crowder, and R. Morant, Barnsley. Eng. Pat. 6750, April 20, 1889. 8d.

This specification describes the operation of lining pipe fittings with glass and gives examples of lining "tees" and elbows. A "tee" is lined by introducing a piece of tubing through the head and suitably holding it while a hole is made in the side in the ordinary way, by means of a blow-pipe flame through the side piece. Another piece of glass tube may then be welded on. The space between the glass tee and the metal tee is packed with pitch, cement, &c. Modifications are described.—O. H.

*Improvements in and connected with Filters for Water and other Liquors.* R. Bruce, C. C. M. Gibson, and E. D. Reeve, London. Eng. Pat. 8265, May 17, 1889. 1s. 3d.

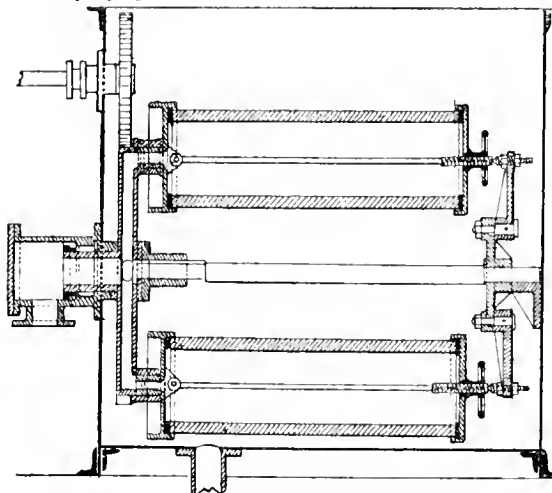
THE inventors make their filter in the shape of a porous tubular vessel, closed at both ends, which they insert either vertically or horizontally in a tank containing the water to be filtered. One end is closed with a cap, the other screwed to a plate which has a central exit passage through which the filtered water is carried away, passing through the side or bottom of the tank by means of a stuffing-box. The plate on which the filtering tube is secured can be made to rotate on its central outlet for the purpose of allowing the outer face of the tube to be cleaned either by a jet of water or steam, or in any other manner. Several filtering tubes may be set on to one rotating plate, and such an arrange-

\* Any of these specifications may be obtained by post, by remitting the cost price, plus postage, to Mr. H. Reader Luck, Comptroller of the Patent Office, Southampton Buildings, Chancery Lane, London, W.C. The amount of postage may be calculated as follows:—

If the price does not exceed 8d. ....	3d.
Above 8d., and not exceeding 1s. 6d. ....	1d.
" 1s. 6d., " " 2s. 4d. ....	1½d.
" 2s. 4d., " " 3s. 4d. ....	2d.

\* These communications, though strictly not within the scope of the Journal of the Society, have been inserted on account of the interest of the subject, but in accordance with a resolution of the Publication Committee, no communications of a controversial nature, apart from reports of discussions of papers in open meetings of the Sections of the Society, will in future be inserted.—ED.

ment, with horizontal tubes, is shown in section in the accompanying illustration.

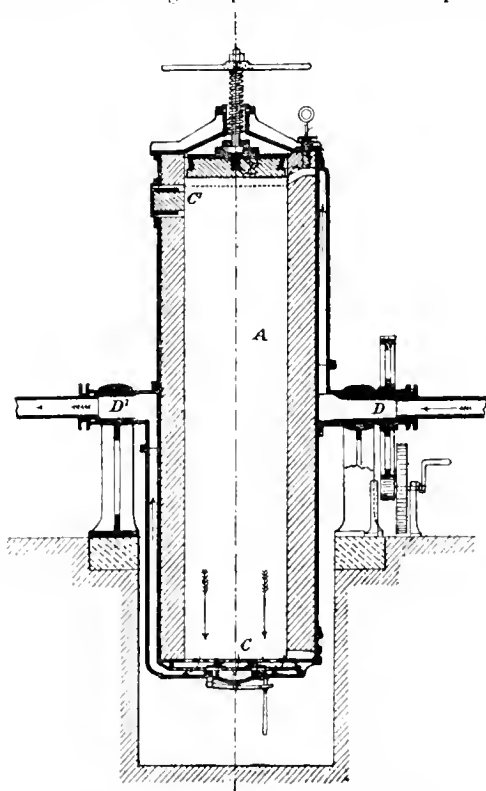


FILTER FOR WATER OR OTHER LIQUID.

In this arrangement of the apparatus, if the rotating gear be temporarily stopped and the water in the tank lowered below the level of the upper tube or tubes, these can be removed, cleaned, and replaced without arresting the action of the other tubes. Six sheets of drawings are attached to the specification, and there are 13 claims.—B.

*Apparatus for the Methodical and Continuous Treatment of Pulverulent Matters by Fluids.* W. L. Wise, London. From Solvay and Co., Brussels, Belgium. Eng. Pat. 8872, May 28, 1889. 8d.

THE treatment of solids by gases has always presented difficulties when the gases pass in from below upwards.



TREATMENT OF PULVERULENT MATTERS BY GASES, &c.

Channels are apt to be formed through which the main body of the current of gas passes, there being thus an excess in the one place and a deficiency in the other. Consequently numerous efforts have been made to overcome these disadvantages by causing the gas to travel in the opposite direction, *i.e.*, from above downwards. Any channels are now kept closed by the action of gravity and by the current of gas itself, the whole reaction being thus uniform. In this invention the vessel A is cupola-shaped, and carried upon hollow trunnions. The gas enters at D, passing downwards through A and issuing at D¹. When the reaction in the upper layer or layers is completed the cupola is turned through half a revolution and a given amount allowed to fall out through C¹, which of course is now lowermost, a fresh charge being introduced at C. If necessary to avoid shaking the material in the vessel A when discharging, a false bottom may be introduced and worked from outside, as shown at E. The apparatus is turned back to its old position and the operation begins again *de novo*. The charges therefore work by gradual stages through the cupola.—S. G. R.

*Improvements in Apparatus for Treating Metallic Ores or Compounds with Chemicals.* S. Alley, Polmadie. Eng. Pat. 9592, June 11, 1889. 8d.

THIS apparatus consists of a metallic vessel coated inside and out with vulcanised rubber and capable of being rotated on hollow trunnions. At one end is a valve opening inwards, kept closed by means of the elasticity of rubber rings placed on its spindle. The valve coincides above with a hopper for charging, and below with a vessel provided with a filtering bed, into which the contents of the cylinder are discharged. The vessel is provided with a flexible cover of rubber. The cover has a ring-shaped opening in its centre, which can be pressed in contact with the above-mentioned valve, thereby forming an air-tight joint when discharging.—H. K. T.

*New or Improved Method of Distilling Products by Means of Steam, and Classifying or Separating them according to their respective Points of Vaporisation, and Apparatus therefor.* W. P. Thompson, Liverpool. From N. Notkin, Moscow, Russia, and P. Marix, Paris, France. Eng. Pat. 10,907, July 6, 1889. 6d.

THE method of distillation recommended in this patent could not well be made clear without transcribing the specification in full, to which readers are referred. There are no drawings attached to illustrate the arrangement of the apparatus, and the claims recorded are the following, *viz.*:—“(1.) A process of continuous distillation and classification of distilled products by means of steam used (a) indirectly (*viz.*, without mixing it) to carry those to a temperature near the point of vaporisation of the more volatile parts, and then (b) directly by acting successively on small parts at a time to produce distillation and classification of the products that distil, substantially as described.

“(2.) In the above process of continuous distillation and classification of products, the special manner or method whereby the steam acts by a direct way consisting in mixing the steam and substance in stills separated from the supply vat, and thus by proceeding either by pulverisation (single or repeated), or by single or repeated steam clearing and at temperatures conveniently increasing from one still to the other, to obtain the classification of the distillation substantially as described.”—B.

*Improvements in Filtering Apparatus.* H. J. E. Jensen and E. F. G. Busch, Hamburg, Germany. Eng. Pat. 10,921, July 6, 1889. 8d.

THE improvements contemplated are more particularly applicable to filters constructed under a previous patent, Eng. Pat. 5669 of 1886 (this Journal, 1887, 364). They refer to details of construction and to methods of cleaning of that class of filters which is composed of a number of hollow porous drums or discs secured to a hollow rotating

shaft and inserted in a tank containing the unfiltered liquid, the drums acting as filters and the shaft serving to conduct the filtered liquid away.

The improvements consist in perforated tubes which are fitted between adjacent drums, and admit of streams of water impinging against the surfaces of the rotating drums at acute angles, for the purpose of carrying away the accumulated impurities. Or rotary brushes through the circumferences of which currents of water are issuing are fitted into the spaces between the drums with the same object in view. There are two sheets of drawings and 10 claims.—B.

*Improvements in Apparatus for Evaporating, Heating, or Cooling Liquids.* R. C. Garton, C. H. Garton, and W. Lawrence, London. Eng. Pat. 11,350, July 15, 1889. 8d.

THE invention relates to apparatus in which the process is carried on under ordinary atmospheric pressure. Vertical (or nearly vertical) tubes made of any suitable material are fluted, grooved, twisted, or otherwise formed so as to increase their external and internal surfaces. The diameter of the tubes, and the angles at which they are twisted, depend upon the nature of the liquid to be acted upon. Steam is admitted to one side of the tube, either externally or internally, as desired, and the liquid to be treated flows down the reverse side in a thin film; or by jacketing the tubes a double effect can be obtained. The invention is stated to be of "special utility in the evaporation of already partly concentrated liquors in chemical and other manufactures when great rapidity of concentration is desirable in the finishing process, and particularly in the evaporation of viscous substances, and the crystallisation of salts."—E. S.

*Improvements in the Crystallisation of Salts or Crystallisable Acids.* R. C. Garton, C. H. Garton, and W. Lawrence, London. Eng. Pat. 11,351, July 15, 1889. 8d.

SOLUTIONS are usually crystallised in open pans. The crystals formed are found at the bottom, and thus impede the evaporation of the rest of the solution. Further boiling is apt to decompose and alter the constitution of the crystals already produced. The patentees use tubes grooved either longitudinally, spirally, or laterally, and heated by hot air, steam, &c. The liquid to be concentrated may either be caused to flow on the outside or inside of the tubes, the heating agent passing along the opposite side, jacketing being employed if necessary. The grooves may run spirally round the tubes, and are cut of different pitches. With a very viscous solution the grooves may be made at an angle of 45°, in other cases at a slower pitch. The diameter of the tubes may vary, from 1½ to 5 in. being recommended both for inside and outside flow, but in the former case the indentations should be deeper. The tubes may be made of metal, glass, porcelain, earthenware, or of other materials. (See preceding abstract.)—S. G. R.

*Apparatus for Purifying Water for Economisers.* G. W. Allen and H. J. A. Bowers, Manchester. Eng. Pat. 11,595, July 20, 1889. 6d.

THIS invention consists chiefly in improvements in Eng. Pat. 7772 of 1887 (this Journal, 1888, 313) so as to apply the same advantageously to an economiser. For this purpose, "collectors" are used instead of the "scum plate" described in the former patent. These are placed in the bottom boxes of the economiser to collect the impurities deposited by the feed water. Each collector is preferably a slotted casting of an inverted V shape in cross section, and communicates, by means of a flow-pipe, with an external "separator." The return pipe from the separator is connected to the bottom boxes of the economiser, and terminates in a nozzle on the injector principle in order to ensure the circulation of water in the proper direction.—E. S.

*Improvements in and connected with Apparatus for Drying Granular and like Materials.* S. Seckendorf, London. From M. Reuland, Dortmund, Germany. Eng. Pat. 3089, February 26, 1890. 8d.

"THE object of this invention is an apparatus for drying granular, pasty, or thick material, in which the drying material is gradually moved over an inclined heated surface by means of obliquely placed scrapers, whence it passes into troughs or additional drying receptacles." For the mode by which this is effected, the specification and the drawings accompanying it must be consulted. There are six claims.—E. S.

*Improvements in Apparatus for Purifying and Cooling Water.* G. Simpson, Philadelphia, U.S.A. Eng. Pat. 4251, March 18, 1890. 6d.

THE apparatus consists of a tank having two partitions, one forming a chamber for receiving the water, and the other a storage vessel for the purified water, the two being separated by a cold well, which communicates with an ice chamber in the upper part of the storage vessel. Outside the tank is a coil supplied with water through a pipe, and controlled by a regulating valve in the tank, the water being heated by a gas jet beneath the coil, and afterwards flowing into the tank, from which it passes through the filtering bottom of the storage vessel to the ice chamber. There are five claims.—E. S.

*Improvements in Kilns for Cement and the like.* W. A. Gibbs, Chingford. Eng. Pat. 4741, March 26, 1890. 8d.

THE inventor proposes to apply a water jacket to kilns for drying and burning cement, &c., for the purpose of heating water or raising steam.

The water jacket may either be a casting or consist of steel or iron plates, or of a solid welded coil of pipe, or of a series of vertical pipes united by junction chambers at the top and bottom. A portion of the steam obtained in this way may be used to increase the heat within the kiln by producing a forced draught of air or of air impregnated with petroleum vapour through the cement, this blast being optionally heated in a regenerator by means of the waste gases.

The waste gases are utilised for drying the cement slurry on the ground level so as to obviate the necessity for pumping it up to the top of the kiln. For this purpose, the fire is lighted at the top of the kiln, and the heated gases drawn down by a fan, and either passed into metal tubes with shallow trays containing the slurry on their upper sides, or blown over the surface of the slurry contained in shallow tunnels. It is claimed that the substitution of water jackets for the interior brick lining of cement kilns greatly reduces the cost of repairs.—S. B. A. A.

*Improvements in the Construction and Working of Double, Triple, and Multiple Effect Evaporating Apparatus.* A. Chapman and S. Vickess, jun., Liverpool. Eng. Pat. 5267, April 5, 1890. 6d.

THE improvement consists in causing the liquid to be evaporated, to flow from the pan or pans of lower pressure to those of higher pressure, and the steam or vapour to go from pans containing liquid of greater density to those having liquid of lower density. By this arrangement liquid can be concentrated to greater density than in the ordinary manner of carrying out the process. The drawing attached to the specification shows the apparatus as arranged for producing a quadruple effect. The pans shown are of the ordinary construction, but those described in Eng. Pat. 1752 of 1888 (this Journal, 1889, 128) may also be used.—E. S.



*Improvements in the Evaporation of Brine and other Solutions or Liquids, and in Apparatus employed therein.* J. C. Mewburn, London. From C. C. Peek, Warsaw, U.S.A. Eng. Pat. 6107, April 22, 1890. 1s. 1d.

THE invention is intended, primarily, for the evaporation of brine in the manufacture of salt, but it can be applied to other liquids. The principal improvements consist (1) in the position of the heating surface, (2) in the construction of the vacuum chamber, (3) in making the process continuous, (4) in heating the brine before introducing it into the vacuum chamber, and in effecting its proper circulation. For details of construction and the mode of working, the specification and eight sheets of drawings must be consulted. There are 26 claims.—E. S.

*New or Improved Preparation or Composition for Dissolving Boiler Scale, and for Preventing Formation of such Scale.* J. Effrem, Berlin, Germany. Eng. Pat. 6478, April 28, 1890. 4d.

THE composition consists of the following substances in or about the proportions mentioned, viz.:—25 parts by weight of potato starch, 25 of concentrated lye, 3 of leather glue, 47 of water.—A. J. K.

#### ERRATUM.

The larger figure at foot of page 594 is made continuous, whereas the horizontal lines should have been broken  $2\frac{1}{2}$  inches from right-hand side of figure. An erasure in a vertical line, or a strip of paper  $\frac{1}{4}$  inch wide pasted across the figure at the part indicated will produce the effect intended.

## II.—FUEL, GAS, AND LIGHT.

*Determination of Ferrocyanide in the Residues from Gas Purifiers.* R. Zoloziecki. Zeits. f. angew. Chem. 1890, 301—302.

See under XXIII., page 767.

#### PATENTS.

*Improvements in Artificial Fuel.* A. Myall, London. From J. H. Lancaster, New York, U.S.A. Eng. Pat. 8040, May 14, 1889. 6d.

"SEXTALIT" is claimed as a new fuel for domestic heating purposes, and it is alleged that it may be burnt in flueless stoves without "emitting any smoke, or noxious gases or vapours." It is prepared as follows:—

100 lb. of finely divided charcoal are re-calcined to expel all the contained gases or vapours, mixed with  $1\frac{1}{2}$  lb. of manganese dioxide, and with a solution of  $3\frac{1}{2}$  lb. of starch and  $3\frac{1}{2}$  lb. of saltpetre, moulded into blocks, &c., and dried at a gentle heat. Coke or other carbonaceous substance may be substituted for charcoal, and "acetate of lead" for the manganese peroxide.—S. B. A. A.

*Improved Burner for Alcohol, Petroleum and other Combustible Liquids.* E. H. Allain, Créteil (Seine), France. Eng. Pat. 8304, May 18, 1889. 11d.

THE heating apparatus fitted with the improved burner consists essentially of a vessel containing a central asbestos wick of "tubular form" surrounded by a layer of an absorbent incombustible substance susceptible of being heated, such as sand, asbestos or "vegetable earth." A thick wire may then be coiled around in a spiral form, and the remainder of the vessel filled with some absorbent substance which conducts heat badly. The wick is immovable and does not project beyond the top of the vessel, and both the top and bottom of the vessel are perforated concentrically with the wick; only the interior surface of the wick is thus exposed, and accordingly combustion takes place on that surface alone. To regulate or extinguish the flame a lever is arranged to raise or

lower a metallic tube moveable in the central perforation of the vessel, thus masking the interior surface of the wick to any extent required. Devices are also shown for distributing the fuel and for preventing its evaporation when the apparatus is not in use. Two modifications are given; in one of these, which is adapted for illuminating purposes, the burner consists of the small central tube, asbestos wick, and one absorbent layer, the latter being connected by a capillary feed wick with a reservoir of the combustible liquid placed beneath.—S. B. A. A.

*Improvements in Furnaces and Grates therefor, and Means for conducting Air thereto.* S. J. Miles, Chicago, U.S.A. Eng. Pat. 255, January 7, 1890. 6d.

THE furnace and appliances are designed especially for burning coal slack and other fine refuse. The combustion chamber is divided into compartments by transverse bridge walls rising to some distance above the level of the grates, there are also transverse curtain walls which hang down from the roof over the centre of each chamber (except the first) for some distance below the level of the tops of the bridge walls. The grate bars are perforated, and each compartment of the combustion chamber is provided with a practically air-tight ash pit into which a blast of air is supplied from a pipe communicating with a fan. Refuse fuel is charged on the first perforated grate and there in great part uniformly consumed; a portion is, however, forced by the blast over the bridge wall into the next compartment, there arrested by the curtain wall and deflected on to the second grate, from which likewise a portion is blown over to the next grate, and so on. An arrangement is also described for cleaning the ash pits.—S. B. A. A.

## III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

*Researches on the most Volatile Constituents of Coal Tar.* J. Biehringer. Dingl. Polyt. J. 276, 78—90 and 184—191.

IN their investigations of the most volatile portions of coal tar, authors have hitherto employed the first runnings obtained by the distillation of benzene hydrocarbons which have been subjected to a preliminary treatment with acids and alkalis. On this account a number of substances present in the crude material have escaped identification owing to their removal by this treatment, whilst new bodies formed by the action of acids and alkalis have been isolated which could not be detected in the raw material, and it is doubtlessly owing to this circumstance that conflicting statements have been published regarding the presence or absence of a number of substances in the most volatile portions of coal tar. The author therefore thought it desirable to employ unrefined benzene hydrocarbons for the purpose of this investigation, special attention being also given to the quantitative determination of the more important constituents by simple and accurate methods. At first the portion of English 90 per cent. benzene boiling between  $30^{\circ}$  and  $80^{\circ}$  was used, of which about 1.5 per cent. was obtained. Subsequently the fractions boiling between  $50^{\circ}$ — $60^{\circ}$  and  $60^{\circ}$ — $70^{\circ}$  were chosen, the necessary material being obtained from Dr. Häussermann of the Griessheim Chemical Works, and the fractions being marked L1 and L2 respectively. They form colourless mobile strongly refractive liquids (assuming a yellow colour on standing), and possess a disagreeable penetrating odour which causes headache and slight giddiness after prolonged inhalation. The taste is caustic. Blue litmus paper is turned red. They burn with a luminous smoky flame which gives off a disagreeable odour. All attempts to isolate the different constituents by fractional distillation were unsuccessful; thus the fraction L1 which gave about 66 per cent. between  $50^{\circ}$  and  $60^{\circ}$  began to boil at  $40^{\circ}$ , and left a considerable residue boiling above  $60^{\circ}$ , and although after several fractionations it was possible to gradually concentrate the carbon disulphide in the portion boiling below  $50^{\circ}$ , the

results were by no means satisfactory, as this end could only be achieved at the expense of the higher boiling portion. It would, in fact, seem as if it were not merely some peculiar property brought about by the process of fractional distillation which gives rise to this anomaly, but as if during the distillation the mixture suffered decomposition, forming new substances which considerably influence the boiling point. The author therefore tried the following methods:—He attempted to remove by some means the bodies known to be present and then searched for new substances, and in a second series of experiments he made a number of tests to detect the presence or absence of certain hydrocarbon series by means of well-known group reagents.

1. *Carbon disulphide* was first obtained by Vincent and Delachanal from the low boiling portions of unrefined benzene. Its presence completely masks the results obtained by fractional distillation, and as acids and alkalis fail to remove it commercial benzenes are almost invariably contaminated therewith. For the quantitative determination Hofmann's process was tried. This is based on the formation of a well crystallised red additive compound when carbon disulphide is brought in contact with triethylphosphine. The author used pure carbon disulphide boiling at  $46.8^{\circ}$ , but failed to obtain satisfactory results although he is unable to explain the cause of this. The numbers were invariably too low. He then tried the xanthic acid reaction. For this purpose a measured quantity of carbon disulphide or liquid containing the latter is added gradually to an alcoholic solution of potash, when potassium xanthate is formed. The amount of the latter is then estimated as cupric xanthate by adding a decinormal solution of cupric sulphate until there is a slight excess of the copper salt which is ascertained by placing a drop of the solution from time to time on a double filter paper and testing the liquid on the lower paper with potassium ferrocyanide. One cc. of the copper solution is equal to 0.0152 gm. of  $\text{CS}_2$ . The method gives good results. The following numbers were obtained:—

	Per Cent. $\text{CS}_2$
Fraction boiling between $36^{\circ}$ and $80^{\circ}$	37.30
Fraction L1	19.50
Fraction L2	19.17

The amount of potassium xanthate may also be estimated by means of a standard iodine solution. The author, however, found that with pure carbon disulphide the results were about 2.5 per cent. too low. In order to remove the carbon disulphide the following method was adopted:—From 1,000 to 1,200 grms. of first runnings are placed in a flask and treated with from 5 to 6 times the weight of alcohol, the flask and contents being kept in a cooling mixture to prevent loss by evaporation. A constant stream of dry ammonia is then introduced through a wide glass tube until the separation of solid xanthate is complete, when the mixture is filtered rapidly through a cotton filter, the filtrate being collected in a separating funnel. The lower layer contains the alcohol, some water and a small amount of hydrocarbons in solution. In order to separate the latter the mixture is diluted with water, the product thus obtained is then added to the upper layer and the whole subjected to another treatment with alcohol and ammonia. It is advisable to repeat this operation a third time in order to ensure the complete removal of all sulphur compounds. The hydrocarbon liquid is then washed thoroughly with water and dehydrated with calcium chloride. The removal of carbon disulphide reduces the specific gravity considerably; thus in the case of fraction L1 it fell from 0.8148 to 0.7401.

2. *Nitriles and Isonitriles*.—Vincent and Delachanal found acetonitrile in light benzene to the extent of from 50 to 70 per cent. Nocting detected the presence of an isonitrile, and showed that in spite of the small proportion of poisonous ingredients found in the first distillate from benzene, the working of light benzenes on a large scale may under certain conditions produce poisonous effects. He quoted a fatal case which occurred at a factory in Thann, where a workman having inhaled the vapour of

isonitrile died from its effects. For the quantitative estimation of nitriles the author used the following method:—A measured quantity of light benzene was boiled in a flask provided with a reflux condenser for 12 to 15 hours, with half its weight of concentrated hydrochloric acid. The brown mass thus obtained was evaporated to dryness, extracted with hot water, the solution filtered off, again treated with hydrochloric acid, evaporated and extracted with water. This operation was repeated until the residue was coloured only slightly yellow. It was then dissolved, treated with hydrochloric acid, evaporated to dryness in a tared dish, dried at  $100^{\circ}$  and weighed. The following results were obtained:—

	Per Cent. Nitrile.
Light benzene boiling between $36^{\circ}$ to $80^{\circ}$	7.49
Fraction L1	11.93
Fraction L2	10.03

3. *Aldehydes and Ketones*.—From the circumstance that aqueous solutions extracted from light benzene reduce ammoniacal silver solutions, the author concludes that aldehydes are contained in the first runnings from benzene. He treated a large quantity of fraction L1 with sodium bisulphite, but obtained only about 1 per cent. of extract, and was, therefore, unable to examine the mixture of aldehydes and ketones more closely. He, however, detected the presence of acetone by the characteristic odour, the formation of iodoform and the indigo reaction with orthonitrobenzaldehyde and sodium hydroxide. According to Michael a bright yellow resinous substance is obtained on boiling aldehyde with an alcoholic solution of resorcinol and a minute quantity of hydrochloric acid (see this Journal, 1884, 253). The author has obtained a similar product by applying the same reaction to light benzene.

4. *Ethyl Alcohol*.—The author has not been able to isolate this product from any of the fractions obtained by the distillation of light benzene. He, however, obtained a faint reaction for ethyl alcohol from a sample which had been in contact with water for a fortnight, and concludes from this result that light benzene contains an ethyl compound which is gradually decomposed by water with formation of ethyl alcohol.

5. *Phenols*.—The examination for phenols was conducted in the following manner. After treatment with hydrochloric acid the light benzene was shaken up with dilute soda lye. The alkaline extract was then acidified with sulphuric acid and distilled with steam or extracted with ether. In both cases a product having a tarry odour was obtained, but the quantity was too small to admit of further investigation.

6. *Mercaptans*.—To detect the presence of these compounds in light benzene the different fractions were heated with mercuric oxide and alcohol, and in a second series of trials they were added to an alcoholic solution of mercuric chloride. The presence of mercaptans could not, however, be established with certainty, as only a slight turbidity was obtained.

7. *Detection of Bodies belonging to the Pyrrol-, Thiophen-, and Furfuran Series, also of Hydrocarbons of the Benzene Series*.—The vapour from fraction L1 imparts a greenish colour to pine shavings moistened with hydrochloric acid. The reaction with phenanthraquinone and with isatin and concentrated sulphuric acid gave no colouration, so that it may be inferred with certainty that no closed chain compounds of the pyrrol, furfuran, or thiophen series are present. It was not possible to separate aromatic hydrocarbons by treatment with a concentrated solution of picric acid, although benzene was found in the residue obtained by fractional distillation.

8. *Unsaturated Compounds*.—The different fractions from the distillation of light benzene absorb bromine very eagerly, and form additive as well as substitution compounds. The group of unsaturated compounds includes derivatives of acetylene, the author having found that on adding an ammoniacal solution of cuprous chloride to light benzene and allowing the mixture to stand, a red-brown precipitate is obtained.—D. B.

*By-products of Wood Distillation.* Vldesco. Bull. Soc. Chim. 1890, 3, 510—514.

THE author has examined two samples obtained during the rectification of methyl alcohol in wood distillation. Both samples combined with sodium monosulphite, and did not reduce silver nitrate in ammoniacal solution, thus indicating the presence of higher ketones.

*Sample No. 1*, distilling 85°—140°, was washed with distilled water to remove any soluble portion. The insoluble part was treated with sodium monosulphite, with which it combined to form a solid substance, with the exception of a certain quantity of liquid, which separated uncombined. The combined portion was recovered by distillation with sodium carbonate, and fractionated, distilling chiefly at 99—102°.

The analysis, together with the fact of its combining with sodium monosulphite and not reducing ammoniacal silver nitrate, pointed to the probability of this substance consisting chiefly of one of the three isomeric ketones, with 5 atoms of carbon.

The author proved it to consist of methylpropyl ketone. The identity was established by analysis and examination of the product obtained by treatment with phosphorus pentachloride, by obtaining the corresponding alcohol, by the action of sodium amalgam, and also by obtaining the corresponding pinacone.

The uncombined portion consisted of a mixture of toluene and xylene. The portion soluble in water was methylethyl ketone, as in the following sample.

*Sample No. 2*, distilling between 70°—143°, of this, 75 per cent. dissolved in water. The insoluble proved to be identical with the foregoing insoluble part. The soluble portion was separated with potassium carbonate, and treated with sodium monosulphite, with which almost the entire quantity combined. After recovery, it was fractionated, distilling chiefly between 79°—82°, the boiling point of methylethyl ketone, with which it was identified by analysis.

As the price of these ketones in commerce is high, the author considers it would be advantageous to produce them from the above-mentioned products, in which they occur in notable quantity.—D. A. S.

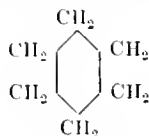
#### The Recent Investigation of Naphthenes or Polymethylenes.

V. Markovnikov. J. Russ. Chem. Soc. 22, 275—279.

SINCE the investigation of hydrocarbons from Caucasian naphtha were commenced some years ago in the author's laboratory, the nature and the object of these researches have been considerably altered. Their chief object having formerly been the study of the composition of naphtha, they are now principally directed to the general and individual properties of the new class of hydrocarbons found in naphtha, and generally termed polymethylenes or naphthenes, in order to determine the relationship to other classes of organic compounds. The investigation of nononaphthene  $C_9H_{18}$  and its derivatives, by Kononov (J. Russ. Chem. Soc. 22, 118—148) is the first of the new researches, while several others of the same nature are being carried on in the author's laboratory.

However, the author finds it even now possible to make several generalisations concerning this class of compounds.

(1.) The identity of hexanaphthenes, hexahydrobenzenes, and hexamethylenes, long ago foreseen by theory, may be now considered almost proved by facts—



represents the only possible construction for all three. Although these simplest members of the series have not been studied yet in the pure state, the identity of some of their derivatives is proved beyond doubt.

Octonaphthene is hexahydrometaxylene; iso-octonaphthene is but the para derivative of the same aromatic

hydrocarbon. Nononaphthene, according to M. Kononov, is hexahydro-pseudocumene, &c. On the other hand, the identity of the hexahydrobenzenes and hexamethylenes may also be considered as established after the research on succinyl ether by Baeyer (Ber. 19, 428).

(2.) But beyond the  $C_6$  methylene group there may exist other methylene groups of  $C_3$ ,  $C_5$ ,  $C_4$ ,  $C_7$ , and, indeed, trimethylene and pentamethylene had already been obtained; hexamethylenes and tetramethylenes are known in the form of their derivatives. Some chemists had been inclined to doubt the possibility of the existence of heptamethylene but the author succeeded in obtaining it from suberone.

(3.) All these hydrocarbons having analogous structure ought to possess analogous properties, therefore they may be all classified as polymethylenes or naphthenes of the general formula  $C_nH_{2n}$ , and placed according to their chemical character between the aromatic series with the group  $C_6$ , and that of the paraffins, without adopting any special fixed group at all.

(4.) The well-known relation of the terpenes to the benzenes and the recently-established connexion between these hydrocarbons and the naphthenes, induce the author to regard them as a third isologous series of  $C_nH_{2n-4}$ ; the second series being formed of naphthylenes or unsaturated derivatives of naphthenes, of the general formula  $C_nH_{2n-2}$ . Thus, all derivatives of 4-atomic terpenes become derivatives of decanaphthenes; whilst those of 2-atomic terpenes ought to be classified with the decanaphthylenes.

(5.) It is remarkable that the boiling points of polymethylenes are almost the same as those of the paraffins with the same number of atoms of carbon in the molecule.

$C_5H_{12}$ 37°;	$C_6H_{14}$ 67°;	$C_7H_{16}$ 100°;
Normal pentane.	Hexane.	Heptane.
$C_5H_{10}$ 35°;	$C_6H_{12}$ 69°;	$C_7H_{14}$ 98°—100°;
Pentamethylene.	Hexamethylene.	Heptamethylene.
$C_5H_9.CH_3$ 70°—71°;	$C_6H_{11}.CH_3$ 99°;	
Methylpentamethylene.	Hexahydrotoluene.	
$C_6H_{10} 2 CH_3$ 123°—124°;	$C_6H_{10} \left\{ \begin{array}{c} C_2H_5 \\ CH_3 \end{array} \right\}$ 150°—152°;	
Iso-octonaphthene (para).	Methylethylhexamethylene (ortho).	
$C_5H_8 \left\{ \begin{array}{c} C_2H_5 \\ CH_3 \end{array} \right\}$ 124°;	$C_7H_{11} 2 CH_3$ 150°—152°.	
Methylethylpentamethylene (ortho).	Dimethylheptamethylene (ortho).	

These considerations and data make the author inclined to suspect that the naphthenes from Caucasian naphtha are mixtures of analogous members of different series of polymethylenes. For every additional  $CH_2$  in the carbon group the boiling point rises from 30° to 34°.—N. W. T.

#### PATENTS.

*Improvements in Distilling Shale, Coal, or other Oil and Tar-yielding Minerals, and in Retorts therefor.* A. Neilson, Inkerman, and W. Black, Airdrie. Eng. Pat. 9783, June 14, 1889. 8d.

THE retort is built of fire-brick in the form of a large cylinder and preferably encased by a shell of iron or other material to exclude air. A hopper is fixed on the top to which the sheet iron is securely jointed. A door is also fixed at the bottom for the removal of the residue from the retort. A jet of steam and a supply of air are introduced into the retort at or immediately above the lower door, both of which can be regulated according to the heat required. The retorts may be built singly or in groups, but no arrangement for furnaces or flues is required, the whole heating being obtained from the combustion of the material within the retort itself. The retort when applied to shale distillation for oil is preferably built in a vertical position. It is of great height and large dimensions and may be oblong at the bottom, tapering slightly and regularly narrower toward the top. Its action is practically continuous, a portion being drawn from the bottom and another fresh portion

charged at the top at regular intervals. In distilling shale for oil the retort is heated up and a quantity of spent shale left in the lower part of the retort, the upper part being charged with fresh shale. A jet of steam with the necessary amount of air is then admitted at the bottom to carry on the combustion of the spent shale, the hot gases from which pass up through the mass of fresh shale, whereby the oil products are distilled. In order to regulate the heat and at the same time improve the quality of the products, steam, gas from scrubbers, spent ammonia, or lined water may be introduced, and in cases in which there is insufficient carbon in the spent material to maintain the necessary heat in the retort, a small quantity of coke breeze, cinder, or anthracite coal may be charged along with the material to be operated on.—D. B.

*Improvements in the Distillation of Mineral Oils and like Products, and Apparatus for that Purpose.* J. Dewar, Cambridge, and B. Redwood, London. Eng. Pat. 10,277, June 24, 1889. 8d.

This invention relates to a method of conducting the distillation of mineral oils in such a manner that the benefit of regular vaporisation under high pressure is obtained, and also at the same time such advantage as can be got from the operation commonly termed "cracking." For this purpose the inventors propose to arrange a suitable boiler and a condenser in free communication with one another, the latter being provided with a regulated outlet for condensed liquid. The space in the boiler and condenser which is not occupied by liquid is charged and kept charged with air or, preferably, with carbonic anhydride under considerable pressure, so that the distillation is also conducted under great pressure which can be regulated at will. In this manner the inventors claim to obtain from the residue of heavy oil a quantity of light oil suitable for illuminating purposes which cannot be obtained by distillation under the ordinary pressure. The upper part of the boiler may also be so arranged as to operate according to the cracking method, the cracking in this case taking place under high pressure.—D. B.

*Improvements in Coke Ovens.* C. Heucken, Aix la Chapelle, Germany. Eng. Pat. 3363, March 3, 1890. 8d.

THESE improvements are in the construction of bee-hive ovens modified for the recovery of the products of distillation, and permit of the ovens being heated from the floor and sides, and not from the floor only as is usual; in this way the time of coking is brought down to the amount required for ovens of the more modern systems, without any deterioration of the high quality of the coke produced. The cross section of the improved ovens is slightly elliptical, that shape permitting of a convenient door at one end of the minor axis without detriment to the floor flues of the oven. The space beneath the floor of the oven is divided by a partition into two parts not communicating with one another, and each divided into seven flues partially open to each other and communicating on each side with an equal number of side flues of varying width, all of which pass into an arched flue placed around the periphery of the oven dome. This arched flue is divided by partitions into three compartments, one of which contains convenient openings into the oven, and another is connected with side flues leading to the chimney. The gases in the oven pass through the openings in the dome into one division of the arched flue, then down one set of side flues through the flues under one half of the floor and up through one set of flues on the opposite side into the second division of the arched flue; they are then forced down the adjacent set of side flues, through the flues under the second half of the floor and up through the opposite set of side flues into the third division of the arched flue, from which they finally escape into flues leading to the chimney. There are convenient openings for observation, supply of air, &c.—S. B. A. A.

## IV.—COLOURING MATTERS AND DYES.

*α-Naphthol-Benzoin.—A New Indicator.* R. Zaloziecki. Chem. Zeit. 14, 605—606.

See under XXIII., page 770.

*The Action of Fuming Nitric Acid on Hexachlorobenzene.* Istrati. Bull. Soc. Chim. 1890, 3, 184—186.

Fuming nitric acid attacks hexachlorobenzene energetically, forming dichlorodihydroxyquinone; this, in fact, is the best method of preparing the latter body, as an excess of nitric acid does not destroy it. It appears in this reaction that (1) the chlorine in the benzene nucleus is partially removed and replaced by oxygen, this action being accompanied (2) by the evolution of a gaseous mixture of 62—64 per cent. of nitrous gases and carbon dioxide, 34—39 per cent. of oxygen, and 4—7 per cent. of nitrogen.—E. B.

*A New Method of Chlorinating Aromatic Compounds.* Pétricon. Bull. Soc. Chim. 1890, 3, 189—191.

THE author recommends stannic chloride for assisting the chlorination of benzene compounds. The stannic chloride is preferably used "in the nascent state," being produced by the action of chlorine on metallic tin in admixture with the compound to be treated. Compared with iodine, antimony pentachloride, &c., stannic chloride has the following advantages: the chlorination is effected in a shorter time; it is especially useful in the preparation of higher chlorinated derivatives such as, for instance, pentachlorobenzene; its action is well defined, a more limited number of compounds being produced than when iodine is used; very little decomposition takes place of the aromatic compound under treatment.—E. B.

*The Action of Sulphuric Acid on Tribromophenol.* Georgesco. Bull. Soc. Chim. 1890, 3, 193—195.

HERTZIG has stated that tribromophenol is destroyed by boiling for four hours with sulphuric acid. The author, guided by the results obtained by Istrati with the chlorobenzenes, has re-examined the reaction. On heating together a mixture of tribromophenol and sulphuric acid for 20 hours on a sand-bath, action takes place accompanied by the evolution of considerable quantities of gas, consisting chiefly of sulphur dioxide along with small quantities of carbon dioxide and hydrobromic acid, the sulphuric acid at the same time becoming deep red-brown in colour. A precipitate separates when the acid mixture is poured into water. This precipitate consists of brominated fractions containing from 42·137 to 63·786 per cent. of bromine, and having the characteristic brown colour, high melting point, and other properties of this class of bodies. In the flask used for the experiment there remained a solid substance, which was found to be a mixture of unaltered tribromophenol, tetrabromophenol, pentabromophenol, and a red substance, with difficulty soluble in hot benzene, crystallising therefrom in small plates, which sublime without melting or decomposition at 250°—258°. It was not obtained in sufficient quantity to permit of an analysis being made. Pentabromophenol is a crystalline rose-coloured body melting at 220°.

When, as in Hertzig's experiment, the action of the acid is continued for four hours only, the result is similar, the higher brominated derivatives, however, being formed in smaller amount.—E. B.

## V.—TEXTILES: COTTON, WOOL, SILK, Etc.

*Thermo-Chemical Investigations on Silk.* L. Vignon.

Bull. Soc. Chim. 1890, 3, 405—410.

See under XXIII., page 771.

*Distinction between Jute, Flax, and Hemp Fibre.* W. Lenz.  
Zeits. Anal. Chem. 29, 133—134.

JUTE is of course recognised by its peculiar structure, chiefly by the irregularly thickened walls of the bast fibres. To be able to make use of such distinction requires a great deal of care and considerable practice. An easier method seems to be by the aid of polarised light. The fibre is warmed with nitric acid and a grain of potassium chlorate, washed with water, then warmed with water containing a little caustic potash, and finally well shaken with pure water. A portion is brought on to a microscope slide, and the moisture is allowed to evaporate; a drop of glycerin is now added and a cover glass laid on. Not only are the peculiar thickenings now well shown under the microscope, but the specimen serves especially well for examination by polarised light. With a crossed Nicol, using a magnifying power of about 200, a beautiful play of colours is obtained with almost every fibre in the case of flax and hemp, whilst with jute only yellow or bluish single shades are obtained; occasional fibres show the colouration of the flax, but not to such a marked degree. It is necessary to have the individual fibres well separated, for colourations are obtained with jute if the individual fibres cross or run counter to each other. The cross lines running at right angles to the walls of the bast cells are much more distinct in polarised light with a magnifying power of 600 than they are in ordinary light.—T. L. B.

## VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

*Methods of Dyeing pursued by the Chinese.* Chem. Ind.  
13, 154—157.

FOR dyeing yellow, the bark of a large tree, the *Pterocarpus flauus* (Chinese name, huang pei), is employed. Silk is softened in boiling water, and immersed in a solution of the colouring matter of the bark, prepared in the cold, and is then dried in the shade. The colour is not fast to hot water. Cotton is first worked in a liquor prepared by steeping meizu plums in water for several days, then the solution of the yellow colouring matter is added to the liquor, and the cotton cloth is passed through the bath until fully dyed, which requires sometimes as many as 80 immersions, and the cloth is finally dried in the shade.

Gamboge (Chin., teng huang) is employed chiefly for painting.

The bark of *Berberis thunbergii* (Chin., hsiaopei), and the wood of *Dereilla versicolor* (Chin., hang lou) are also employed.

Turmeric (Chin., kiang huang) is largely used.

*Rhamnesia linensis* (Chin., tihuang), and the buds of *Sophora japonica* (Chin., huai hua or hua ni), are also used for dyeing yellow, the former extensively.

Five different qualities of indigo are in use for dyeing blue. The indigo vat is prepared by gently heating for some time a mixture of indigo, oat- and rice-refuse, and lime.

Silk is dyed green by mordanting it first with alum, dyeing yellow with hwaimi, and then dipping in an indigo vat set with Shanghai indigo. In dyeing cotton, the order of the operations is reversed, a blue colour being first dyed, and then, after mordanting with alum, a yellow.

The preparation of Chinese green is described in the original paper. The price of this colour has fallen from 225 frs. in 1865 to 66 frs. per kilo. at the present time.

For dyeing red there are employed the following dye-stuffs: Safflower (*Carthamus tinctorius*), the roots of lithospermum erythoxylon, sanderswood, sapanwood, and madder.

Vermilion and sapanwood are employed for dyeing brown on cotton or silk, which has been previously dyed blue with indigo. Bright reds are obtained by treating white silk or cotton with these materials.

Grey is produced by working blue-dyed cotton or silk in a decoction of the nut-galls of *Rhus semialatus*.

In dyeing black, the leaves and husks of *Mimosa ferox* and gull-nuts are employed along with ferrous sulphate. Another black is obtained on dark blue-dyed fabrics with hwa kuo and black alum.

There are some 10 fairly large dye-houses in Shanghai, employing about 400 hands.—E. B.

*Dye-Wood Extracts, and their Modern Manufacture.*  
J. H. Soxhlet. Chem. Zeit. 14, 667.

THIS industry sprang up in France at the beginning of this century, Meissonier being the first manufacturer of a liquid and solid material suitable chiefly for printing. Logwood is occasionally submitted to a process known as "oxidation" before being placed in the extractors. The chips are spread out in thin layers, and moistened with water containing in solution or suspension the following substances: ammonium chloride, ammonium carbonate, putrid urine, chalk, clay, saltpetre, potassium permanganate, and bichromate; the exact procedure being kept secret. This "oxidation" must be condemned, owing to the fact that the resulting extracts yield much less permanent colours.

The extraction of the colouring matter from the wood is conducted either by maceration in open vessels—French method—or under steam pressure at high temperatures, as first employed by Lanford in America. The yield of extract at 45° B. by the former process is 16½ per cent., by the latter from 20 to 21 per cent. For printing purposes, the "French" extract is to be preferred, higher prices being obtainable, but for dyeing woollen yarn, where fastness rather than purity of tone is necessary, the "American" extract is better. The "French" extract is often adulterated with molasses, glucose, dextrin, &c., to the extent of 50 per cent. in the cheaper qualities.

In America logwood extract is sold under the name of "black dye extract," adulterated with quercitron and hemlock; and fustic extract, called "substitute for fustic," adulterated with quercitron.

Extracts manufactured under pressure usually contain more woody particles than the "French." To avoid this, the liquors may be allowed to subside before evaporation, or they may be filter-pressed, or precipitated with salt or sodium sulphate.

The pressure in the pans should not exceed 1 or 1½ atmospheres.—F. H. L.

*A New Bleaching Process.* Kassner. Monit. Scient.  
1890, 103.

THE author proposes to take advantage of the well-known oxidising action of potassium ferrieyanide in presence of alkali. The resulting ferrocyanide is oxidised by digestion with peroxide of lead, the action being facilitated by passing a stream of carbonic acid. The deposit from this operation is dried, and the lead peroxidised by calcination, the product being boiled with sodium carbonate solution, and the precipitated peroxide used as above.—C. F. C.

*The Chroming of Wool.* Scurati-Mauzoni. Mon. de la teinture, 1890, 34, 29.

IN order to determine whether on treating wool with bichromate, the fixation of the chromium is due to the reducing power of the wool itself, or to organic sulphur compounds contained by it, the author has examined the final liquor and found that it contained potassium sulphate, which increased in quantity with the length of the boiling; that the colour of the wool was proportional to the amount of sulphate formed, and that by long-continued boiling the

wool became green, whilst the liquor remained yellow as long as it contained bichromate.

If the treatment with bichromate be allowed to take place under pressure (two atmospheres for three hours), then at the conclusion the liquor contains no more bichromate, the wool becomes a dirty green, shrinks together and is quite valueless, and the interior of the vessel is blackened.

It is thus evident that it is the sulphur contained in the wool which produces the reduction, being itself oxidised to sulphate. The author has shown that finely divided sulphur is capable of thus acting by impregnating wool with sodium thiosulphate, treating with hydrochloric acid, which causes a deposit of sulphur in the wool, and after washing, treating with bichromate; after half an hour's boiling this is reduced and the hydroxide deposited in the wool, the finely divided sulphur first forming sulphuretted hydrogen which performs the reduction. The same effect may be produced by a stream of sulphuretted hydrogen. The practical objection to both these methods is that two operations have to be performed. The author has attempted to find a sulphur compound which might be added with the bichromate to the bath. He has found the potassium tri- and tetra-thionates and sodium tri-thionate the most efficient. They may be kept mixed with the bichromate in the cold without action, and by warming produce a slow reduction. For 100 grms. of wool, 2.5 grms. of potassium bichromate and 8 grms. of potassium tri-thionate are dissolved in 5 litres water, and boiled one hour. It is evident that the price of the thionate must be considerably reduced before its use becomes practicable. (See this Journal, 1890, 58—61.)—A. L. S.

#### PATENT.

*Manufacture of Bleaching and Disinfecting Liquor.* E. Hermite, E. J. Paterson, and C. F. Cooper, London. Eng. Pat. 8177, May 16, 1889. 4d.

Blocks of carnallite are by mechanical means divided up into three portions, one of which is a double chloride of

magnesium and potassium. This is dissolved in water and a current of electricity passed through the solution (see this Journal, 1887, 727; and 1888, 126 and 321), which is thereby decomposed and may now be employed as a valuable bleaching and disinfecting agent. Some magnesia is also liberated which is found to be advantageous as it renders the solution alkaline.—S. G. R.

### VII.—ACIDS, ALKALIS, AND SALTS.

*Reduction of Nitrous Vitriol by Coke.* G. Lunge. Zeits. f. angew. Chem. 1890, 195—196.

THE author has already shown (Chem. Ind. 1885, 2; also this Journal, 1885, 31) that the nitric acid gradually disappears from a mixture of nitric and sulphuric acids by contact with coke, even at ordinary temperatures; whilst at higher temperatures such disappearance is very rapid. Nitrosylsulphuric acid is formed. This observation led to an explanation of the fact that even in the Gay-Lussac tower containing nitrogen peroxide, the nitrous vitriol gave no marked evidence of the presence of nitric acid. Later on (Zeits. f. angew. Chem. 1889, 387) it was shown that the reducing action of the coke could extend even to the nitro-compound. The present work deals more in detail with this latter action of the coke.

Solutions of chamber crystals in pure sulphuric acid were used—the sulphuric acid being of two strengths, 1.8375 and 1.7250. The coke used was:—(a) gas coke, in pieces the size of a pea; (b) the same, roughly powdered; (c) hard oven coke in pieces; (d) the same, powdered. The experiments were made at the following temperatures, 15°, 40°, 70°, and 100° C., and were carried out in flasks closed by means of a Bunsen valve, the air being displaced by dry carbon dioxide. Amount of nitrogen trioxide was ascertained by running the acid into warm permanganate solution. The accompanying table gives the experimental results.

Material.	Temperature.	Duration in Hours.	Original $N_2O_3$ in Grms. per Litre.	Decrease.		
				In Grms. of $N_2O_3$ per Litre.	In Percentage of $N_2O_3$ .	
I.—LIQUID OF SP. GR. 1.8375.						
Gas coke, pieces .....	{	15	24	18.93	0.330	1.71
		40	2	18.92	0.539	2.86
		70	2	19.30	0.712	3.84
Oven coke, pieces .....	{	15	24	19.30	0.285	1.48
		40	2	18.92	0.362	1.91
		70	2	19.30	0.452	2.34
Gas coke, powdered.....	{	15	24	19.30	0.790	4.09
		40	2	18.92	0.858	4.54
		70	2	16.22	0.903	5.57
Oven coke, powdered .....	{	100	2	16.22	4.611	28.43
		15	24	19.30	0.379	1.96
		40	2	18.92	0.451	2.38
		70	2	16.22	0.527	3.25
		100	2	16.22	2.770	17.08
II.—LIQUID OF SP. GR. 1.725.						
Gas coke, powdered.....	{	15	24	19.50	0.386	1.98
		40	2	19.50	0.574	2.94
		70	2	19.50	0.891	4.57
		100	2	19.50	3.410	17.49

Of course nitric oxide is evolved, and this must naturally escape unused from the Gay-Lussac tower. Some of the coke is also transformed into carbon dioxide, as was shown by displacing the air of the flask in one experiment by nitrogen instead of by carbon dioxide.

It is concluded that coke has here a reducing action, and the action increased with the temperature, and also with increase in the coke surface; the soft gas coke has a greater reducing action than the harder coke. A temperature of 40° may prevail in the Gay-Lussac tower in summer, and so after two hours' contact the nitrogen trioxide may be reduced to the extent of 2·4 to 4·5 per cent. The greatest reduction given (28 per cent.) takes place over 70°; this temperature never exists in the Gay-Lussac tower, but it may be found in the upper part of the Glover tower. The action is more pronounced in the case of solutions of nitrosyl-sulphuric acid in concentrated sulphuric acid than in the case of solutions in only moderately strong sulphuric acid.

The following practical conclusions are drawn:—It is useless to employ coke wherever the temperature rises above 70° C.; the upper portions of the Glover tower should not, therefore, be filled with coke, otherwise the acid becomes denitrated by coke instead of by sulphur dioxide; filling the Gay-Lussac tower with coke must lead to some loss (by evolution of nitric oxide). Nevertheless, by using hard oven coke in large pieces, and by not having the acid too strong, the action is more moderate, and so the coke may last also a considerable time. (See also this Journal, 1889, 164-168; 1890, 175-182.)—T. L. B.

#### *A Central African Salt Lake.* Chemist and Druggist. 37, 18-19.

"On the north of the Albert Edward Nyanza, and within a few miles of the equator, Mr. Stanley discovered the salt lakes of Katwe, 3,265 ft. above the sea. The temperature of the lake was 78·4° F.; a narrow thread of sulphurous water indicated 84°. Its flavour was that of very strong brine. A bottle full of this brine was brought to Cairo and analysed in the Laboratoire Khedivial there, and estimated by the chemists, Messrs. A. Pappe and H. Drop Richmond, to be composed as follows:—

Sodium chloride .....	18·67
Sodium sulphate .....	5·63
Sodium carbonate .....	2·72
Potassium carbonate.....	3·87
Water .....	68·77

"The residue is potassium sulphhydrate ·04, silica ·01, with traces of lime, magnesia, and organic matter. A sample of the natural crystalline salt taken from beds where the water had evaporated was brought, and is reported upon by Mr. Henry S. Wellcome, who, after giving the exact analysis of bases and acids, assumes the composition to be as follows:—

Potassium sulphate .....	8·43
Sodium sulphate .....	5·32
Sodium carbonate .....	2·46
Sodium chloride.....	82·71
Oxide of iron.....	0·15
Water.....	0·82"

#### PATENTS.

*Application of the Residues of Soda Manufacture for Building Purposes.* W. Schleuning, Heidelberg, Germany. Eng. Pat. 6433, April 15, 1889. 6d.

THE residues are exposed to oxidation so as to convert them into "soda gypsum"; burnt gypsum is added thereto, together with lime and sufficient water to form a mortar or plaster, or with little water to form a paste, which is subjected to pressure in suitable moulds, for the production of building blocks.—E. G. C.

*A Process first for Producing a Clean Gaseous Fuel to be used in the Evaporation of Brine, and secondly for Recovering the Ammonia formed during the Production of said Gaseous Fuel.* S. W. Wilkinson, Manchester. Eng. Pat. 8993, May 30, 1889. 6d.

COAL is burned in a closed furnace or producer with a limited supply of air and steam, so regulated that the gaseous products may contain a maximum of ammonia. The gases are passed through a scrubber or tank containing sulphuric acid which retains the ammonia; they are then led into a chamber consisting of chequer brickwork and there mixed with the quantity of air necessary for complete combustion. The ignited gas may be led over the surface of brine without any carbon being deposited. An arrangement is also shown for utilising the waste gases for heating brine tanks by means of flues, &c.—S. B. A. A.

*Improvements in the Manufacture of Phosphorus.* T. Parker and A. E. Robinson, Wolverhampton. Eng. Pat. 10,437, June 27, 1889. 4d.

A SUITABLE material, such as a natural phosphate, is mixed with carbon or carbonaceous material in a furnace or crucible, together with a non-volatile metal, such as iron or copper. The whole is then kept at a sufficient temperature to effect the reduction by an electric current, the molten metal acting as a bath to keep the temperature more uniform. Phosphorus comes off as a vapour and is condensed by known means.—E. T.

*Improvements in the Manufacture of Carbon Bisulphide.* T. Parker and A. E. Robinson, Wolverhampton. Eng. Pat. 11,099, July 10, 1889. 4d.

A MASS of charcoal is placed between two suitable electrodes, preferably constructed of carbon, which are connected with an electric current of sufficient density to bring the charcoal to the temperature necessary for it to combine with the sulphur, which latter is passed through the heated mass in the form of vapour. The supply of charcoal is renewed continuously or from time to time, either by hand or by automatic feeding arrangements. The vapours of carbon bisulphide are led off and condensed in the usual manner.—C. A. K.

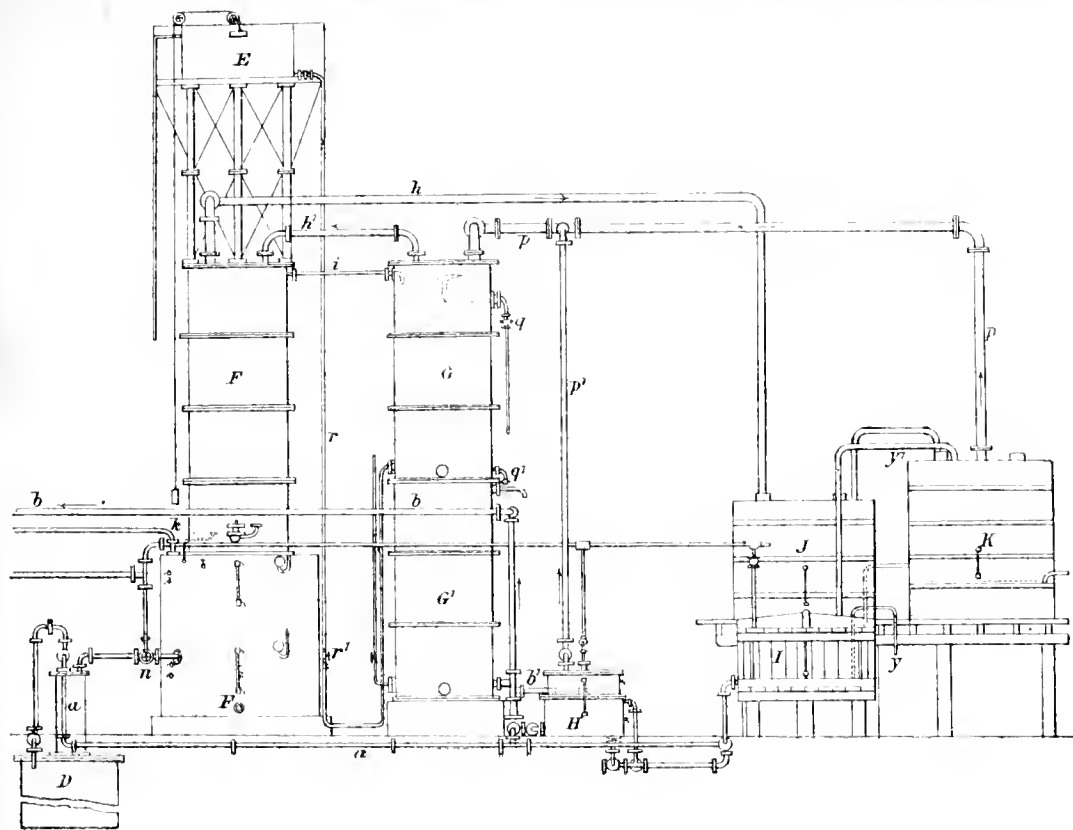
*Process and Apparatus for the Production of Fuming Sulphuric Acid or of Sulphuric Anhydride from the Acid Waste or Residual Matter obtained in the Petroleum, Paraffin, and Tar Industry.* J. D. Starek, Iseheim, Bohemia. Eng. Pat. 12,028, July 29, 1889. 8d.

THE acid waste or other material is fed in at a proper speed to the lower end of an inclined iron retort within which is an endless screw working for the purpose of drawing outwards the residual coke-like mass. The sulphurous anhydride and water produced are passed through a condenser and a wash-chamber to remove any water or hydrocarbons coming over. The gas is then led through a tower similar to a Glover tower, to dry it completely, and mixed with dried air or oxygen in the required amount to form sulphuric anhydride. The mixture thus made is conducted through a tube filled with platinised asbestos, pumice stone, or metallic oxides precipitated upon porous bodies, and the sulphuric anhydride produced is either condensed or absorbed in sulphuric acid. The residual gases containing sulphurous anhydride are passed into lead chambers to be converted in the ordinary way into sulphuric acid. The specification is illustrated by drawings.—S. G. R.

*Improvements in the Treatment and Distillation of Ammoniacal Liquor of Gas and other Works, and in the Manufacture of Sulphate of Ammonia therefrom, and in the Apparatus to be employed therein, and for the Prevention of Nuisance in such Manufacture.* A. W. Ellis, London. Eng. Pat. 13,003, August 17, 1889. 1s. 3d.

THE specification is of great length and is illustrated by six sheets of drawings. Of these the most important is





THE DISTILLATION OF AMMONIACAL LIQUOR.

the one showing the general arrangement of the whole plant, and which is given above.

The liquors to be treated pass downwards from the overhead tank E, through the pipe *r* controlled by the tap *r'*, to the bottom of the heater G. In their passage upwards they are warmed by waste gases obtained in a manner which will be seen later. Here also any light tarry matters or scum are run off by the pipe *q* and the heavier portion or tar sinks to the bottom and is removed at *q'*. The now hot liquors pass away at the top by *i* into the distilling column F, and any gases evolved also escape to F by the pipe *h*. The distilling column F is made of iron and is preferably cylindrical in shape, and placed within it are a series of trays one below the other over which the liquid runs in a thin continuous stream. The plates are of special form. The liquid is then steamed and the free ammonia in the liquors is evolved and passes upwards to the top of E. The liquor in its passage downwards finally reaches the fixed ammonia still F<sup>1</sup>, which by means of a continuous horizontal partition is divided into two portions, an upper and a lower. The upper is again subdivided into three, one chamber being central and cylindrical, the other two annular, whilst the lower is made up of five divisions, one as before being central and the remaining four peripheral and annular. A steam coil passes through all the sections and any residual tar is here removed. Lime is introduced by the pipe *k*, and thus the fixed ammonia is liberated. The steam enters first into the chamber *d* in which is the oldest and most nearly exhausted liquor. This column is also cylindrical and made of iron. The spent liquors pass away at *n* to the settler D. The ammonia which has reached the top of the still F is led away by the pipe *h* to the saturator J, containing dilute sulphuric acid. The hot gases unabsorbed in J are passed through the interceptor K, also containing sulphuric acid, to remove the last traces of ammonia, and the hot gases, rich in sulphur compounds, are taken by the pipe *p* to the heater G, down

which they are carried by a coil to the condenser G<sup>1</sup> through which water is kept circulating. The residual gases, rich in sulphuretted hydrogen, pass off by the pipe *b* and can be treated by any of the known methods for the utilisation of that gas. The impure strongly smelling liquid separated in the condenser G<sup>1</sup> flows by *b'* to H, which is heated either by steam or by the nearly boiling spent liquors from the settling tank D and which are conveyed by the pipe *a*. The vapours given off are either led back to *p* and G by *p'* or may be treated for the recovery of cyanogen. The liquid in the saturator J, which should contain a slight excess of ammonia, is run into the steam-tight receiver I, where, either by the hot spent liquors from D which have already passed H or else by steam, every trace of ammonia, sulphuretted hydrogen, &c., can be driven off. The gases pass by *y'* to the interceptor K; *y* is a safety tube. In I any precipitate of sulphides of iron and arsenic separates out, and the clear liquor, which has been further concentrated by the steaming or heating to which it has been subjected, is run out and caused to crystallise. This residue in I is removed from time to time by a separate exit. For further details of the construction of the different parts of the plant, the original specification should be consulted.—S. G. R.

*Improvements in the Evaporation of Brine and other Solutions or Liquids, and in Apparatus employed therein.* J. C. Mewburn, London. From C. C. Peck, Warsaw, U.S.A. Eng. Pat. 6107, April 22, 1890. 1s. 1d.

See under I., page 728.

## VIII.—GLASS, POTTERY, AND EARTHENWARE.

*Red Glass.* C. Guignot and L. Magne. *Monit. Céram. et Ver.* **21**, 26.

THE authors consider the colour of red glass (coloured with copper) to be due to the presence of cuprous oxide, in opposition to the views of *Ebell*, who ascribes it to finely divided metallic copper (this *Journal*, 1888, 123; and 1890, 68). They found their contention on the fact that basic cupric chloride heated between two glass plates gives a red colour, holding that the soda in the glass reacts with the basic cupric chloride giving cuprous oxide and sodium chloride. Even admitting that cuprous oxide may at a high temperature decompose (*Proc. Chem. Soc.* **78**, 19) and the metallic copper dissolve in the glass with the formation of a red colour, yet this experiment must deal with a distinct phenomenon, inasmuch as it is completed at the softening point of the glass, provided air be excluded.

By fusing together 100 parts of sodium carbonate, 50 of calcium carbonate, 260 of sand, and 10 of cupric oxide, a green-blue glass is obtained; the same mixture with 15 parts of forge scale substituted for the cupric oxide gives a yellow glass; if now these be fused together they react upon each other and yield a dark green glass streaked with purple-red veins.—B. B.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

*Influence of Sea-Water on Portland Cement.* E. Candlot. *Monit. Céram. et Ver.* **21**, 42. (Compare this *Journal*, 1889, 543.)

THE nature and quantity of the water used are of influence on the setting of cement. Sea-water, by forming calcium chloride and sulphate by double decomposition of the magnesium salts it contains with the lime compounds present in the cement, retards the setting. Neat cement gauged with sea-water, is at first stronger than when fresh-water is used, but retrogrades after some months. Cement, when made up into mortar by admixture with sand, shows no such tendency; the use of sea-water may delay its reaching its full strength, but this is attained after the lapse of a year; this fact applies particularly to finely ground samples.—B. B.

*Magnesia Cements.* Sprechsaal, **23**, 247.

THE various cements for stone and terra-cotta depending on the formation of magnesium oxychloride, generally consist of Portland cement or hydraulic lime mixed with magnesia and any inert substance such as sand or granite, according to the use to which they are to be put, moistened with magnesium chloride, and applied to the surfaces to be repaired after moistening them with the same solution.

—B. B.

### PATENTS.

*Application of the Residues of Soda Manufacture for Building Purposes.* W. Schlenning, Heidelberg, Germany. Eng. Pat. 6433, April 15, 1889, 6d.

See under VII., page 734.

*Process and Apparatus for Producing Artificial Stone.* B. Mertelmeier, Berlin, Germany. Eng. Pat. 13,957, September 1, 1889. 8d.

THE powdered constituents are exhausted of the inherent air in a hermetically closed receptacle, and are afterwards mixed, by means of a stirring device within the receptacle, with "air-exhausted water." The product is then cast into moulds, and is said to be "equal or even superior in solidity and durability to natural stone." Drawings of the apparatus are given.—E. G. C.

*An Improved Manufacture of an Artificial Solid Material and Apparatus therefor.* H. H. Lake, London. From J. F. Gesner, New York, U.S.A. Eng. Pat. 1097, January 21, 1890. 11d.

MATERIALS made by mixing inert solids with alkaline silicates are not suitable for use as artificial stone, inasmuch as the alkaline silicate remains soluble and is liable to be washed out by rain. Attempts have been made to get over this difficulty by treating the stone with solutions of metallic salts such as calcium chloride, with the object of depositing insoluble silicates within the pores of the substance, but owing to its imperviousness thorough impregnation has been impracticable, even when the use of a pump or heated solution has been resorted to, or the mass mixed with sand to give it greater porosity. The patentee proposes a process, of which the following is an outline, to obviate these defects: Soluble glass either alone or, more generally, mixed with some inert solid, is allowed to dry in the air until it is stiff enough to be handled, and then subjected to what is termed "calcining." By this is meant the process of gradually raising its temperature above 212 F. until the combined water is driven off, and it swells to a light porous mass. If this be effected while it is confined in a mould which may be held together by springs, or a screw press with springs inserted to give a yielding pressure, it can be made to assume and retain any desired shape, and become reasonably compact although porous. On immersion in a solution containing some substance such as a free acid or a soluble base or a metallic salt capable of decomposing the alkaline silicate, rapid absorption takes place, after which the moulded blocks are usually washed, preferably by a spray of water, to remove soluble salts, dried, and if necessary pressed again, and if desired rendered impervious by treatment with some waterproofing substance such as paraffin wax, sulphur, fatty or resinous substances. The product may be coloured by the addition of some inert pigment to the soluble glass at the beginning of the process. Besides the finished article thus described, the material at the end of some of the steps of the process may be used for certain purposes. Various forms of press may be employed, and the heating effected in different ways; in that most approved and minutely described the mass to be treated is held between perforated metal plates covered with asbestos cloth, which fit into frames provided with a set of horizontal pipes for the transmission of steam (which may be superheated), a series of such arrangements being placed one above the other, and subjected to the action of a screw press provided with spiral springs to regulate the pressure.

The claims, which are 44 in number, deal with variations in the mixtures used, the conditions of manufacture, and the structure of the press.—B. B.

*Improvements in Kilns for Cement and the like.* W. A. Gibbs, Chingford. Eng. Pat. 4741, March 26, 1890. 8d.

See under I., page 727.

## X.—METALLURGY.

*Experiments on the Solubility of Minerals.* C. Dölter.  
Monatsh. **11**, 149—150.

See under XXIII., page 767.

*On the Reduction of Oxygen Compounds by Magnesium.*  
C. Winkler. Ber. **23**, 44—57, 120—130, and 772—792.

THE author has examined the behaviour of magnesium, the heat of combustion of which is very high, towards compounds containing oxygen. He gives the following results of experiments on the oxides and carbonates of the elements contained in the first group of the periodic system.

The substances under examination were previously dried or fused carefully in order to dehydrate, and then mixed in a warm mortar with the requisite quantity of powdered magnesium, and transferred to a glass tube sealed at one end. Owing to the difficulty of obtaining the oxides of the alkali metals pure, *i.e.* free from carbonate, water, &c., and retaining them in that state, it was decided to employ the carbonates.

A.—Chief Group (Carbonate  $R_2CO_3$ ).

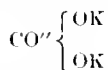
1. *Lithium* (7.01).—On heating, violent detonation accompanied by the rapid combustion and vaporisation of the reduced metal. (Mixture being 1 mol. of the carbonate to 3 atoms of magnesium.)

2. *Sodium* (23).—(Mixture as before.) Detonation and vaporisation of the metallic sodium which partially condensed in the cooler part of the tube.

3. *Potassium* (39.03).—The reaction was quiet and safe; no deflagration or volatilisation of the metal. The reduction appeared to require less heat than was necessary in the case of sodium carbonate. By introducing the mixture into a porcelain boat and carrying out the reaction in a current of hydrogen in a combustion tube it was found that at a low red heat a metallic mirror was formed, further increase of temperature driving off greenish vapours. A greyish black residue consisting of magnesia and carbon remained in the boat, and as the residue was only slightly alkaline the potassium must practically have been driven over completely.

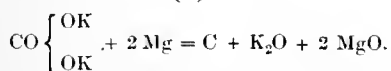
It seems, therefore, very probable that potassium can be prepared on a commercial scale in a similar manner, *viz.*, by heating freshly calcined potassium carbonate with magnesium in an iron retort, and condensing the potassium vapour in a suitable receiver. Illuminating gas could, however, hardly be substituted for the hydrogen as it contains carbon monoxide, which might form the dangerous explosive compound with the potassium, though in carrying out the process the presence of carbon monoxide can scarcely be avoided.

Of the three oxygen atoms in the molecule of potassium carbonate, two can be more easily taken up by magnesium than the third. This observation is of theoretical importance as it yields a proof of the constitutional formula—

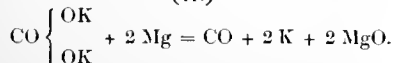


also that the group OK is more easily reducible than the CO group. When 1 mol. proportion of potassium carbonate is heated with two atomic proportions of magnesium instead of three, the reduction must take place according to one or other of the following equations:—

(I.)



(II.)



It was found in the following experiment that the *second* equation represents the reaction.

A mixture of 138 parts (1 mol.) of potassium carbonate and 48 parts (2 atoms) of powdered magnesium was introduced into a combustion tube, at first heated gently and then to low redness, a moderately rapid stream of hydrogen being meanwhile passed through the tube. The metallic mirror observed in all the preceding experiments could not be seen, but instead in the cooler part of the tube was obtained a considerable layer of a green powder, which could be no other than the compound of potassium carbon monoxide; also no separation of carbon took place in the residue. The residue consisted of magnesia, so it may be taken that the formula of the potassium carbon monoxide compound is—



By heating together 56 parts (1 mol.) of potassium hydroxide and 24 parts (1 atom) of powdered magnesium, the reaction is very violent, but if 56 parts of magnesia be added, the reduction, which commences at a red heat, is quiet and safe, the residue remaining behind consisting of almost pure magnesia. This reaction may serve for the manufacture of potassium, employing the apparatus devised by C. Netto (this Journal, 1889, 122) for preparing metallic sodium continuously by allowing molten caustic soda to trickle through red-hot carbon contained in a vertical retort, the sodium which collects at the bottom being siphoned off. Instead of the carbon, bars of magnesium might be employed, though the magnesia formed at first may hinder further action by forming a coating on the magnesium.

The author gives the following data for the (theoretical) preparation of potassium:—

	M.
1.44 kilos. of potassium hydrate, at 2 M.....	2.88
0.62 kilos. of magnesium, at 10 M.....	24.80
	<hr/> 27.68

39 M. against 130 M., the present commercial price.

N. Beketow (J. d. russ. Phys. Chem. Gesellsch. Protok. 1888, 363—365) prepared potassium by reducing potassium hydrate with aluminium, but only half of the metal was obtained as such, the other half remaining in the residue as potassium aluminate.

4. *Rubidium* (85.2).—On gentle heating no reduction occurs, but on strongly heating the reaction takes place quietly, with a slight glow and partial volatilisation of the metal.

5. *Cesium* (132.7).—No reduction.

B.—Sub-group (Oxide  $R_2O$ ).

1. *Copper* (63.18).—Moderately strong decrepitation and partial deflagration.

2. *Silver* (107.66).—Reduction takes place with explosive violence.

3. *Gold* (196.20).—Quiet decomposition of the gold oxide into its elements, without the aid of the magnesium present.

The author's experiments, which disprove the statement of Warren (Chem. News, 1889, **60**, 187) that magnesium does not reduce the alkalis and alkaline earths, appear to show that the reducing power of the magnesium diminishes as the atomic weight of the element increases. This is in accord with the observation of Beketow, that, beginning at lithium and as far as rubidium, the heat of oxidation increases with the atomic weight.

The heavy metals belonging to the sub-group exhibit an opposite behaviour, *i.e.*, the reduction of their oxides is more violent the higher the atomic weight of the element. The anomalous behaviour of gold is only apparent, and is caused by the slight stability of the gold oxide at a high temperature.

## SECOND GROUP.

The reduction takes place according to the equation—



Some of the hydroxides as well as the oxides were experimented upon.

## A.—Chief Group (Oxide RO).

1. *Beryllium* (9.08).—Reduction takes place quietly, the mixture becoming dark coloured and glowing faintly, but the reaction is incomplete.

2. *Magnesium* (24.3).—No formation of a lower oxide.

3. *Calcium* (39.91).—Almost complete reduction, the mass becoming green coloured, but without glow.

4. *Strontium* (87.3).—Reaction commences quietly, the glow gradually extending throughout the mass. Reduction apparently complete.

5. *Barium* (136.9).—Tolerably violent reaction accompanied by strong glowing and partial ejection of the mass.

## B.—Sub-Group (Oxide RO).

1. *Zinc* (65.1).—Reduction takes place with explosive violence, accompanied by flame and decrepitation.

2. *Cadmium* (111.7).—Reaction commences with hissing noise and glowing of the mass. Complete reduction.

3. *Mercury* (199.8).—Powerful detonation accompanied by flame and complete volatilisation of the mercury.

From the foregoing it will be seen in the reduction of the oxides of the elements of the chief group, that an increase takes place, which, omitting magnesium, is in direct relation to the increase of atomic weight. The behaviour of the hydroxides is more marked. The reduction of the oxides of the sub-group is generally very violent, the reduced metal being volatilised completely or almost so.

## THIRD GROUP.

A.—Chief Group (Oxide R<sub>2</sub>O<sub>3</sub>).

1. *Boron* (10.9).—Boric anhydride and borax are easily reduced on heating with magnesium, the reduction of the latter being accompanied by delagration and volatilisation of sodium. Boron is never obtained, but magnesium boride of varying composition and behaviour towards acid. Hydrochloric acid either dissolves the magnesium boride completely with evolution of hydrogen mixed with boron, or leaves a solid residue consisting of a compound of boron and hydrogen. In keeping with the acid character of boron, acid borates are formed during reduction. A monoxide of boron does not appear to exist.

2. *Aluminium* (27.01).—Aluminium oxide on heating with magnesium forms a dark-coloured powder, the mixture glowing and swelling up during the reaction. According to the proportion of reducing agent employed, the aluminium is found in the above residue, either as finely divided metal or as monoxide. This monoxide, which up till now has not been known, could not be prepared in the pure state. The formation of the latter in accordance with the acid character is always accompanied by that of magnesium spinelle.

3. *Scandium* (43.97).—Omitted.

4. *Yttrium* (88.90).—Reaction takes place quietly, the mass glowing and blackening. Reduction apparently complete.

5. *Lanthanum* (138.6).—Reaction quiet, accompanied by tolerably strong glowing of the mixture. Apparently complete reduction.

6. *Ytterbium* (172.6).—Omitted.

B.—Sub-Group (Oxide R<sub>2</sub>O<sub>3</sub>).

1. *Gallium* (69.9).—Reduction accompanied by hissing noise and partial ejection of the mass.

2. *Indium* (113.6).—Violent detonation. A lower oxide could not be obtained by employing small quantities of magnesium.

3. *Thallium* (203.70).—Thallium oxide decomposes into the lower oxide and oxygen at a temperature below that necessary for reduction by magnesium. The suboxide formed is either not reduced at all or only incompletely

by magnesium, though the carbonate decomposes almost with explosive violence. The action of magnesium upon the oxides of the elements of the chief group increase only to a slight extent with the increase of the atomic weight. With boron there is an unmistakable tendency to combine with magnesium and hydrogen, and the acid character is also exhibited by the simultaneous formation of acid borates. Similarly, in the reduction of aluminium, a deficiency of magnesium results in the formation of a lower oxide before that of an aluminate.

The reducing action of magnesium on the oxides of the elements of the sub-group is very powerful, and increases with the atomic weight. The exceptional behaviour of thallium is only apparent, the previous decomposition of the thallium oxide sufficiently accounting for it.—O. H.

*Properties of Aluminium.* A. E. Hunt, J. W. Langley, and C. M. Hall. Eng. and Mining J. 49, 284—285, 314—316, 334—336.

*Purity of the Metal.*—The earlier information on the physical properties of aluminium is not very reliable, owing to the impurity of the metal, which rarely contained more than 95 per cent. of aluminium. More recently metal of 99 per cent. has been manufactured by Deville's process and by the electrolysis of alumina dissolved in a fluoride bath. In the first process the impurities are half silicon and half iron. In the latter they are mainly silicon. The latter impurity occurs chiefly in the graphitoid form.

*Specific Gravity.*—A table of the specific gravities of the metal, from various sources and variously treated, is given. The following values may be useful. Taking the specific gravity of aluminium as 1, that of soft steel is 2.95, of copper 3.6, of ordinary high brass 3.45, of nickel 3.5, of silver 4, of lead 4.8, of gold 7.7, a sheet of aluminium 12 in. square and 1 in. thick weighs 14.03 lb. A bar of aluminium 1 in. square and 12 in. long will weigh 1.17 lb. A bar of aluminium 1 in. in diameter and 12 in. long will weigh 0.918 lb. A cubic inch of cast aluminium weighs 0.092 lb. A cubic foot of cast aluminium weighs 158.967 lb. Aluminium has the tensile strength of cast iron, with only about one-third the weight. It may, therefore, replace the latter with advantage in the moving parts of machinery.

*Action of Heat.*—Pure aluminium melts at about 1,200° F. One per cent. of iron raises the melting point over 100°. Aluminium passes through a pasty stage, commencing at 1,000° F., and can be readily welded by pressure. It is very red-short at this temperature, and will not bear hammering. The metal is not injured by heating for a moderate period. The melting point of aluminium is given as 700° C. by Roscoe and Schorlemmer; by Deville it is stated to be higher than that of zinc, but lower than that of silver. Aluminium in a compact mass is chemically unaltered when heated under ordinary conditions by combustion of carbon at any furnace temperature, although it will burn readily in oxygen when in the attenuated form of thin foil. The presence of a flux is absolutely injurious, as it assists the absorption of silicon and iron from the walls of the crucible. When fused, the metal becomes coated with a thin film of oxide, which protects it from further action. It is best melted in plumbago crucibles, from which it absorbs .25 per cent. of silicon at each melting. Where it is desired to retain the purity of the metal, it may be melted in crucibles brasqued with pure magnesia mixed with sufficient tar to give it plasticity. Aluminium is most malleable between 200° and 300° F., but can be rolled or drawn cold with frequent annealing. It becomes red-short at 400° F. The annealing of aluminium is best carried out by heating it to a temperature of 800° F. At this temperature a bar of iron appears of a dull red in weak daylight. A piece of pine-wood drawn across the metal should leave a streak of carbon, which should burn away very slowly. Small articles may be annealed by plunging them in boiling linseed oil or water, and allowing them to cool with the liquid.

Aluminium when slowly cooled from fusion crystallises in octahedra. When pulled in a testing machine a granular fracture is obtained. If aluminium is cooled rapidly the

silicon does not combine with the metal and its hardness is not increased. The specific gravity of aluminium is 2.143 (Richards). The coefficient of linear expansion of metal of 98.52 per cent. (Pittsburg Reduction Company) is .0000206 per  $1^{\circ}$  C. between  $0^{\circ}$  and  $100^{\circ}$ , a value approaching that for tin and higher than that for copper. Sound castings of aluminium can be obtained in dry sand moulds preferably lined with plumbago. The metal should be heated to a temperature very little above its melting point and should be poured quickly. The shrinkage of the above metal is  $0.2\frac{1}{2}$  inch to the foot (2.26 per cent. of the length of the mould).

Aluminium, when exposed to the air, becomes coated with a very thin layer of oxide, which does not interfere with the surface polish but prevents the oxidation from going further. It is equally stable in the presence of steam and hot air. It is unacted upon by sulphuric acid and nitric acid if these acids be free from hydrochloric acid. It is not attacked by sea-water nor by weak solutions of salt in acetic acid.

Aluminium is a very soft metal, but is hardened by cold working. Castings can be hardened by being drop-forged in dies if not required to bear a great weight. As above mentioned aluminium is not hardened by chilling.

Aluminium is very ductile and malleable. In fact the latter property is a very good test of its purity. It is absolutely non-paramagnetic if it contain less than 0.05 per cent of iron. With .15 per cent. of iron a slight polarity was observed. With 2 per cent. the polarity was distinct.

The tensile, crushing and transverse tests of aluminium vary considerably according to the amount of work which has been put upon the metal. The flow of the metal under tensile tests is very local, the percentage of elongation decreasing very rapidly as it is calculated in increasing lengths from the point of fracture. Metal containing:—

	Per Cent.
Aluminium from.....	97—99
Silicon, graphitic, from.....	0.10—1.00
Silicon, combined.....	0.90—2.80
Iron.....	0.04—0.20

gave the following average tension and compression tests:—

Elastic limit per square inch in tension:	Pounds.
Castings.....	6,500
Sheet.....	12,000
Wire.....	16,000
Bars.....	14,000
Ultimate strength per square inch in tension:	
Castings.....	15,000
Sheet.....	24,000
Wire.....	30,000
Bars.....	28,000
Percentage of reduction of area in tension:	Per Cent.
Castings.....	15
Sheet.....	35
Wire.....	60
Bars.....	40
The modulus of elasticity of aluminium:	
In castings, is about.....	11,000,000
Sheets.....	13,000,000
Drawn wires.....	19,000,000
Elastic limit per square inch under compression in cylinders with length twice the diameter.....	3,500
Ultimate strength per square inch under compression in cylinders with length twice the diameter.....	13,000

Taking the tensile strength of aluminium in relation to its weight, it is as strong as steel of 80,000 lb. per square inch.

**Conductivity and Electro-chemical position of Aluminium.**  
—Aluminium is more electro-positive than any other metal, with the exception of the metals of the alkalis and alkaline earths. It is electro-positive to sodium in oxygen compounds since it liberates that metal from its oxide. When combined with sulphur it is electro-negative to iron. Its heat of combination with oxygen is about three times that of an equivalent quantity of carbon ( $C + O_2 = 96,000$  h.u.,  $Al_2 + O_3 = 388,000$  h.u.) The great affinity of aluminium for oxygen is made use of in the removal of oxygen from

molten steel by its means. At ordinary temperatures it resembles platinum and gold, and in nitric acid is nearly as electro-negative as platinum or carbon. It has been suggested that at low temperatures the atoms of aluminium are combined with one another. The following is the electrical resistance of aluminium (of 98.52 per cent.) as determined by C. K. McGee, of the University of Michigan:—

Specimen used.	Diameter in Inches.	Resistance in "Legal Ohms." of 1 Yard.	
		At $14^{\circ}$ C. (57 $^{\circ}$ F.).	At $76^{\circ}$ C. (176 $^{\circ}$ F.).
Unannealed Al wire.....	.0325	.05749	.07202
Annealed Al wire.....	.0325	.05484	.06928
Pure copper wire has the following resistance.....	.0325	.0315	.0387

The thermal conductivity of aluminium is nearly inversely as its electrical resistance, the thermal conductivity of aluminium being to that of copper as 1:1.88.

**Action of Impurities.**—Silicon hardens aluminium considerably, increasing its tensile strength without materially decreasing its ductility; it, however, very materially reduces its malleability, and takes away its capacity of taking a high polish and even prevents its retaining whatever polish it has received. Silicon in aluminium oxidises by the action of the atmosphere or of moisture, and if present in proportions above 3 per cent., very soon coats the metal with a dull grey and unsightly varnish. For certain purposes where it is desired to retain the lightness of the metal and where the polish is of no consequence, an alloy containing 6—8 per cent. of silicon is advantageous. As before remarked, the silicon is in the graphitoid state. Were it possible to convert it into the amorphous form a metal having the advantageous hardness without the tarnishing qualities might be obtained. Iron in small quantities hardens aluminium, but at the same time increases its specific gravity and renders it magnetic. It also reduces the malleability and injures the polish of the metal. If equal parts of aluminium and iron are melted together, an apparently true alloy is obtained which, however, on solidifying soon crumbles to powder. Aluminium may be advantageously alloyed with copper. The latter adds hardness and decreases the shrinkage of castings. Carbon combines with aluminium only at very high temperatures and in proportions not greater than 3 per cent. It renders the metal brittle and porous.

Sulphur combines with aluminium only at a very high temperature, and is not usually found in aluminium of commerce. Lead occurs in proportions up to  $\frac{1}{4}$  per cent. It has no appreciable influence on the properties of the metal. Antimony does not unite with aluminium. Chromium alloys readily with aluminium, increasing the hardness and tensile strength of the metal, but decreasing its malleability. Tungsten hardens aluminium, but gives no useful alloys. Platinum unites readily with aluminium, but the alloys are brittle and unsound. The most useful metal for improving aluminium seems to be silver. Five per cent. of silver increases the elasticity and hardness of aluminium without injuring the malleability of the metal. The alloy will also take a very fine polish. The addition of tin to aluminium renders the latter brittle, but it appears that 2 per cent. of aluminium added to tin renders the latter metal harder and more elastic. Cadmium unites with aluminium, giving fusible and malleable alloys; the aluminium is, however, weakened. Bismuth forms brittle, very fusible alloys. Nickel, in quantity, produces brittle alloys. In amounts less than 3 per cent. it increases the hardness without injuring the malleability and ductility of the metal. An alloy of 70 per cent. copper, 23 per cent. nickel, and 7 per cent. aluminium has a fine yellow colour, and takes a high polish. Alloys of zinc and aluminium are very brittle and crystalline. The best solder for aluminium is this alloy of zinc and aluminium, Venetian turpentine being used as a flux. It

does not flow very well, and the soldered surfaces will not withstand hard usage.

*Practical Hints.*—A number of instructions are given for working aluminium.

*Alloys of Aluminium and Copper.*—Ten per cent. aluminium with 90 per cent. copper (called 10 per cent. aluminium bronze) when rolled into plates has an elastic limit of 70,000 to 80,000 lb. per square inch, a tensile strength of from 100,000 to 120,000 lb. per square inch, a reduction of area of from 20 to 40 per cent., with an elongation of from 5—10 per cent. in eight inches. The metal has a beautiful yellow colour, and is capable of taking a high polish. It is unacted upon by dry or moist air. Its specific gravity in castings is 7.84, in rolled sheets about 7.89. Its modulus of elasticity is about 18,000,000 lb. In castings it has a tensile strength of between 70,000 and 80,000 lb. per square inch, with a reduction of area of about 20 per cent. Ten per cent. aluminium bronze melts at about 1,700° F., its melting point being a little higher than that of brass. It solidifies very rapidly and must be poured quickly. Care should be taken to prevent oxidation. Aluminium bronze is a very close-grained dense metal. It can be worked at a bright red heat and does not change at this temperature, hence it is well adapted for blast-furnace tuyères. It can also be remelted several times without loss, this property enabling scrap metal to be easily worked up. It can be brazed with a solder consisting of zinc and copper in equal proportions. One part of solder is used with three parts of a mixture of equal parts of borax and cryolite.

*Aluminium in Wrought Iron.*—Aluminium has been used with wrought iron in the well-known Mitis process for making castings in that metal.

*Aluminium in Cast Iron.*—The effect of aluminium on cast iron is to convert the combined carbon into graphite, thus changing white iron into grey. It has been used in proportions of  $\frac{1}{10}$  to 2 per cent. with good results.

*Aluminium in Steel.*—The effect of the metal on steel is to convert the combined carbon into graphite and to destroy the hardening action of the carbon in tool steel. In structural steel of .2 per cent. to 1 per cent. of carbon a small amount of aluminium increases the tensile strength without decreasing the ductility. When aluminium high in silicon is added to steel the silicon becomes converted into the amorphous form. It is frequently stated that aluminium lowers the melting point of steel and renders the steel more fluid and freer from blow-holes. It can be most advantageously used in proportions of from  $\frac{1}{10}$  to 3 per cent.

Methods of preparing alumina are given.—H. K. T.

#### *Use of Alloys in Metal Work.* W. C. Roberts-Austen. Soc. Arts J. 1890, 689.

THE subject of this paper is the colouring of metal-work, and its principal theme is the clever manner in which the Japanese impart a charming variety of colours and tints to bronzes and other metals by means of alloys and certain pickling solutions. They mix together copper, silver, gold, lead, and iron, and by varying the proportions of the different ingredients produce effects which are at once artistic and permanent. One of their chief mixtures is called "shaku-do," of which the analysis is as under:—

Copper .....	94.50
Silver .....	1.55
Gold .....	3.73
Lead .....	.11
Iron and arsenic .....	Traces
Total .....	99.89

Another analysis of this mixture shows:—

Copper .....	95.77
Silver .....	.08
Gold .....	4.16
Total .....	100.01

This mixture has been used by the Japanese for very large works, some of which, cast in the seventh century, are specially remarkable. Another important alloy is called "shibu-ichi," of which the following are said by Roberts-Austen to be typical analyses:—

Copper .....	67.31
Silver .....	32.07
Gold .....	Traces
Iron .....	.52
Total .....	99.90
Copper .....	51.19
Silver .....	48.93
Gold .....	.12
Total .....	100.15

The precious metals used in these mixtures vary considerably, but in the shaku-do the gold is made to yield a beautiful purple patina when treated with the pickling solutions, whilst the shibu-ichi possesses a very attractive silver-grey tint of its own. As to the pickling solutions the following are made up respectively as given here, and are used boiling:—

—	I.	II.	III.
Verdigris .....	438 grains	87 grains	220 grains
Sulphate of copper ..	292 "	437 "	540 "
Nitre .....	..	87 "	..
Common salt .....	..	146 "	..
Sulphur .....	..	233 "	..
Water .....	1 gall.	..	1 gall.
Vinegar .....	..	1 gall.	5 fl. drachms

The action of these solutions is said to be remarkable, especially in bringing out the tints and colours of which the Japanese workers in metals are so fond. The author, after enumerating some of these effects, asks, "Why cannot we in this country use the alloys named in order to produce similar results?" The question is quite pertinent and proper. We should be very pleased indeed if some of our gold and silver smiths, for instance, could see their way to following up, and possibly improving upon, the Japanese practice in these respects. Up to the present time only one English artist (Alfred Gilbert) has done anything of the kind, but the Americans and French have taken hold of the idea, and are working it already with conspicuous success. In Paris, Christoffe, and in New York, Tiffany, are producing beautiful pieces of work by using alloys, and the latter are even said to have discovered the secret of the celebrated Japanese "lobster red." What these firms can do we also ought to be able to perform, and as the abolition of the plate duties will be calculated to increase competition from without, it is absolutely necessary that our metal workers—of all grades—should bestir themselves in order to meet the threatened increase of rivalry from foreign and American houses. Roberts-Austen's elaborate explanation of Japanese work seems to us to offer an entirely new field to our artists and artisans. It is to be hoped that they will lose no time in making practical use of the information.

#### PATENTS.

*Improvements in Apparatus for Coating with Tin, Lead, or other Metals or Alloys.* H. F. Taylor, Neath. Eng.  
Pat. 6594, April 17, 1889. 11d.

THESE improvements relate to the cradle and nipping apparatus for immersing the plate to be coated, and refer especially to the coating apparatus described in Eng. Pat. 4350 of 1888. The nipper hook which seizes the plate is continuous with a tumbler or short lever having two faces

set at an angle. These faces come in contact with tappets which open and close the nipper hook as the cradle moves up and down, the plate being nipped between the nipper hook and cradle. In order to maintain the nipper hook in its open or closed position a rod is provided which is held in a yielding manner at its upper end, and whose lower end presses into a recess formed in the tumbler, and maintains the hook on one or the other side of its pivot centre.—H. K. T.

#### *Improvements in obtaining Alloys of Aluminium.*

G. Bamberg, London. Eng. Pat. 7666, May 8, 1889. 4d.

IRON or copper rich in carbon or silicon is introduced while in a molten condition into a Bessemer converter. The temperature is raised by blowing air through the metal in the usual way. Vapour of aluminium chloride or of the double chloride of aluminium and sodium are then introduced. Zinc or lead can be used instead of iron or copper.

—H. K. T.

#### *Improvements in obtaining Aluminium or its Alloys.*

G. Bamberg, London. Eng. Pat. 7667, May 8, 1889. 4d.

METALLIC zinc and aluminium chloride (or the double chloride of aluminium and sodium) are heated in separate retorts to a white heat, and the vapours produced are allowed to mix in a third retort or chamber also maintained at a high temperature. The zinc is used in excess. The resulting alloy of aluminium and zinc is heated to about 1,040 C. for the purposes of volatilising the zinc. (See preceding abstract.)—H. K. T.

#### *Improvements in the Production of Alloys of Aluminium.*

R. E. Green, London. Eng. Pat. 9358, June 5, 1889. 4d.

A MIXTURE of pig iron, aluminium fluoride, or cryolite and sand, silicate of potash, or "silicon disulphide" is heated in a crucible to a white heat until the silicon fluoride is driven off. The iron-aluminium alloy is then run into moulds.—H. K. T.

#### *Improvements in Apparatus for treating Metallic Ores or Compounds with Chemicals.*

S. Alley, Polmadie. Eng. Pat. 9592, June 11, 1889. 8d.

See under 1, page 726.

#### *Improvements in the Treatment of Zinc Ores.*

The Alkaline Reduction Syndicate, Limited, and A. B. Cunningham, Hebburn-on-Tyne. Eng. Pat. 9987, June 18, 1889. 4d.

ZINC ores containing silica and lead which have hitherto proved troublesome to smelt, are fused with caustic soda, the quantity required varying with the percentage of these impurities; e.g. an ore containing 30 per cent. of zinc and 30 per cent. of lead is treated with 50 per cent. of caustic soda. The lead is reduced to the metallic state and can be drawn off; it contains any silver or gold that may be present. The alkaline melt is lixiviated preferably by running it direct into water, the sodium silicate, sulphide, and excess of caustic soda being dissolved, and the purified "zinc-compound" left for reduction in the ordinary way, or for use after purification as a pigment. Iron and copper, if present in the original ore, remain in the treated ore, but do not hinder the reduction to metallic zinc. The alkaline liquor may be causticised with lime, the silica being thereby precipitated, concentrated and used again.—B. B.

#### *Improvements in the Manufacture of Copper Alloys.*

J. C. Bull, Belvedere. Eng. Pat. 10,154, June 21, 1889. 4d.

MAGNESIUM carbonate is mixed with carbonaceous matter and granulated copper, and the mass run down in a furnace,

an alloy of copper and magnesium containing about 2 per cent. of the latter metal being obtained. By repeating the process, using the product of the first melt instead of pure copper, an alloy containing about 6 per cent. of magnesium results. Suitable proportions are eight parts of copper, two of charcoal and four of magnesium carbonate. A preferable plan however is to use magnesium chloride as the source of magnesium. It is mixed with half its weight of common salt, to delay its decomposition until the melting point of the copper has been nearly reached, and the same quantity of quicklime, and the charge thus made gives an alloy containing as much as 5 per cent. of magnesium at the first melting, and 8 per cent. on repetition of the process.

Suitable proportions are eight parts of copper, two of carbon, two of magnesium chloride, one of common salt, and one of quicklime.

In both processes the temperature is kept as closely as possible below the melting point of the copper or alloy for one hour, the charge then melted and poured at the end of half an hour.—B. B.

#### *Improvements in the Wet Method of Extracting Gold and in Apparatus to be used therewith.*

J. H. Pollok, Glasgow. Eng. Pat. 10,298, June 25, 1889. 8d.

AFTER the ore has been treated with chlorine in the chlorination process, caustic or carbonated alkalis, or alkaline earths, or reducing agents which will absorb the chlorine without precipitating the gold, are added, thereby preventing the escape of noxious fumes. The charge is then drawn off and filtered and the filtrate is returned to the generator together with a fresh quantity of ore, a small quantity of bleaching powder and sufficient acid to liberate the whole of the chlorine. The process is repeated until the liquor is sufficiently rich in gold, which is then precipitated. A combined apparatus is described, consisting of a spherical generator and a filter bed connected by means of pipes and steam injectors with a series of vats. The same liquor is used repeatedly as above described, the washings from the ore being collected separately. The washings and concentrated gold solution are then mixed and the gold precipitated. (See this Journal, 1889, 444—446.)—H. K. T.

#### *Improvements in the Treatment of Gold and Silver Ores by the Hydro-metallurgical or Wet Method.*

J. Harper, London. Eng. Pat. 10,394, June 26, 1889. 8d.

THE ore is first roasted with salt and poor copper ore. It is then introduced into a chlorinator together with water or a solution of copper chloride, bleaching powder, and sulphuric acid. After a sufficient treatment the charge is transferred to a closed filter, air pressure being applied above, or suction below the filter. When the amount of gold in the ore is less than 1½ oz. per ton the treatment in the chlorinator is omitted. To the clear liquid from the filter sufficient calcium polysulphide is added to precipitate the gold and silver. The latter are filtered off and the filtrate is stored in a tank to be used again in a hot or cold condition in the first filter with a fresh quantity of ore. The filter beds are composed of layers of basket work, moss, pebbles, and coarse sand, the basket work being uppermost and the sand filling the interstices. A system of tanks and leaching vessels is described.

—H. K. T.

#### *Process for Removing Gases and Foreign Impurities from Metals or Alloys.*

J. L. Schenius, Stockholm, Sweden. Eng. Pat. 3440, March 4, 1890. 6d.

THE process is designed to increase the homogeneity of iron and other metals by subjecting them while fluid or semi-fluid to the action of "centrifugal force" in a "rotator." A machine adapted for this purpose consists essentially of a vertical shaft capable of being rotated, carrying a horizontal disc or radial arms at its upper end; from the edge of the disc or the ends of the arms moulds for receiving the molten metal can be hung so that when the whole is revolving they may fly out like the arms of a governor and eventually, when a sufficient speed is attained, become horizontal. By



this means the metal they contain is driven to the further extremity of the mould, while any lighter impurities, e.g. slag or gases, are forced to the near end, the metal thus gaining in homogeneity. In some cases the metal may be "centrifugalled" in the ladle before casting.—B. B.

*A Process for Concentrating Magnetic Iron Ore.* H. J. Haddan, London. From G. Conkling, Glensfalls, New York, U.S.A. Eng. Pat. 4690, March 25, 1890. 6d.

THE comminuted ore, suspended in water, falls vertically in contact with an endless band which moves in the opposite direction. Behind the endless band an electro-magnet is placed. The magnetic particles are attracted to the band. They are carried upwards and are scraped off into a suitable receptacle.—H. K. T.

*An Improved Process to be Employed for Hardening Steel.* J. H. Bramwell, Sheffield. Eng. Pat. 6529, April 29, 1890. 4d.

THE patentee hardens steel in a concentrated solution of common salt at a temperature of 100°–150° F., by immersion and agitation in the case of tools that are to be hardened uniformly, or by the local application of a stream of the liquid to such articles as are to be hardened in parts.—B. B.

## XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

### PATENTS.

*Improvements in Galvanic Cells.* R. A. Burnell, London. From T. Wilms, Hamburg, Germany. Eng. Pat. 6110, April 15, 1889. 8d.

THE cell is a modification of the ordinary Leclanché. The carbon is in the form of a large rod with deep longitudinal grooves. In one of these is placed a zinc rod suitably insulated; in the others are porous sheaths containing broken manganese peroxide. These sheaths project partly into the air to absorb oxygen for depolarising purposes and can be turned about in their grooves. For dry cells, a carbon of the same shape is used, but the manganese peroxide not only fills the grooves, but forms a layer all round. This layer is surrounded by absorbent material soaked with sal-ammoniac. A square perforated zinc tube encloses the absorbent material and is surrounded by more of the absorbed sal-ammoniac.—E. T.

*An Improved Exciting Liquid for Electric Batteries.* M. Süssmann, Berlin, Germany. Eng. Pat. 9183, June 3, 1889. 4d.

FERRIC chloride is used as an electrolyte with the addition of about 14 per cent. of zinc chloride, if zinc is the positive metal used. Without the chloride of the positive metal the current very rapidly falls off.—E. T.

*Improvements in Grids or Containers for the Elements or Plates of Secondary Batteries and Moulds to be Used in their Manufacture.* The Electrical Power Storage Co., Lim., and P. J. Rea, London. Eng. Pat. 9723, June 12, 1889. 8d.

THIS invention relates to moulds for the casting of secondary battery grids or containers, such that the active material contained shall not be liable to split or fall away. In the direction at right angles to the barrel section of the mould, the plug is further held by small projections cast

on the corresponding walls of the grid. Thus the active material is securely held even if it should split, which is not likely to occur. Moulds for various other sections of grid are described, the two parts of the mould, wherever under-cut sections are adopted, being separated in an oblique direction.—E. T.

*Improvements in Apparatus for Separating Gold and other Metals from their Ores by the Aid of Electricity.* G. J. Atkins, Tottenham. Eng. Pat. 10,061, June 19, 1889. 6d.

THIS invention is for improvements in the carbon screw anode of Eng. Pat. 12,295 of 1886. The screw sections are now fixed on a metal shaft, preferably of square section, by being threaded on into position, and having molten lead, type-metal, or other conductor cast in between the bosses of the sections and the shaft, good mechanical and electrical contact being thereby obtained to every section. Similar means are employed for fixing the shell form of anode. All the metal part of the apparatus is of course protected by insulating material, and the carbon part preferably impregnated with black earth-wax.—E. T.

*Improvements in the Manufacture of Sodium, Potassium, and Magnesium.* T. Parker and A. E. Robinson, Wolverhampton. Eng. Pat. 11,707, July 23, 1889. 4d.

THE carbonates, hydrates, or oxides of sodium, potassium, or magnesium are mixed with a quantity of finely divided carbon sufficient to reduce them to the metallic state and heated by means of "an electric arc or electrical resistance," the metal volatilising and being condensed by suitable means. The electrodes used may be of iron, and a liquid bath of iron or other metal may be maintained at the base of the furnace for the purpose of diffusing the heat. The charge may be heated before introduction in order to economise current.—B. B.

*Improvements in Treating and Rectifying or Ageing Alcohol or Alcoholic Liquors or the like by Electricity.* A. de Méritens, Paris, France. Eng. Pat. 14,162, September 7, 1889. 4d.

See under XVII., page 758.

*Improvements in Electric Batteries.* C. Payen, Philadelphia, U.S.A. Eng. Pat. 18,481, November 19, 1889. 8d.

A CERTAIN amount of foreign material, such as zinc or cadmium, or their chlorides, is added to lead chloride, and the whole cast as a plate in a two-part mould. As the mass cools, crystals form and extend inwards from the edges. A metal supporting frame is then cast round the plate and the salts reduced by chemical action or by electrolysis. The foreign metal is removed in this reduction or by fusion, volatilisation, or sublimation, the plate being well washed afterwards. Such plates possess great porosity and conductivity. Instead of using the metal supporting frame, contact may be made by platinum strips cast into the plates.—E. T.

*Improvements in Electric Dry Batteries.* C. R. Bonne, London. From H. Meinecke, jun., Breslau, Germany. Eng. Pat. 19,129, November 28, 1889. 4d.

"The invention consists in saturating the carbon electrode in sulphate or nitrate of silver and in bringing it in contact with sal-ammoniac or with other liquid or substance saturated with liquid, in order to form chloride of silver, which is deposited in finely-disintegrated particles on the carbon. The intervening space between the zinc and carbon elements of the battery is filled with porous or absorbent material, to contain the sal-ammoniac or other liquid."

*Improvements in Electric Insulating Compounds.* J. B. Williams, Oakland, California, U.S.A. Eng. Pat. 1878, February 4, 1890. 6*d*.

AN insulating material for ordinary wires is made by mixing 60 parts by weight of india-rubber, 10 of a paraffin—preferably that obtained from ozokerite or mineral waste—22 of shellac, and 8 of sulphur. For a harder material for stationary conductors, inert material, such as silica, or a bituminous material that is solid and brittle when cold, may be added up to 30 per cent., or to 50 per cent. for telegraph insulators, &c. The amount of india-rubber may be varied, according to the degree of flexibility desired. The india-rubber is dissolved in "benzole" or other volatile solvent, the paraffin added and melted in; the solvent is distilled off, and the other ingredients incorporated with the help of steam rollers. Vulcanisation may be resorted to if desired, but is not considered so good.—E. T.

*Improved Storage or Secondary Batteries.* W. B. Hollingshead, East Chester, N.Y., U.S.A. Eng. Pat. 3008, February 25, 1890. 6*d*.

THE two electrodes are of iron and manganese peroxide respectively, the electrolyte being potassium bichromate, permanganate or cyanide, or other suitable solution. In charging, the peroxide is reduced to a lower oxide by hydrogen, and the iron oxidised. With bichromate electrolyte, chromic anhydride is deposited on the iron, and caustic potash is produced at the other electrode. The electrodes may be rods, or granules suitably encased.—E. T.


*Improvements in Storage or Secondary Batteries.* C. J. Hartmann, New Jersey, U.S.A. Eng. Pat. 3013, February 25, 1890. 8*d*.

IN one method of construction, each electrode is made up of three parallel and similar lead sheets, separated by a short distance from each other, the spaces between them being packed with the active material. To increase the surface, pockets of the shape of step pyramids are stamped in each plate in exactly similar positions, so that the pockets of A project into the inside of those of B. Holes are punched all over the surface of each sheet by a sharp tool, so as to leave a considerable burr. In another construction, the outside sheets are merely furnished with small corrugations, and are joined at the sides, so as to form a rectangular case. The central sheet is, however, furnished, in addition, with much larger corrugations, so that it consists of corrugated sheet made from metal already finely corrugated. Each sheet is here also covered with burr holes. The active material is either packed into the plates in a dry condition, or after mixing with gum or rosin made semi-fluid by an alkali. The plate is placed in an acid bath, which neutralises the alkali, and leaves the active material in a highly spongy condition.—E. T.

*Improvements in Secondary or Storage Batteries.* S. C. C. Currie, Philadelphia, U.S.A. Eng. Pat. 3439, March 4, 1890. 8*d*.

THE metallic portion of each electrode consists of a frame like an ordinary grid, with the top and all the meshes cut out, the sides, however, being farther apart at the top than at the bottom. A slab of active material is placed in this frame from the top and retained by its V-shaped edges, which fit into corresponding grooves in the two sides of the frame. India-rubber bands are placed vertically round the whole, and act as separators besides keeping the whole plate together. By drawing the bands to one side, the slabs can be drawn vertically out of the frames without disturbing the rest of the plates. A second frame of ebonite is sometimes placed outside the metal one.—E. T.

*Improvements in and Relating to Electric Secondary Batteries or Accumulators.* H. H. Lake, London. From T. Ewing, New York, U.S.A. Eng. Pat. 3455, March 4, 1890. 8*d*.

EACH plate is built up of a series of narrow strips of lead, one above another, each bent to the shape  indicated, the whole being suitably bound round. Each plate is supported on a strip of hard rubber of the same length and width as the plate, and furnished with short legs to enable the liquid to circulate under it. Vertical separating rods are employed, all the plates being bound together by horizontal belts of hard rubber, passing through both separators and plates. The lead oxide or other material is first mixed with sulphuric acid or other agent that will cause it to expand as much as possible; it is then heated to expel moisture, pulverised, and packed into the plates.

—E. T.

*An Improved Electrolytic Solution for Galvanic Batteries.* L. Weigert, Berlin, Germany. Eng. Pat. 3717, March 8, 1890. 4*d*.

CELLS containing the chlorides of iron, copper, &c., are known to be inconstant owing to the formation of insoluble salts. To remedy this defect the inventor adds oxidising agents, such as hypochlorites, to saturated solutions of the above mentioned salts. These agents are further said to have a regenerating effect on the electrolyte, and the cell can be made up as a single fluid one.—E. T.

## XII.—FATS, OILS, AND SOAP MANUFACTURE.

*On the Examination of Wax.* H. Rottger. Chem. Zeit. 14, 606—607.

See under XXIII., page 774.

*The Melting and Solidifying Points of some Fatty Bodies and Mixtures.* A. Terceil. Bull. Soc. Chim. 1890, 3, 195—200.

FOR the determinations given in the table below, there was used a specially constructed thermometer, graduated to half a degree, with a comparatively large flat bulb of the following dimensions: length 20 mm., width 10 mm., and thickness 5 mm. The fatty substances were melted and small portions transferred on the tip of a fine glass stirring-rod to the flat surface of the bulb. The thermometer was then heated. It was observed that on re-heating the thermometer, higher melting points were obtained than in the first determinations, in consequence of the imperfect adherence at first of the fatty matters to the glass; the points of solidification, on the other hand, were invariable. The fatty bodies examined were all carefully filtered, whilst hot, through paper, and dried for several hours at 110°—120°.

With regard to the results obtained, it will be observed, in the latter part of the table, that the melting and solidifying points of mixtures do not in any case correspond with the mean of the melting and solidifying points of the individual fatty matters present, but are invariably much higher, this being especially the case when one of the substances present melts and solidifies at a high temperature. Further, when oleic acid, petroleum, or paraffin enter into the composition of a mixture, its melting and solidifying points are lower than when neutral fats alone are present, but are still above the mean.

The results obtained are shown in the following table:—

	Parts.	Melting Point.	Commence-ment of Turbidity.	Solidifi-cation Point.
Pork fat .....	..	36	33	32
Veal tallow .....	..	42	36.5	35.5
Beef tallow .....	..	46	38	36
Mutton tallow .....	..	52	38	37
Fatty acids of pork fat ..	..	43	41	39
Fatty acids of veal tallow	..	46	41.5	39.5
Fatty acids of beef tallow	..	49.5	45	43.5
Fatty acids of mutton tallow .....	..	54	49	47
Stearic acid (for candle-making) .....	..	58	53	52
Stearic acid (old) .....	..	59	54.5	53.5
Palmitic acid .....	..	69.5	62	60
White paraffin .....	..	45	43.5	43
White beeswax .....	..	64	63.5	63
Caruaba wax .....	..	85.5	79	78
Beef tallow .....	1	30	24	23
Neatsfoot oil .....	1			
Beef tallow .....	2	30.5	28	27
Neatsfoot oil .....	1			
Beef tallow .....	1	31	26	25
Olive oil .....	1			
Beef tallow .....	2	32.5	28	27
Olive oil .....	1			
Beef tallow .....	1	29	27	25
Oleic acid (96 per cent.)	1			
Beef tallow .....	2	33	30.5	29
Oleic acid .....	1			
Beef tallow .....	1	38	35	30
Petroleum .....	1			
Beef tallow .....	2	40	36	33
Petroleum .....	1			
Beef tallow .....	1	40	38	37
Paraffin .....	1			
Beef tallow .....	2	37.5	35.5	34.5
Paraffin .....	1			
Beef tallow .....	1	43	41.5	40
Paraffin .....	2			
Fatty acids of beef tallow	1	42	40	38
Olive oil .....	1			
Fatty acids of beef tallow	2	45	43	40
Olive oil .....	1			
Fatty acids of beef tallow	1	39.5	37	34
Oleic acid (96 per cent.)	1			
Fatty acids of beef tallow	2	43	39	36
Oleic acid .....	1			
Fatty acids of beef tallow	1	35.5	30	28
Oleic acid .....	2			

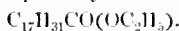
	Parts.	Melting Point.	Commence-ment of Turbidity.	Solidifi-cation Point.
Fatty acids of beef tallow	1	38	36	34
Petroleum .....	1			
Fatty acids of beef tallow	2	42.5	39	37
Petroleum .....	1			
White beeswax .....	1	62	59	58
Olive oil .....	1			
White beeswax .....	2	63	60.5	59.5
Olive oil .....	1			
White beeswax .....	1	59.5	57	56
Olive oil .....	2			
White beeswax .....	1	58	56	54.5
Oleic acid (96 per cent.)	1			
White beeswax .....	2	60	58	56.5
Oleic acid .....	1			
White beeswax .....	1	55.5	52	49.5
Oleic acid .....	2			
White beeswax .....	1	55	53	51
Petroleum .....	1			
White beeswax .....	2	58	55.5	54
Petroleum .....	1			
White beeswax .....	1	62	59	58
Beef tallow .....	1			
White beeswax .....	2	63	60	59.5
Beef tallow .....	1			
White beeswax .....	1	59	56.5	55
Beef tallow .....	2			
White beeswax .....	1	58	53.5	52
Paraffin .....	1			
White beeswax .....	2	60	57	56
Paraffin .....	1			
White beeswax .....	1	55	49	48
Paraffin .....	2			
White beeswax .....	1	81	78	76
Caruaba wax .....	1			

E. B.

*Linoleic Acid.* A. Reformatzky. J. prakt. Chem. 1890, **41**, 529—551.

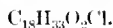
THE author, after a résumé of the work already done on the subject, describes the method of research adopted by him. Linoleic acid was prepared by the method given by Schüler in 1857 (*Annalen*, **101**, 252—256). Fresh cold-pressed linseed oil prepared in the laboratory was saponified with caustic soda, salted out, redissolved, and precipitated by calcium chloride. The lime soap thus obtained was extracted with ether, in which calcium linoleate is soluble, and the ethereal solution treated with hydrochloric acid freeing the fatty acid, which was then purified by separating and washing its ethereal solution and distilling off the solvent. An ultimate analysis having shown the product to be impure, it was neutralised with ammonia and precipitated with barium chloride, and the barium salt treated by the process given above for the calcium salt, save that the final distillation was performed in an atmosphere of carbon dioxide. The

results being still unsatisfactory, the acid was distilled at  $292^{\circ}\text{C}$ . under a pressure of 100 mm., but the distillate on analysis gave numbers corresponding to no probable formula. Norton and Richardson (Ber. 20, 2735) and Hazura and Grüssner (Ber. 21, 436; this Journal, 1888, 506), have obtained by similar treatment products differing from this and from each other, so that the matter is still obscure. Another attempt was made to prepare pure linoleic acid by exposing the crude fatty acids from commercial linseed oil to cold, and purifying the still fluid portion by means of its barium salt. The product, however, yielded much dihydroxystearic acid on oxidation with permanganate, pointing to the presence in it of a considerable amount of oleic acid, and the method was abandoned. Ethyl linoleate was then prepared by acting on an alcoholic solution of crude linoleic acid, with gaseous hydrochloric acid. After washing with water any unacted-on linoleic acid was removed by washing with 80 per cent. alcohol, the product washed once more with water and distilled at  $270^{\circ}$ – $275^{\circ}\text{C}$ . at a pressure of 180 mm., by which process an almost colourless liquid, with a faint yellow tone, was obtained. It was mobile, slightly fluorescent, and possessed of a smell at first pungent but afterwards fairly pleasant; its specific gravity at  $20^{\circ}\text{C}$ ., compared with that of water at  $4^{\circ}\text{C}$ ., was 0.8865. The substance is fairly stable, altering only slowly in air, and its composition was proved by its ultimate analysis giving numbers very fairly agreeing with those required by the formula—

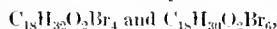


Bromine acted on it, giving a substance melting at  $178^{\circ}\text{C}$ ., apparently a hexabromide; an oily product was also formed.

Linoleic acid was obtained by saponifying the ethyl linoleate thus prepared, acidulating with sulphuric acid, and shaking out with ether; the ethereal solution left on evaporation a brownish mobile odourless oil, which, when dried in a vacuum receiver over sulphuric acid lime and ferrous sulphate, grew darker in shade. Analysis gave (1) carbon = 76.47 per cent.; hydrogen = 11.16 per cent.; (2) carbon = 76.36 per cent.; hydrogen = 11.10 per cent.; instead of carbon = 77.14 per cent.; hydrogen = 11.43 per cent., corresponding with the formula,  $\text{C}_{18}\text{H}_{32}\text{O}_2$ . This result was confirmed by the iodine number (determined by Bültz's modification of Hübl's method (J. prakt. Chem. 1888, 39, 82), the values found being 172.65 and 180.30, instead of 181.43 calculated for  $\text{C}_{18}\text{H}_{32}\text{O}_2$ , and by the analysis of the zinc salt, that being the most stable of those prepared. A final reason for believing linoleic acid to have the formula  $\text{C}_{18}\text{H}_{32}\text{O}_2$ , and not  $\text{C}_{16}\text{H}_{28}\text{O}_2$ , is found in its yielding stearic acid and not palmitic acid when hydrogenised. By long continued heating in a sealed tube with fuming hydriodic acid a product containing iodine corresponding with the formula  $\text{C}_{18}\text{H}_{33}\text{O}_2\text{I}$  was obtained, which on saponification gave an acid which melted at  $70^{\circ}\text{C}$ ., solidified at  $67^{\circ}\text{C}$ ., and gave numbers on analysis of its sodium and silver salts proving it to be stearic acid. Hydrochloric acid acts on linoleic acid even more slowly than hydriodic acid, not even so much being absorbed as corresponded with the addition product—



Linoleic acid when dissolved in glacial acetic acid and treated with bromine in the cold yields two bodies—



the former being a viscid oil, the latter a crystalline solid melting at  $177^{\circ}$ – $178^{\circ}\text{C}$ ., and solidifying at  $175^{\circ}\text{C}$ .

On oxidation with potassium permanganate in dilute alkaline solution according to the method of Saytzeff, (this Journal, 1888, 506) linoleic acid yields tetrahydroxystearic acid  $\text{C}_{18}\text{H}_{36}\text{O}_6$ , as the main product, azelaic acid,  $\text{C}_9\text{H}_{16}\text{O}_4$ , being formed in smaller quantity as well as a little formic acid.

The composition of the drying oils as determined from the products which they yield on oxidation with potassium permanganate in alkaline solution, is a matter still in dispute between the author and Hazura. The researches of the latter, alone and in conjunction with other chemists, have led to the belief that small quantities of linolic and isolinolic acids (this Journal, 1888, 506) are formed. The author

considers that their formation is not characteristic, but is due to some secondary reaction governed by alterations in the conditions of oxidation. He also doubts their being hexahydroxy acids and prefers to regard the substance he obtained as an anhydride derived from two molecules of tetrahydroxystearic acid. The conclusions which are finally arrived at are that the formula for linoleic acid is not  $\text{C}_{16}\text{H}_{28}\text{O}_2$ , but  $\text{C}_{18}\text{H}_{32}\text{O}_2$ , and that the hypothesis that it contains two doubly-linked atoms best explains its reactions.

—B. B.

## PATENTS.

*Improvements relating to Toilet and Cleansing Soaps.* J. Snowden, London. Eng. Pat. 9201, June 5, 1889. 4d.

TEX to 20 per cent. of the finer kinds of asbestos in a fibrous or pulverulent form are incorporated with toilet soaps, and 20–25 per cent. of the coarser varieties with industrial or domestic soaps, the product being termed "Amianthine soap."—B. B.

*Improvements in the Manufacture of Soap and Glycerin.* J. Y. Johnson, London. From M. Duclos, Paris, France. Eng. Pat. 4809, March 27, 1890. 4d.

IN order to obtain the glycerol resulting from the saponification of fats in the ordinary course of soap-making free from the alkaline salts that accompany it when it is recovered by merely boiling down the spent lyes, the patentee saponifies his material with milk of lime (1 of quicklime to 10 of water), effecting his purpose by sufficient dilution and vigorous boiling by open superheated steam, without having recourse to the use of excess of lime (14–15 per cent. instead of the 10 per cent. he employs) or of pressure, as in the autoclave process. An acid lime soap is formed, and the liquor separated from it together with the washings contain almost the whole of the glycerol, which can be obtained of good quality simply by evaporation. The lime soap is decomposed by sulphuric or hydrochloric acid, and the freed fatty acids, which are of good quality, used for making soap in the ordinary way.—B. B.

## XIII.—PAINTS, PIGMENTS, VARNISHES, AND RESINS.

### PATENTS.

*An Improved Preservative and Waterproof Coating for Metals, Timber, Masonry, Concrete, or other Structures or Surfaces.* T. Pridham, Petersham, New South Wales. Eng. Pat. 18,286, November 15, 1889. 4d.

A MIXTURE of or containing mineral bitumen and oil or oily materials, rolled or pressed into sheets, applied to the article to be covered by an adhesive solution.—E. G. C.

*Improvements in Electric Insulating Compounds.* J. B. Williams, Oakland, California, U.S.A. Eng. Pat. 1878, February 4, 1890. 6d.

See under XI., page 743.

#### XIV.—TANNING, LEATHER, GLUE, AND SIZE.

*On the Oxidation of Gallic Acid, Tannin, and Quercitannic Acid.* C. Boettinger. *Annalen*, **257**, 248—252.

WHEN gallic or tannic acid is oxidised with nitric acid of moderate dilution, oxalic acid is the chief product; if this be removed by crystallisation and lime added to the residue free from nitric acid, there are formed two lime salts besides the oxalate, one difficultly and the other easily soluble in water. Gallic acid yields only a very little of the first and tannin only a small quantity of the same; the analytical numbers agree with those of calcium trioxylglutarate. The easily soluble lime salt is calcium trioxylbutyrate. By using more nitric acid easily soluble calcium salts may be obtained containing 15—16 per cent. of calcium.

In the same way the acetyl derivative of quercitannic acid yields on oxidation oxalic acid and two acids which form respectively a difficultly soluble and an easily soluble calcium salt. The first is trihydroxyglutaric acid and the second trioxylbutyric acid; by using larger quantities of nitric acid other acids may be obtained which yield salts containing more calcium. (See also this Journal, 1890, 84).—A. L. S.

#### XV.—AGRICULTURE, MANURES, Etc.

*Determination of Nitrogen in Manures.* E. Anbin and J. Quenot. *Bull. Soc. Chim.* 1890, **3**, 322—326.

See under XXIII., page 773.

*Destruction of Clover Silk by Iron Sulphate.* Boiret. *Jour. d. Agric.* 1889, **2**, 779—782. P. Marguerite-Delacharlonny. *Ibid.* 1890, **1**, 182—187.

BOIRET expressed unfavourable opinions with regard to the statements:—that ferrous sulphate killed clover silk without injury to the clover plant; that the composition of

the soil had any influence on the action of ferrous sulphate on plant life, and that mowing clover plants stricken with clover silk was of any use either before or after use of ferrous sulphate. All these opinions Delacharlonny shows to be unfounded and unsupported by scientific evidence, and then summarises the results of his own investigations. He points out that ferrous sulphate is admirably adapted for the extinction of clover silk in all stages of development; that for young clover plants it should be used in the solid form in quantities of from 300 to 400 kilos. per hectare; that in cases where the clover silk has attained complete development the clover plants should be cut down and removed, and then the stubble should be dressed with ferrous sulphate solution varying in strength from 4 to 10 per cent. according to the age of the plant and the composition of the soil, using weak (4 to 5 per cent.) solutions on clayey and sandy soils, and strong ones (10 per cent.) on calcareous land.

Finally, it is considered advisable to dress the cut clover with the same solution before removal, to prevent the spread of the parasite.—D. A. L.

*Tobacco Experiments.* Kentucky Agricultural Experiment Station Bulletin, **28**.

THE experiments covered one acre consisting of one-tenth acre plots. The nature of the soil has been already described (*Bulletin*, **26**, 1; also this Journal, 1889, 555). The land had been in cultivation for many years, and had never been fertilised till spring 1888, when 100 lb. each of acid black (waste bone black treated with oil of vitriol), potassium chloride and sodium nitrate were mixed and sown broadcast over the plot.

1. *The Effect of the Leading Elements of Plant Food on the Production of Tobacco.*—The "leading elements of plant food" are potash, nitrogen, and phosphoric acid.

Two series of experiments were tried; in the first the plots were one-twentieth acre, and in the second one-fortieth acre. In both series the fertilisers were sown broadcast just before the plants were set. The plants were set 22 inches apart in rows 3 ft. wide. Tables are given showing the results of each series; as those of the second series are similar to those of the first, the table showing the latter need only be transcribed. The last column shows the total yield calculated to a perfect stand, i.e. the yield which would have been produced if every plant which was set had succeeded.

No. of Plot.	Fertiliser used.	Amount of Fertiliser. Pounds per Acre.	Yield in Pounds per Acre.						Total Actual.	Total Corrected.
			Bright.	Red.	Lugs.	Tips.	Trash.			
1	Sodium nitrate . . . . .	160	230	450	310	90	530	1,610	1,710	
2	Acid phosphate . . . . .	320	..	490	450	70	490	1,110	1,140	
3	Potassium sulphate . . . . .	160	..	610	580	20	370	1,590	1,620	
4	No fertiliser . . . . .	..	..	200	360	60	540	1,160	1,160	
5	{ Sodium nitrate . . . . . Acid phosphate . . . . .	{ 160 320 }	{ .. .. }	{ 15 200 }	{ 20 200 }	{ 970 20 }	{ 1,295 970 }	{ 1,295 1,230 }	{ 1,230 1,230 }	
6	{ Potassium sulphate . . . . . Sodium nitrate . . . . .	{ 160 160 }	{ 190 .. }	{ 755 705 }	{ 605 425 }	{ 120 30 }	{ 140 290 }	{ 1,810 1,690 }	{ 1,810 1,770 }	
7	{ Acid phosphate . . . . . Potassium sulphate . . . . .	{ 320 160 }	{ 240 .. }	{ 705 400 }	{ 425 360 }	{ 30 40 }	{ 290 570 }	{ 1,690 1,370 }	{ 1,770 1,400 }	
8	No fertiliser . . . . .	..	..	400	360	40	570	1,370	1,400	
9	{ Acid phosphate . . . . . Potassium sulphate . . . . . Sodium nitrate . . . . .	{ 320 160 160 }	{ .. 310 .. }	{ .. 810 .. }	{ .. 420 .. }	{ .. 10 .. }	{ .. 350 .. }	{ .. 2,090 .. }	{ .. 2,050 .. }	

"Acid phosphate" (superphosphate) is South Carolina rock treated with sulphuric acid.

2. *Financial Results.*—All the crops were valued by the same expert, at the following prices per pound:—bright and red leaf, 15 cents; lugs, 6 cents; tips, 8 cents; trash, 2 cents.

The following table shows the results :—

No. of Plot.	Fertiliser used.	Cost of Fertiliser per Acre.	Value of Tobacco per Acre.	Value of Increased Yield of Tobacco per Acre.	Profit or Loss.
		Dollars.	Dollars.	Dollars.	Dollars.
1	Sodium nitrate .....	3'60	138'40	59'70	53'10
2	Acid phosphate.....	4'60	102'40	20'70	16'70
3	Potassium sulphate.....	4'20	135'30	53'60	49'40
4	No fertiliser .....	..	67'20	..	..
5	{ Sodium nitrate..... } { Acid phosphate..... }	7'60	35'25	..	(Loss) 7'60
6	{ Potassium sulphate..... } { Sodium nitrate .....	7'80	190'45	108'75	100'95
7	{ Acid phosphate..... } { Potassium sulphate..... }	8'20	175'45	93'75	85'55
8	No fertiliser.....	..	96'20	..	..
9	{ Acid phosphate..... } { Potassium sulphate..... } { Sodium nitrate .....	11'80	201'20	119'50	107'70

3. *In what Form should Nitrogen be applied?*—Sodium nitrate, ammonium sulphate, and cotton-seed meal were the three forms of nitrogen tried, and they were applied in quantities such that each plot received the same amount of nitrogen. The results were as follows:—

[illegible]

4. *In what Form should Potash be applied?*—It is generally believed that potassium chloride is injurious to the quality of tobacco, especially smoking tobaccos, and therefore sulphate is generally considered to be the best form for this crop. Both were tried with the following results:—

[illegible]

5. *The Amount of Nitrogen required per Acre.*—From the results of the experiments having this object it would seem that 160 lb. of sodium nitrate or 340 lb. of cotton-seed meal or rather more than 120 lb. of ammonium sulphate would furnish nitrogen in sufficient quantity for the crop.

6. *The Amount of Potash Fertiliser required per Acre.*—The experiments detailed in the above table serve to show that rather more than 160 lb. of either sulphate or chloride of potassium would be beneficial to the crop.

The conclusions to be drawn from these experiments are (1) that commercial fertilisers materially increase the yield, and improve the quality of Burley tobacco; (2) that the increase is obtained whether potash, phosphoric acid, or nitrogen is used separately or in various combinations; (3) that potash and nitrogen in combination seem to be required to produce the best results; (4) that there is a handsome profit by using fertilisers in the best combinations; (5) that the effect is the same whether potash is used in the form of chloride or sulphate; (6) that nitrogen in the form of nitrate of sodium seems to produce the best results as to quality.—A. G. B.

## XVI.—SUGAR, STARCH, GUM, Etc.

*Experiments on Gum Arabic and Gum Senegal.*  
L. Liebermann. Chem. Zeit. 14, 665—666.

See under XXIII., page 770.

*On the Isolation of Crystallised Cane Sugar from Maize.*  
J. H. Washburn and B. Tollens. Annalen, 257, 156—160.

MAIZE is ground and extracted with 72 per cent. alcohol containing a little lime, in order to destroy all acids, soluble ferments, &c., which might invert the cane sugar. The extract is distilled and the residue treated with water and one-third ether. The aqueous extract is evaporated to a syrup, this is dissolved in three or four times its weight of water, 20 per cent. alcohol added, and an excess of crystallised strontium hydroxide. The whole is boiled 5—10 minutes, and the excess of strontium hydroxide and the precipitate quickly filtered out and washed with alcohol. This is then suspended in water and carbon dioxide passed through until the liquid is nearly neutral. The filtrate from this is evaporated and the strontium compound precipitated with one-fourth to one-fifth its volume of alcohol. This is suspended in water, the strontium removed by carbon dioxide, and the filtrate treated with animal charcoal and evaporated. The syrup thus obtained will either crystallise spontaneously or after the addition of a little alcohol. The crystals have all the properties of cane sugar, and are undoubtedly this body.

The authors have made complete analyses of American sweet maize in various stages of growth, and among other factors they have determined the amount of nitrogen-free extract, and they find that besides starch, cane sugar, dextrin, glucose, this includes a non-crystallisable body which does not reduce Fehling's solution, not even after treatment with hydrochloric acid.—A. L. S.

*On the so-called Bi-Rotation of the Sugars.* E. Parcus and B. Tollens. Annalen, 257, 160—178.

THE authors have prepared solutions of most of the crystallised sugars which show bi-rotation, and determined the optical activity as soon as possible after solution and the rate at which the activity decreases or increases to the constant value. The solutions were in all cases, except lactose, 10 per cent. in strength; the observations were made in a 20 cm. tube, which was kept at a constant temperature of 20° C. by a water jacket. The chief results are epitomised in the following table; the time is taken from the moment

of addition of the water to the finely-powdered sugar. As complete solution takes quite an appreciable time, these times do not represent the absolute age of a freshly prepared solution.

*Dextrose.*—Greatest angle observed  $[\alpha]_D = +105.16$  in  $5\frac{1}{2}$  minutes, constant value of  $[\alpha]_D = +52.5$  in 6 hours.

*Galactose.*—Greatest angle observed  $[\alpha]_D = +147.48$  in 7 minutes, constant value of  $[\alpha]_D = +80.3$  in 6 hours.

*Lactose.*—Greatest angle observed  $[\alpha]_D = +82.91$  in 8 minutes, constant value of  $[\alpha]_D = +52.53$  in 24 hours.

*Arabinose.*—Greatest angle observed  $[\alpha]_D = +156.65$  in  $6\frac{1}{2}$  minutes, constant value of  $[\alpha]_D = +104.6$  in  $1\frac{1}{2}$  hours.

*Maltose.*—Angle observed  $[\alpha]_D = +118.75$  in 6 minutes, constant value of  $[\alpha]_D = +136.8$  in 5 hours.

*Xylose.*—Greatest angle observed  $[\alpha]_D = 78.61$  in  $4\frac{1}{2}$  minutes, constant value of  $[\alpha]_D = +19.2$  in  $2\frac{1}{2}$  hours.

*Leucrose.*—Greatest angle observed  $[\alpha]_D = -104.02$  in 6 minutes, constant value of  $[\alpha]_D = -92.1$  in 35 minutes.

Other determinations of the constant value for the last sugar gave  $[\alpha]_D = -92.25$  as the mean.

The authors calculate that at the moment of solution the optical activities are, for xylose  $[\alpha]_D =$  above 100, for arabinose about 190, for galactose about 127, and for leucrose above 100. They take objection to the term bi-rotation, as only in the case of dextrose is the maximum activity double the normal, and suggest the terms more-rotation and less-rotation.—A. L. S.

*Action of Lime on Raffinose.* L. Lindet. Bull. Soc. Chim. 1890, 3, 413—414.

ONLY one compound of lime with raffinose exists; 100 parts of raffinose combine with 10 or 11 parts of lime. The solution of this compound does not become turbid on boiling, differing in this respect from calcium saccharate. At a given temperature raffinose dissolves less lime than cane sugar; thus, at 15°, a 12 per cent. solution of raffinose hydrate dissolves 11.58 per cent. of CaO, a similar solution of cane sugar dissolving 25.42 per cent. Again, a 4 per cent. solution of raffinose dissolves 10.14 per cent. of CaO, whilst cane sugar, under the same conditions, dissolves 21.80 per cent. of the same. Calcium raffinolate, like calcium saccharate, is precipitated by alcohol, but is less soluble in this liquid than the latter. The author attempted to base on this circumstance a process for the fractional separation of the two sugars. A sample of molasses from a sugar refinery was mixed with alcohol so that the mixture contained 50 per cent. of alcohol, and then lime was added in fractions. The compositions of the precipitates obtained were as follow:—

	Raffinose.	Cane Sugar.
	Per Cent.	Per Cent.
1st fraction	20.0	80.0
2nd fraction	18.1	81.9
3rd fraction	15.8	84.2
4th fraction	12.3	87.7

—E. B.

*The Action of Mineral Acids on Saccharification by Means of Malt, and on the Fermentation of Starchy Substances.*  
J. Effront. Monit. Scient. 1890, 449—462.

DURING the saccharification of starchy substances on the large scale in distilleries and breweries, the activity of the diastase is greatly checked by the growth of objectionable organisms, especially the lactic and butyric ferments. The growth of these organisms may be checked by the use of



high temperatures or by the addition of mineral acids. The first method is scarcely admissible, for the temperatures most suitable to diastatic conversion—50° to 60° C.—do not check the development of the false ferments. On the other hand, the author considers the use of mineral acids more rational. It is well known that acids in comparatively small quantities retard or completely arrest the lactic and butyric fermentations; and, according to Delbrück, a small amount of acid also accelerates the action of the diastase. The

author has made numerous experiments in order to determine the precise effect of small quantities of different acids on the lactic and butyric ferments, on diastase, and on yeast. A solution of commercial maltose was prepared of 7° Balling. To separate portions, small amounts of hydrofluoric, hydrochloric, and sulphuric acids were added.

The mixtures were then caused to enter into lactic fermentation at the most suitable temperature. The following results were obtained:—

Number of the Experiment.	Acid used.	Quantity added to 100 cc. of Wort.	Lactic Acid formed per 100 cc. of Wort.			
			24 Hours.	3 Days.	7 Days.	10 Days.
1	..	Mgrms. 0	Mgrms. 190	Mgrms. 453	Mgrms. 493	Mgrms. 828
2	HF	2	133	331	507	540
3	..	3	54	153	315	460
4	..	8½	18	45	135	180
5	..	25	..	..	..	..
6	HCl	10	135	378	450	720
7	..	20	90	360	360	450
8	..	80	22	45	63	81
9	..	200	..	..	..	..
10	H <sub>2</sub> SO <sub>4</sub>	10	144	132	468	765
11	..	20	108	414	522	549
12	..	80	36	81	90	99
13	..	200	9	18	36	43
14	..	300	..	..	..	..

The results show that hydrofluoric acid acts much more energetically than the other two acids. A peculiarity about its action is that it is considerably more vigorous during the first 24 hours than it is later on. The effect of these

acids on the butyric fermentation is very similar to the above. In the case of a mixed lactic and butyric fermentation, the following results were obtained:—

#### THE ACTION OF HYDROFLUORIC ACID ON A SIMULTANEOUS LACTIC AND BUTYRIC FERMENTATION.

Number of the Experiment.	Acid added to 100 cc. of Wort.	Total Acid formed per Litre of Liquid.				Butyric Acid.	Butyric Acid per 100 of Total Acid.
		24 Hours. cc. of N.NaHO per Litre.	Two Days. cc. of N.NaHO per Litre.	Three Days. cc. of N.NaHO per Litre.	Four Days. cc. of N.NaHO per Litre.		
1	Mgrms. 0	24	40	56	57	Mgrms. 431	Mgrms. 8'5
2	1	18	30	42	54	352	8
3	2	12	30	37	39	248	7'1
4	3½	7	12	27	30	220	8'3
5	1½	6	11	20	30	176	6'6
6	5	6	10	23	25	132	6
7	5½	5	10	22	23	88	4'3
8	6	5	8	16	24	12	0'62
9	7	4'8	9	13	20	8	0'5
10	8	1'8	4	5	9	8	..
11	10	1	1'5	1'5	3	..	..
12	20	0'3	0'5	0'5	0'5	..	..

The butyric acid was determined after the fourth day of acidification. The total acidity is expressed by the volume of normal soda required to neutralise it; and in the column headed "Butyric acid," the quantity of that body per litre is given. Butyric acid, even in small quantity, considerably

retards the growth of yeast and the yield of alcohol. Lactic acid, on the other hand, seems to act favourably in these respects. Hydrochloric and sulphuric acids behave very similarly to hydrofluoric acid, except that considerably more of them is required to produce a given effect.

It now became necessary to determine whether the small doses of acid added had any deleterious action on the activity of the diastase. For this purpose a cold water extract of green malt was prepared and portions of it were treated with different amounts of hydrofluoric acid, and

heated for seven days to 30° C. At regular intervals, small quantities were withdrawn, allowed to act on starch-paste at 55° C., and the maltose formed determined.

The results are given in the table below:—

No.	Acid added to 100 cc.	At Beginning.		Second Day.		Third Day.		Fourth Day.		Seventh Day.	
		Maltose per 100 cc.	Sacchari-fying Power of the Diastase.	Maltose per 100 cc.	Sacchari-fying Power of the Diastase.	Maltose per 100 cc.	Sacchari-fying Power of the Diastase.	Maltose per 100 cc.	Sacchari-fying Power of the Diastase.	Maltose per 100 cc.	Sacchari-fying Power of the Diastase.
1	0	2.76	100	1.89	68	1.59	57	0.32	12	..	..
2	2	2.73	99	2.55	85	2.32	84	0.7	25	0.57	20
3	3	2.76	100	2.30	87	2.38	87	1.08	40	0.83	30
4	5	2.78	101	2.57	93	2.47	89	1.38	50	1.14	41
5	7	2.88	101	2.61	95	2.53	91	2.21	80	1.18	45
6	10	2.85	103	2.22	80	1.87	68	1.37	50	1.21	44
7	13	2.79	101.08	1.80	65.2	1.77	64	1.23	45	1.23	45
8	15	2.73	99	1.77	64	1.71	62	1.20	44	1.14	41
9	20	2.37	86	1.83	66	1.41	51	0.87	32	0.81	29
10	25	2.34	85	1.74	63	1.38	50	..	..	..	..

All the experiments were carried out under precisely the same conditions. The quantity of maltose formed in the first experiment was taken as a standard and called 100.

From the above results it will be seen that hydrofluoric acid favourably influences the preservation of diastase, and that 7 mgrms. per 100 cc. is the most suitable quantity to use. This amount, however, is not sufficient to check entirely the growth of lactic and butyric ferments; and, on reference to the first table, experiment 5, it will be seen that 25 mgrms. are required for this purpose. Hydrochloric and sulphuric acids in small doses at first exalt the activity of the diastase; but later on they exert a decidedly retard-

ing influence and, therefore, become objectionable. In the above experiments the malt extract was separately heated with the acid, and the small portion of this malt extract subsequently allowed to act on starch-paste would contain only a trace of acid. Further experiments were therefore made to determine the effect of acid on the actual course of the saccharification. To each of several portions of starch-paste (200 cc.) were added 20 cc. of cold water extract of malt, and small quantities of acid. The solutions were then heated for 2½ hours at 55° C. The following results were obtained:—

Number of Experiment.	Name of Acid.	Quantity added to 100 cc. of Solution.	Maltose.	Saccharifying Power of the Diastase.
1	.. .. .	Mgrms.	5.1	100
2	Hydrofluoric acid .....	1.5	5.28	103
3	" .. .. .	3	5.20	101
4	" .. .. .	5	5.22	101
5	" .. .. .	7	4.92	96
6	" .. .. .	15	4.03	79
7	" .. .. .	20	3.66	71
8	Hydrochloric acid .....	2	5.30	101
9	" .. .. .	5	4.68	92
10	" .. .. .	10	4.53	87
11	" .. .. .	30	4.61	91
12	Sulphuric acid .....	5	5.26	102
13	" .. .. .	10	5.21	101
14	" .. .. .	15	4.63	91
15	" .. .. .	20	4.02	79

The normal saccharifying power of diastase, expressed by 100, corresponds to 5.1 grms. of maltose per 100 cc.

obtained in the first experiment. These results show us that the quantities of mineral acids necessary to arrest

lactic and butyric fermentation have an unfavourable action on diastase at 55° C. Their use is, therefore, inadvisable under these conditions. The influence of hydro-

fluoric acid on diastatic conversion at 30° C. was next determined, the results obtained being as follows:—

Number of Experiment.	Acid added to 100 cc.	After 15 Hours.		After 32 Hours.		After 72 Hours.	
		Maltose per 100 cc. of Solution.	Maltose per 100 of Starch Transformation Products.	Maltose per 100 cc. of Solution.	Maltose per 100 of Starch Transformation Products.	Maltose per 100 cc. of Solution.	Maltose per 100 of Starch Transformation Products.
1	Mgrms. 0	3.80	69.50	4.08	74.50	4.06	74.31
2	1.5	3.78	69.50	4.16	75.85	4.17	75.82
3	2	3.82	69.48	4.24	77.15	4.22	77.25
4	2.5	3.88	70.91	4.21	77.25	4.27	77.41
5	5	4.08	74.51	4.32	78.40	4.51	81.93
6	7	4.06	74.10	4.29	78.10	4.92	82.85
7	10	3.86	69.48	4.48	81.92	5.15	93.12
8	15	3.60	66.05	3.76	68.99	3.75	68.80
9	25	2.45	45.20	2.44	45.10	2.44	45.30
10	40	1.84	33.96	..	..	..	..
11	50	1.25	21.60	..	..	..	..

These results show that by the use of 7—10 mgrms. of hydrofluoric acid per 100 cc., a high degree of saccharification is obtainable.

Similar experiments were made with hydrochloric and sulphuric acids. The increase in the yield of maltose was very slight, and not to be compared with that produced by hydrofluoric acid. A similar series of experiments was made with a wort prepared from maize, since raw grain is more likely to be used in practice than pure starch. Four

kilos. of maize were heated for two hours under a pressure of three atmospheres, with about 15 litres of water. The mash was then cooled to 30° C. and mixed with malt extract. Different portions of the mixture were then treated with hydrofluoric acid, and kept at 30° C. for three days. Portions of the solutions were filtered and analysed at the end of 24 and 72 hours. The results are given in the following table:—

Number of Experiment.	Acid added to 100 cc.	After 24 Hours.			After Three Days.			
		Dry Matter per 100 of Solution.	Maltose per 100 of Solution.	Maltose per 100 of Dry Matter.	Dry Matter per 100 of Solution.	Acid per 100.	Maltose per 100 of Solution.	Maltose per 100 of Dry Matter.
1	Mgrms. 0	3.80	0.15	5.94	3.84	9.62	..	..
2	1	4.95	0.40	8.08	5.21	7.3	0.8	15.35
3	2	8.19	4.02	19.63	9.06	6.88	6.08	66.00
4	3	8.00	4.15	51.87	9.00	6.81	6.02	66.88
5	4	9.57	5.03	52.56	9.65	6.21	6.60	68.39
6	5	9.68	5.82	60.12	10.04	5.3	7.07	70.42
7	6	9.87	6.01	60.89	10.45	4.91	7.31	69.95
8	7	9.96	6.00	60.42	10.84	4.48	7.44	68.63
9	8	9.89	6.12	61.88	10.31	4.56	7.51	68.85
10	9	10.31	6.83	66.25	11.01	3.51	7.86	71.39
11	10	10.51	6.84	65.98	11.19	3.00	8.55	74.62
12	12	11.20	8.31	74.19	11.20	3.00	8.31	74.19
13	15	10.62	7.51	70.91	10.76	3.00	7.74	71.95
14	20	9.62	6.54	67.98	9.65	2.8	6.60	68.49

The numbers in the column marked "Acid per 100" represent the number of cubic centimetres of normal soda required to neutralise 100 cc. of the liquid. In the case of the first experiment the liquid after three days' reaction gave a blue colour with iodine. Under the same conditions 2—5 were coloured red, whilst the rest yielded no colour with iodine. A glance at the column headed "Dry Matter" shows that the liquefying power of the diastase increases

with the quantity of acid employed. The solution in experiment No. 11 was fully analysed and found to contain besides the maltose, 1.27 of dextrin and 0.571 of other substances. This corresponds to a yield of 86.79 of maltose per 100 parts of starch transformation products. Another experiment with maize gave even better results, yielding 96 per cent. of maltose and 4 per cent. of dextrin. These results show that the current notions regarding the most

suitable temperatures for diastatic conversion are a little incorrect. It is generally considered that the best conversion is obtained between 50—60 °C., but the above experiments prove that the highest yield of maltose is got at 30 °C. by using hydrofluoric acid. The vigorous diastatic action is no doubt due to the fact that the acid used almost entirely checks the butyric and lactic ferments, so fatal to diastase.

—H. T. P.

*Diastase considered as a Mixture of Maltase and Dextrinase.* H. P. Wijsman, jun. Rec. des trav. Chim. des Pays-Bas. 1890, 9, 1.

THE author has attempted to determine whether diastase of malt consists of two different enzymes or not. He explains the transformation of starch by the action of two enzymes; one, *maltase*, which converts starch into maltose and erythro-dextrin, and the other *dextrinase*, which converts starch into malto-dextrin; erythro-dextrin being further acted on by *maltase* and converted into aerodextrin and maltodextrin being converted by *maltase* into maltose. In order to prove this he adds to a gelatin solution some of Lintner's soluble starch; this yields on cooling a solid transparent jelly. This is cut into thin sheets and small quantities of diastase placed on them. If then these sheets are placed in iodine solution the gelatin is coloured dark blue; in the diffusion field of the diastase there is formed a colourless ring and around this a violet ring. The colourless spot is where both enzymes have acted, the violet ring where the *maltase* alone has diffused and acted.—A. L. S.

*Results of the United States Government Sugar Experiments.* Eng. and Mining J. 1890, 50, 6—7.

IN 1885 the United States Department of Agriculture, under direction of the chief chemist, Dr. H. W. Wiley, began a series of experiments in the manufacture of sugar from sorghum cane. The main idea was to adapt the diffusion process which had been so successfully applied to sugar beets in Europe to the working of cane.

In order to make these experiments on such a scale as to be of practical value, this work was undertaken in conjunction with the large sugar works at Fort Scott, Kan. The first machinery was either imported from Germany or built from European plans, but it was found that, owing to the fibrous nature of the cane, neither the cutting machinery nor the diffusion batteries would work successfully.

It was also found necessary, in order to compensate for the additional water added to the juice by the diffusion process, to provide evaporating appliances that would not only be more economical, but also of correspondingly greater capacity than what was hitherto used with the more concentrated mill juice from the rolls.

The results of these experiments proved very successful, and attracted a large number of people who were interested in the sugar industry. France, Germany, Austria, Algiers, and Chili sent representatives, and delegates from a number of Sugar Planters' Associations were present during the progress of these experiments.

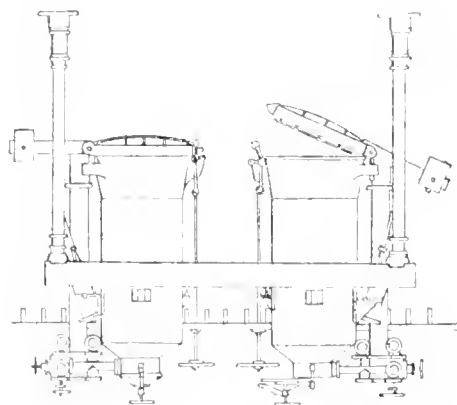
It was singularly fortunate that these sugar works were built adjoining the extensive works of the Fort Scott Foundry and Machine Works Company, whose management had an extensive experience in the field of practical sugar making, and the improvements brought out by that enterprising company during the last few years may justly be said to be the beginning of a new epoch in the history of sugar production in this country.

A series of attempts had heretofore been made to introduce diffusion in Louisiana, but all failed, owing to the difficulty of reducing the cane to a sufficiently fine condition to effect a good extraction of the sugar.

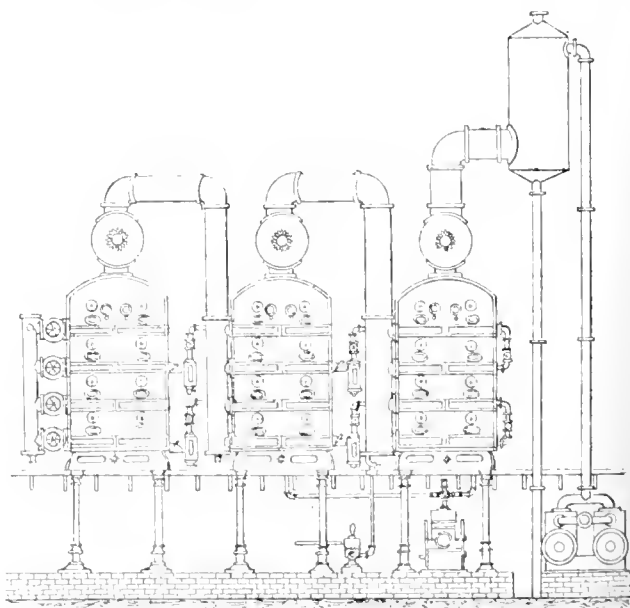
The cutters built by the German manufacturers that worked well on beets were found to have but a fraction of the same capacity when applied to cane, and the knives were constantly clogging, owing to the tough fibre of the cane. Moreover, they had to be fed by hand, making the operation very laborious and expensive.

The cutting machinery built by the Fort Scott Foundry and Machine Works Company overcomes all these difficulties. All that has to be done is to place the cane on an endless carrier, and the fine cane chips are delivered into the cells of the diffusion battery without any further manual labour. Moreover, the capacity of these cutting machines is almost unlimited, a number having been built with a capacity of 500 tons of cane per day.

One of the most important features is the multiple safety-dead knife, which is so arranged that it will at once yield and let through any hard substance like iron without any injury to the cutter. Last year all other cutting devices were abandoned, and the above-described cutters are now used in every sugar factory in this country where diffusion has been adopted.



THE TWO OPPOSITE CELLS OF A DOUBLE LINE DIFFUSION BATTERY.



TRIPLE EFFECT EVAPORATING APPARATUS.

The diffusion batteries have also been greatly improved by these manufacturers, one of the most important of their improvements being the working of the heavy doors by the use of hydraulic cylinders, this work having previously been done by manual labour.

A more recent improvement, but of still greater importance, is their new heating arrangement, by which the heat in the battery can be kept up independent of the circulation, and by the aid of which the juice in the battery may be kept above the temperature where fermentation can take place, if for any reason the work should be suspended for a few hours.

In the field of evaporation the Fort Scott Foundry has also produced much higher results, and the apparatus it manufactures for this purpose is radically different from that which has been commonly in use, and has shown a degree of economy and efficiency not heretofore attained by any evaporating device.

In the so-called standard double or triple effect evaporators, the heating surface is similar to a vertical tubular boiler, the liquid to be evaporated being on the inside of the tubes. This necessitates a deep body of liquor being from four to eight feet deep; and the liquid being confined inside the tubes, the vapour meets with a great deal of resistance. Moreover, the lime which had been used for clarifying the juice produces a deposit on the inside of the heating tubes, where it adheres very firmly and causes much trouble and loss of heat and capacity.

In the apparatus manufactured by the Fort Scott Foundry and Machine Works Company this construction has been reversed. The heating tubes are horizontal. The evaporation takes place from the outside of the tubes, causing any deposit to be easily detached. The heating tubes are inserted in such a manner that they can easily be removed, all joints being made by a very effective packing device. The heating surface can also be readily cleaned on both sides, and thus the deposit caused by lubricating oils from exhaust steam can be removed, which otherwise soon reduces the efficiency of an apparatus.

Another very important feature in the construction is that the apparatus can at any time be enlarged to any desired capacity by simply adding more shelves, and no change in foundations or in the original plan is necessary.

In evaporating appliances that produce the evaporation by causing the liquid to be evaporated to pass through a series of tubes in the form of spray, or run down the inside or outside of the tubes in a thin film, great loss is occasioned by the small particles of juice becoming entangled with the escaping vapour and loss of sugar and discolouration takes place owing to parts of the heating surface being at times bare. The heating tubes we are describing being entirely submerged in the apparatus, these troubles are entirely overcome, while the great efficiency due to shallow evaporation is retained. The liquid to be evaporated is only four inches deep, and this depth of liquor is regulated automatically so as to be just deep enough to cover the tubes.

The heating surface is also divided into compartments, which gives the apparatus a large evaporating surface. This, together with the shallow evaporation, gives a very high efficiency, and the amount of water evaporated per square foot of heating surface is nearly four times greater than the best results obtained with the vertical tube apparatus. The flow of liquor through the apparatus is continuous, so that liquor flows in at one end of the apparatus and out at the other at any desired density.

The evaporating apparatus is made to work from a single to a quadruple effect, and has been adapted to the evaporation of all classes of liquors, more especially for the evaporation of cane or beet juice, glucose liquors, packing house tankage, and tan bark extracts. The firm has also modified the apparatus so as to make it perfectly adapted to the manufacture of salt, and the fuel necessary to produce the salt has been reduced 75 per cent. as compared with what is used by the open pan process, and the manual labour has been reduced almost in the same proportion.

The salt produced is also of very superior quality. Instead of flat scale-like crystals produced in the open pans, the crystals are regular and uniform, and of much greater

purity, and the most superior table and dairy salt is produced without any grinding, screening, or other subsequent operation.

The great merit of this evaporating apparatus is shown by the fact that three-fourths of all the contracts made in Louisiana this year for multiple effects have been awarded to the Fort Scott Foundry and Machine Works Company.

One triple effect which the company is now building for the Caffrey Central Sugar Refinery at Franklin, La., will be the largest evaporating apparatus in the United States. It will evaporate one million pounds of water per day, and is guaranteed to evaporate three pounds of water for every pound of steam delivered from the steam boilers into the apparatus.

By the use of an apparatus consisting of six effects, it is claimed to be possible to evaporate six pounds of water for each pound of steam introduced into the apparatus, this being rendered feasible by the very shallow evaporation and large evaporating surface, and it is said that this claim is fully sustained by the performance of their evaporators erected on several plantations last year.

## PATENTS.

*A Process or Method of Extracting Sugar from Sugar Cane or other Sugar-Yielding Substances, and Apparatus for the Purpose.* A. Chapman, Liverpool. From C. A. Mattley, St. Lucia, West Indies. Eng. Pat. 12,021, July 29, 1889. 8d.

THE sugar cane or other sugar-yielding substance is first thoroughly disintegrated by a crushing, shredding, grinding, or other mechanical process, and the saccharine juice is afterwards removed therefrom by displacement by means of water or other liquid. The latter operation is carried on preferably in a tall cylindrical vessel provided with a false bottom and also with a true bottom which opens with a hinge for facilitating the removal of the exhausted pulp. The pulp is filled into this vessel on to the false bottom and water is added at the top through a perforated plate until all the liquor is driven out through a cock at the bottom of the vessel.—A. J. K.

*Apparatus for Estimating the Quantity of Glucose or Grape Sugar in Fluids.* A. W. Gerrard, London. Eng. Pat. 18,856, November 25, 1889. 4d.

See under XVIII., page 774.

*Improvements in the Manufacture or Production of Raw or Refined Sugar, and Apparatus to be used therein.* A. Fairgrieve, Edinburgh. Eng. Pat. 1247, January 23, 1890. 8d.

In the production of dry sugar from the purified liquor of the refinery or manufactory the following process is adopted. Concentration is effected in a closed pan under vacuum as speedily and at as low a temperature as possible. Having reached the "proof" point without, however, allowing grain to form, and the pan being full up to the practical working limit, feeding is discontinued and the vacuum gradually lowered to zero in such a manner as will maintain the contents of the pan at "proof" point. The increasing temperature due to the falling vacuum should never be allowed so to thin the "liquor" as to cause it to froth. Neither should the liquor be allowed to become so thick as to force grain to separate out. The massecuite is now ready for the drying apparatus, which must be thoroughly heated before use. The massecuite is run into the pan, which is jacketed with steam at 15 lb. pressure, to the depth of about 6 in., and well stirred with arms which revolve at about 40 or 50 revolutions per minute. By this means the massecuite is rapidly and continuously turned over and over and exposed to the action of the heat

and air. It quickly changes from the viscous state in which it enters the pan into that of a dry free sugar in a very fine state of division, as fine as flour, and well suited to its economical use in consumption. Lumps formed in the drying are broken up by rollers attached to each alternate arm. Twenty or thirty minutes suffices for the mixing and drying operations. The sugar is now discharged into a set of sieves set vertically one above another and below the drying apparatus. Besides the usual back and forward action the sieves are so arranged as to slightly oscillate and "dump" after the manner of the self-acting machine for arranging wooden match sticks in the sets for heading. Advantages claimed for this process over the one usually adopted are extreme simplicity and economy, saving in cost of plant, space, fuel and hand labour. The resulting product is in the form of "flour," instead of being grainy as usual. The apparatus required is described by aid of drawings.—A. J. K.

*Improved Process for the Manufacture of Sugar.* C. Steffen, Vienna, Austria. Eng. Pat. 3589, March 6, 1890. 8d.

Is the evaporation of sugar in a vacuum pan liquor of good quality is first run in, which produces a crop of crystals and a syrup less pure than the original liquor. A liquor is now run in of the same purity-coefficient as the syrup in the pan, with the result that the crystals are enlarged and a still more impure syrup is formed as a mother-liquor. This is again fed by a liquor of same purity-coefficient as the mother-liquor, into which it is run, and the process is carried on until finally a very impure liquor is fed in. It is claimed that better results are obtained by this process than by feeding continuously with liquor of a uniform purity, and the process is applied both in the factory and in the refinery.—A. J. K.

*Improvements in the Method of and Apparatus for the Manufacture of Starch and the Treatment of the Residues obtained therein for Various Purposes.* S. Spitzer, New York, U.S.A. Eng. Pat. 4181, March 17, 1890. 8d.

The process consists of a method of preparation of starch by means of water only, without the use of any acids or other chemicals. The grain is first softened by steeping in water, and is then conveyed to a pair of crushing rollers geared so as to produce friction by their surfaces travelling at different velocities. From the crushing rollers the grain passes to a separating apparatus which consists of a vessel with a false lining, through the perforations of which the starch is forced by revolving beaters into an outer vessel. From the outer vessel the starch milk is conveyed into a series of sieves arranged vertically so that the starch flows through the coarser sieve into a finer one and so on, thereby removing any remaining husk, bran or other foreign matter. From the bottom sieve the starch liquor flows away into a settling tank, where the pure white starch separates out. It is afterwards dried and is ready for the market. The bran and husk being quite free from injurious chemicals, can be worked up into a good food for cattle, and the gummy matters separated and made into glue or paste. Drawings of the plant used are given.—A. J. K.

#### ERRATUM.

This Journal, page 527, 2nd col., delete line 46—"so as to obtain *L*-mannonic acid."

## XVII.—BREWING, WINES, SPIRITS, Etc.

*Saké-Brewing in Japan.* M. Ikuta. Chem. Zeit. 14, 439.

SAKÉ is a very popular drink in Japan, it is prepared from rice by fermentation, the manufacture being carried on from September to February only, as in the summer the fermentation cannot be properly controlled. It is a pleasant light yellow liquid, smelling of alcohol, its specific gravity is about 0.99 and contains on an average 15 per cent. of alcohol.

The manufacture takes place in three stages.

1. *Preparation of Koji.*—The finest rice is boiled in an iron kettle with sufficient water to soften it; after cooling it is brought into the koji chamber, the outside walls of which are covered with earth and the interior with straw, by which means the temperature of the interior is kept up.

The rice is here mixed with a little moyaschi (a yeast obtained from a previous fermentation) and remains in the chamber for two days, by which time most of the starch of the rice is converted into dextrose and it is then called koji.

2. *Preparation of Motoh.*—Koji, fresh boiled rice, and water are mixed in a wooden vat in the proportion to form a thin paste. Wooden casks containing warm water are placed in this liquid every day; after one or two days a frothy fermentation takes place, which is allowed to continue 30—40 days, during which time the temperature rises to 37° C.; at the end of the period the liquid contains 10 per cent. of alcohol and is called motoh.

3. *The Third Stage.*—Freshly boiled rice, koji, and motoh are mixed with a little water. The mixture ferments a little, but without appreciable rise in temperature. As soon as the froth ceases to form on the surface, i.e., in 8—10 days, the process is finished.

The fermented fluid is filtered through cotton bags and put into casks and allowed to stand till clear. It is then heated in an iron vessel to 44° C. and is then run into casks.

The residue remaining in the cotton sacks has been used from time immemorial in Japan for the manufacture of vinegar.—A. L. S.

*Communication from Guayaquil. The Quality of the Spirit obtained from the Sugar Cane.* C. Monheim. Chem. Zeit. 14, 439.

By the use of modern plant it is quite possible to obtain a spirit of fair quality. In the interior of Ecuador the most primitive form of still is in use and the product is very impure. It is used in the manufacture of liqueur, cognac, &c. The liquors prepared from this impure alcohol have more terrible intoxicating effects than any other that the author is acquainted with, being much worse than the crude spirit from potatoes, grain, &c. This alcohol retains its noxious impurities with more tenacity than any other crude spirit and there seems to be no satisfactory way of purifying it.—A. L. S.

*Contributions to the Knowledge of the Composition of Wort and Beer.* H. Elien. Zeits. f. angew. Chem. 1890, 291—298, and 321—326.

1. *The Determination of the Solid Extract.*—A weighed quantity of the beer or wort of known specific gravity is introduced into a small stoppered glass weighing tube 40 mm. diameter and 35 mm. high. This is placed in an oven, the temperature of which is kept at about 97° C. by a steam jacket; the oven is hermetically closed and is connected with a water injector pump. By admitting a proper quantity of air through a second tube the pressure in the oven is maintained at about 250 mm. of mercury. The water quickly evaporates from the wort, and when most of it has been driven off the supply of air is

further diminished until the pressure within falls to 35—45 mm.; the small quantity of air which is still admitted is first dried by sulphuric acid. The residue in the tube froths up and soon dries. The tube is weighed at intervals until it ceases to lose weight after 4—5 hours further heating. This usually takes 10—15 hours. The author has satisfied himself, by making cupric reduction determination before and after drying, that the wort suffers practically no decomposition by this treatment.

The results obtained are considerably lower than those obtained by previous observers (Schulze and Ostermann, *Zeits. f. das ges. Brauw.* 1878, 248; 1883, 10; and Balling, *Die Bierbrauerei*, 3 Aufl. 1865, 128). This is due to the fact that these observers dried their extracts imperfectly, Schulze working with a stream of air at 70°—75°, at which temperature maltose does not lose its water of crystallisation. The author has made determinations with hopped and unhopped wort and beer residues, and finds that the same table will apply to all. The table given below is abbreviated from the one accompanying the paper:—

Extract in 100 cc. Wort at 15° C.	Factor.	Sp. Gr. 15° 15°
Grms. 25	397.0	1.00025
20	398.0	1.07960
15	399.0	1.05985
10	400.0	1.04000
5	401.0	1.02005
1	401.8	1.001018

Sp. Gr. 15° 15°	Extract in 100 cc. Wort.	Extract in 100 Grms. Wort.	Sp. Gr. 15° 15°	Extract in 100 cc. Wort.	Extract in 100 Grms. Wort.
1.0090	24.94	22.60	1.0760	19.09	17.74
1.0080	24.68	22.48	1.0750	18.83	17.52
1.0070	24.43	22.27	1.0740	18.58	17.30
1.0060	24.17	22.05	1.0730	18.33	17.08
1.0050	23.92	21.84	1.0720	18.07	16.86
1.0040	23.66	21.63	1.0710	17.82	16.64
1.0030	23.41	21.42	1.0700	17.57	16.42
1.0020	23.15	21.20	1.0690	17.31	16.20
1.0010	22.90	20.99	1.0680	17.06	15.97
1.0000	22.64	20.77	1.0670	16.81	15.75
1.0090	22.39	20.56	1.0660	16.55	15.53
1.0080	22.13	20.34	1.0650	16.30	15.31
1.0070	21.88	20.13	1.0640	16.05	15.08
1.0060	21.63	19.91	1.0630	15.80	14.86
1.0050	21.37	19.70	1.0620	15.54	14.64
1.0040	21.12	19.48	1.0610	15.29	14.41
1.0030	20.86	19.26	1.0600	15.04	14.19
1.0020	20.61	19.05	1.0590	14.79	13.96
1.0010	20.36	18.83	1.0580	14.53	13.74
1.0000	20.10	18.61	1.0570	14.28	13.51
1.0090	19.85	18.39	1.0560	14.03	13.28
1.0080	19.59	18.18	1.0550	13.78	13.06
1.0070	19.34	17.96	1.0540	13.52	12.83

Sp. Gr. 15° 15°	Extract in 100 cc. Wort.	Extract in 100 Grms. Wort.	Sp. Gr. 15° 15°	Extract in 100 cc. Wort.	Extract in 100 Grms. Wort.
1.0530	13.27	12.60	1.0260	6.49	6.32
1.0520	13.02	12.38	1.0250	6.24	6.09
1.0510	12.77	12.15	1.0240	5.99	5.85
1.0500	12.52	11.92	1.0230	5.74	5.61
1.0490	12.26	11.69	1.0220	5.49	5.37
1.0480	12.01	11.46	1.0210	5.24	5.13
1.0470	11.76	11.23	1.0200	4.99	4.89
1.0460	11.51	11.00	1.0190	4.74	4.65
1.0450	11.26	10.77	1.0180	4.49	4.41
1.0440	11.01	10.54	1.0170	4.24	4.17
1.0430	10.75	10.31	1.0160	3.99	3.93
1.0420	10.50	10.08	1.0150	3.74	3.68
1.0410	10.25	9.85	1.0140	3.49	3.44
1.0400	10.00	9.62	1.0130	3.24	3.20
1.0390	9.75	9.38	1.0120	2.99	2.95
1.0380	9.50	9.15	1.0110	2.74	2.71
1.0370	9.25	8.92	1.0100	2.49	2.47
1.0360	9.00	8.68	1.0090	2.24	2.22
1.0350	8.74	8.45	1.0080	1.99	1.98
1.0340	8.49	8.21	1.0070	1.74	1.73
1.0330	8.24	7.98	1.0060	1.49	1.48
1.0320	7.99	7.74	1.0050	1.24	1.24
1.0310	7.74	7.51	1.0040	1.00	0.99
1.0300	7.49	7.27	1.0030	0.75	0.74
1.0290	7.24	7.04	1.0020	0.50	0.50
1.0280	6.99	6.80	1.0010	0.25	0.25
1.0270	6.74	6.56			

II. (A.) *The Determination of Maltose in Wort and Beer.*—The usual method for determining maltose in wort and beer is to determine the cupric-reducing power and to calculate this as all due to maltose. The author considers that other reducing bodies are present in these liquids and that consequently the method gives erroneous results. If some wort be sterilised and then fermented at 25° C. with a pure cultivation of *saccharomyces cerevisie* until the fermentation is completed, it is found that the solution still possesses a reducing power; the author has set himself to determine, (1) if the sugar which has fermented be all maltose and, (2) if the reducing power remaining after fermentation be due wholly or in part to maltose. A definite quantity of wort is fermented with a pure cultivation of *saccharomyces cerevisie* and the following factors are determined both before and after fermentation:—*Specific gravity*, which gives by the tables above, the total solids; *the reducing power*, determined gravimetrically, and *the same* after boiling with dilute hydrochloric acid which converts all maltose and dextrin into dextrose. Several experiments of this kind were made, of which the following may be considered a fair specimen.

Before Fermentation.	17 Days after.	Difference.
Sp. gr. .... 1.05630	1.02144	..
Extract ..... 13.46	5.21	8.25
Reduction .... 9.433	1.208	8.23
Dextrose ..... 13.098	4.514	8.58 = 8.15 maltose



All the experiments indicated that the sugar which had fermented was all maltose. Of these three processes for determining maltose, the author considers the fermentation one the best, the difference between the amount of extract before and after fermentation will always be greater than the amount of maltose fermented, as part of the maltose and other matter is used up in the growth of the yeast, which is filtered out. A correction therefore should be applied for this, which the author is engaged in determining.

The second method by determining the decrease in reducing power the author does not consider sharp enough.

The third method by estimating the amount of dextrose produced by boiling with hydrochloric acid both before and after fermentation is open to the same objection as the second, and also owing to the fact that other bodies besides maltose produce dextrose under these conditions, and the amount of dextrose produced from these bodies appears to increase after fermentation.

It is evident that in order that the first method may be adopted it must be shown that all the maltose is destroyed when the fermentation has stopped, *i.e.*, that the reducing power which the beer still possesses after complete fermentation is not due to maltose. This the author has done as follows: two portions of the same wort were fermented completely by a pure yeast; when the fermentation had stopped one of the liquids was analysed, but to the other a small quantity of pure maltose was added, when a brisk fermentation took place. After this second fermentation was finished the liquid was analysed and found to have the same composition as the first, thus showing that whatever remained in it was not free maltose.

In converting dextrin and maltose into dextrose, the author has found that the best proportions are 1.5 per cent. extract solution and one-twentieth volume of hydrochloric acid sp. gr. 1.19, heated in boiling water for three hours.

(n.) *The Value of the Maltose Determination in Practical Brewing.*—It may generally be considered that the amount of maltose in beer should not exceed certain limits, and so a knowledge of the amount of maltose remaining after fermentation is very useful, as if too large a quantity be present the beer cannot have been properly fermented, and therefore will not be stable.—A. L. S.

*On Cider and Perry.* P. Kulisch. Landw. Jahrb. 1890, 83.

CIDER and perry have many points of resemblance in their chemical composition. The common idea that the latter contains more sugar than the former is erroneous, the larger proportion of acid in cider masking the taste of the sugar. The total soluble matter is higher in perry than in cider, as a rule, and it also contains more tannin. The author discusses the question of distinguishing chemically between true wine made from grapes and that derived from other fruit. The characteristic difference appears to be the absence of tartaric acid free and combined in cider and perry; other differences exist and are useful in aiding judgment, but are not separately conclusive. For example, the percentage of alcohol in a fruit wine is lower than is met with in a genuine grape wine except in very poor years; further, the proportion of acid is not correspondingly high, while the rest of the soluble matter as well as the percentage of ash is higher than in a grape wine, and the ash itself is poorer in phosphoric acid. At the same time the total nitrogen in the fruit wine is lower than in the other. No safe deductions can be made in the case of doctored or fortified fruit wine, as the introduction of a little genuine wine renders useless the one certain test, *viz.*, the absence of tartaric acid.—B. B.

*Some Chemical Properties of an Alcoholic Extract of Brewers' Yeast.* J. de Rey-Pailhade. Bull. Soc. Chim. 1890, 3, 171—174.

AN alcoholic extract of brewers' yeast is prepared by mixing washed and pressed yeast with an equal volume of alcohol of 86 per cent.; the mixture is filtered, a clear yellow liquid, containing about 46 per cent. of alcohol,

being obtained. When 5 cc. of this extract are placed in a test tube along with 1 grm. of washed sulphur or 1 cc. of a saturated ethereal solution of sulphur, hydrogen sulphide is evolved, as is shown by the blackening of a strip of paper moistened with lead acetate. The name of *philothion* is given to the substance in the extract which possesses this curious property of causing the formation of hydrogen sulphide. One litre of the freshly prepared extract produces 10 mgrms. of hydrogen sulphide. After exposure to the air for two or three days the extract loses its property of generating hydrogen sulphide from sulphur, or, in other words, the philothion is destroyed. The addition of small quantities of hydrochloric or sulphuric acid renders philothion inactive; on neutralising the acid, the activity of the philothion is restored. When the extract is decolourised by animal charcoal the philothion is removed. Alkalis hasten the destructive action of oxygen (air) on philothion. Philothion is found in most animal tissues and in the young and active parts of plants; it probably plays an important physiological part in the living cell.—E. B.

*Wine from Dried Grapes and its Richness in Total Nitrogen.* P. Cazeneuve and L. Ducher. Bull. Soc. Chim. 1890, 3, 514—518.

THE problem of identifying wine made from dried grapes and distinguishing it from natural wine has never been solved. So recently as July 30th, 1889, the International Congress at Paris declared that there was no process for detecting with certainty the presence of wines from dried grapes, in natural wine. Various unsuccessful methods have been proposed, such as determinations of the levo-rotary or dextro-rotary power, the percentage of gum, or the variety of bacilli present.

The authors failed to confirm Giraud's supposition that a levo-rotary unfermentable principle was present in the wine of dried grapes; but, considering that as fruit matures its albuminoid matters increase, they believed that dried grapes would be found to be richer in nitrogen, which quality might also be persistent in the wine.

To determine if this were so, 5 cc. of wine were dried on a water-bath with plaster; 5 grms. of plaster sufficed for dry wines, but 10 grms. were required for sweet wines. The dried residue was ignited with copper oxide according to the method of Dumas.

The results were as follow:—

	Nitrogen in Grms. per Litre.
Dry white wines—	
Natural.....	0.275
From dried grapes (average of three samples) ...	0.248
Sweet white wines—	
Natural—1st sample.....	1.106
2nd sample.....	1.085
From dried grapes—1st sample.....	1.510
2nd sample.....	1.424
3rd sample.....	1.523
Red wines—	
Unclearified.....	0.340
Clarified.....	0.276

Thus, in the above samples of sweet wines, there is a marked increase of nitrogen in wine from dried grapes. The close agreement between the different samples is noteworthy. The authors account for the absence of difference between the dry wines by the explanation that the albuminous matters would be diminished by the longer fermentation and by clarification and filtration. The effect of clarification is shown in the above results with red wines.

—D. A. S.

*The Vacuum Method of Drying Barley and Malt, with a Direct Determination of the Extract in Beer and Worts.* C. N. Rieber. Zeits. f. d. ges. Brauw. 13, 97.

THE author makes use of a drying apparatus with paraffin bath. By means of a water-jet pump the pressure is kept down to 20 mm. of mercury. The substance to be dried is contained in a small thin glass tube, which during cooling

and weighing is closed by a well-fitting glass cap. If it be required to dry beer or worts in this manner, about 2 grms. of filter-paper, cut into strips, are introduced into the tube and about 5 cc. of the liquid dropped on top.

Barley at 110° C., under a pressure of 40 mm. of mercury, continues to give off water for about 30 hours; furthermore it seems that the barley after two hours' drying loses still 0.87 per cent. of its original weight, independent of the original moisture. For purposes of technical analysis, therefore, it is only necessary to heat for two hours under the above-mentioned conditions and to then add 0.87 per cent. to the result obtained.

Malt dried under the same conditions is slightly decomposed. The differences between two and 28½ hours' drying varied from 1.02 to 1.03 per cent. The author proposes to dry as above for two hours and take the result obtained, without correction, as this is identical with the result obtained by drying in an air-bath for 24 hours at 105°—110° C.

In determining the extract of worts and beers at 20 mm. pressure and a temperature of 70° C. constancy was attained after 66 hours' drying. The following results were obtained with beers and worts; they are here compared with the results given by Schultze's tables at 17½° C. and by Balling's tables at 15° C. :—

Schultze.	Balling.	Rieber.
Per Cent. 14° 10.	Per Cent. 13° 48.	Per Cent. 13° 38.
7.89	7.64	7.45
5.65	5.45	5.27

—T. L. B.

#### PATENTS.

*Improvements in the Treatment or Preparation of Rice and other Grain or Cereals for Brewing and other purposes, and in Means or Apparatus employed therein.* A. Perry, London. Eng. Pat. 7909, May 11, 1889. 11d.

THE rice or grain to be treated is ground by any suitable means and washed in a chamber which is continually supplied with cold water. A screw conveyor at the bottom of this chamber transfers the grain as it subsides from the water to one end of the apparatus into a kind of well. From thence the now washed grain is elevated by another screw conveyor, and dropped into the hopper of the cooking apparatus. From this hopper the grain passes into a long tapering shaft, which is surrounded by a jacket into which high-pressure steam can be admitted. The grain is forced towards the narrower end of the shaft by means of a screw conveyor passing along its centre. The feeding end of the apparatus is provided with taps which convey steam and hot water to the material under treatment in order to soften and scald it. As the grain approaches the narrow end of the tube it is compressed, and is in consequence more readily gelatinised by the action of the heat. The cooked material issuing from the end of the apparatus is preferably converted into shreds or ribbons. This is best done by forcing it by means of pressing blades or rollers through a die or grating fixed at the bottom of a circular chamber. The shredded grain is dried by any suitable means. If desired, vegetable or fruit juices or colouring matters may be mixed with the grain in the cooking or shredding apparatus. Starch may also be added in the shredding apparatus. For designs and full description of the various apparatus above referred to, the original specification must be consulted.—H. T. P.

*Improved Apparatus for Cooling or Icing Beer or other Drinkables, also applicable for Warming or Mulling Liquids.* M. T. Neale, London. Eng. Pat. 10,506, June 28, 1889. 6d.

"The object of this invention is a cheap, simple, and expeditious means or apparatus for icing or cooling beer

or other drinkables" chiefly for use in public-houses or hotel bars. The liquid to be cooled flows through a tube or coil contained in a vessel filled with a freezing mixture, and the vessel is coated with felt on the outside. By replacing the freezing mixture by hot water liquids may be warmed or "mulled" in the same apparatus.—A. J. K.

*An Improved Method of Facilitating the Drying of Distillers' Spent Wash and similar Matter.* E. Larsen, London. Eng. Pat. 10,740, July 3, 1889. 4d.

THE invention consists in the addition of some light bulky substance, such as husks from oats, rice cones, malt combs, or brewers' grains, to the "wash," which is previously dried as much as possible by settling and pressing. The mixture thus formed is easily completely dried by means of any of the ordinary machines commonly used for drying grains, &c. The grains or cones added are then removed by sifting, and may be used again for a fresh portion of "spent wash."—H. T. P.

*Improvement in Apparatus for the Treatment of Yeast.* T. Vogel, Saalfeld, Germany. Eng. Pat. 10,764, July 3, 1889. 8d.

THE object of this apparatus is the aeration of yeast by forcing through it air in a finely-divided state. The special advantage claimed for the machine is that it can be easily cleaned as the separate parts can be readily dismounted for cleaning purposes. Full description is given with reference to drawings.—A. J. K.

*A Process for Purifying Alcohol, Wine, and other Alcoholic Liquids and Beverages.* P. C. Rousseau, M. J. de Chanterae, and M. J. D. A. de la Baume, Paris, France. Eng. Pat. 12,186, July 31, 1889. 4d.

ACCORDING to this invention alcoholic liquids are freed from impurities by treatment with a basic tartrate followed by a hyposulphite (thiosulphate). Tartrate of potash or soda, or preferably the double salt known as "Sel de Seignette" may be used. Among the hyposulphites those of barium and sodium have given excellent results. The alcoholic liquid contained in a suitable closed vessel is mixed with small quantities of the tartrate, being well agitated after each addition, until it reacts neutral or faintly alkaline to test paper. The impurities are now allowed to settle, which requires from a few hours to several days according to circumstances; but in any case the operation is considerably hastened by the application of heat, or by the action of light. Hyposulphite is now added in quantity equal to about one-third of the tartrate used. The liquid is well stirred and allowed to settle, when the clear portion may be drawn off and rectified in the usual way. This process is applicable to all kinds of alcoholic liquids, notably wines and rum, the flavour of which is greatly improved.

—H. T. P.

*Improvements in the Method of Producing Ferments.* L. Meyer, Stuttgart, Germany. Eng. Pat. 13,054, August 19, 1889. 6d.

THIS invention practically consists in impregnating some suitable material or "ferment-carrier" with a growth of the ferment it is desired to employ. Various animal and vegetable fibres and other substances may be used for this purpose; but, weight for weight, raw sheep's wool, freed from grease, is the most efficient. In carrying out the process any desired quantity of cleansed sheep's wool is dipped into a well-fermenting liquid, still containing about two-thirds of the sugar present at the commencement of the fermentation, fermenting wort, grape juice, or other liquid being taken according to the nature of the ferment required. After soaking, the wool is placed in a vessel which can be tightly closed, and kept at a temperature of 25—30° C. for a period of three days. The fermenting material is now

finished, and is employed by bringing it into contact with the liquid in which it is desired to produce fermentation. The improved fermenting material is very durable, and may be used over and over again. The process is applicable for all alcoholic fermentations. It may also be used for the manufacture of yeast, and for fermenting champagne made by the French method, a piece of the material being attached to the cork.—H. T. P.

*Improvements in Treating and Rectifying or Ageing Alcohol or Alcoholic Liquors or the like by Electricity.* A. de Méritens, Paris, France. Eng. Pat. 14,162, September 7, 1889. 4d.

This patent has reference to a method of increasing the electrical conductivity of alcoholic liquors. This is effected by dissolving in them sodium bisulphite. This salt has the advantage of liberating an equivalent of sulphurous acid, which, during the passage of the current, reduces the aldehydes and allied bodies present in the alcohol, so that by this process, in conjunction with treatment in the apparatus previously patented (this Journal, 1888, 860), alcohol free from aldehyde may be obtained. Further, this method when applied to worts, washes, &c., stops or retards secondary fermentation.—H. T. P.

*Improvement in the Process of Treating Cereals.* F. Dorr, Baltimore, U.S.A. Eng. Pat. 2450, February 14, 1890. 4d.

CEREALS, such as barley, wheat, rye, &c., in their fully matured condition, are first cleaned from the bran or husk in the usual manner, and are then subjected to the action of steam of from 150° to 250° F. When thoroughly cooked the cereals are partially dried and then passed through fast revolving burr-mills, dressed and ground, in contradistinction to being passed through rolls. The mills are heated by friction of the two faces of the stones, until the heat obtained is at least 200° F. before the grain is passed through. The product obtained is designated "Barlyne," and will keep in any climate.—A. J. K.

*Improvements relating to the Production of Malt.* C. Hof, London. From L. Röhmsam, Bamberg, Bavaria. Eng. Pat. 3579, March 6, 1890. 4d.

This invention deals with the manufacture of colour malt. The chief novelty consists in the conversion of the starch of the malt into sugar, previous to caramelisation. Charring of the grain is altogether avoided, and as a consequence beer brewed with this malt is altogether free from any burnt flavour. In practice "green malt" is placed in a revolving cylinder or "toaster," and moistened with 9—10 pints of warm water per cwt. of malt taken. The toaster is kept in constant rotation, the temperature inside it not being allowed to exceed 100° C. In about two hours the starch of the malt is converted into sugar. Thereupon the temperature is somewhat raised, and the rotation continued for about five hours more. By this means the sugar is more or less completely caramelised, although the husk of the malt hardly darkens at all. Finally, the malt is thrown out of the toaster, and heaped on a floor, preferably of stone, to cool. After removing the "comes" and cleansing, it is ready for use.—H. T. P.

## XVIII.—CHEMISTRY OF FOODS, SANITARY CHEMISTRY, AND DISINFECTANTS.

### (A).—CHEMISTRY OF FOOD.

#### PATENT.

*Improvements in Apparatus used in the Purification and Collection of Carbonic Acid Gas prior to its being used for Aerating (Soda) Water or other Liquors.* J. Childs, London. Eng. Pat. 11,565, July 19, 1889. 8d.

THE carbon dioxide gas, instead of being first washed and then collected in an ordinary gasholder, is passed through a chamber provided with shelves, upon which dry crystalline soda is placed. It is found that this removes the impurities in the gas, and any liquid which may be formed during their removal is run away by a pipe at the bottom of the purifier. The object of thus purifying the gas is to enable it to be collected and preserved in pliable bags (made up of two layers of canvas and three of rubber) instead of the ordinary holder. These would otherwise be rapidly destroyed by the action of the impure gas upon them. The specification is illustrated by drawings.—S. G. R.

### (C).—DISINFECTANTS.

#### PATENT.

*Manufacture of Bleaching and Disinfecting Liquor.* E. Hermite, E. J. Paterson, and C. F. Cooper, London. Eng. Pat. 8177, May 16, 1889. 4d.

See under VI., page 732.

## XIX.—PAPER, PASTEBOARD, Etc.

*Andanson Paper.* W. Herzberg. Mitt. k. Techn. Vers. Anstalt, Berlin, 1890, 82—89.

THE author has investigated sample lots of paper prepared from this well-known bast. They were of a pale yellow colour, but "dirty," i.e., flecked with spots, varying in colour from yellow to brown. These spots consisted of cellular residues, which, notwithstanding the thorough washing to which the fibre was subjected, it was found impossible to remove.

Subjoined are the results of the mechanical tests, (a) being a thin unsized paper, (b) a thicker paper, sized:—

	Tearing Length.		Mean.	Elongation at Fracture.		Mean.
	Along the Web.	Across the Web.		Along the Web.	Across the Web.	
(a)	Km. 6'49	Km. 3'69	4'79	Per Cent. 2'5	Per Cent. 4'1	3'3
(b)	6'31	3'72	5'02	1'0	6'2	5'1

The fibre possesses extraordinary fineness and capacity for forming a strong interlaced texture, which would render it capable of yielding paper as strong as and similar to the well-known Japanese paper.

A noteworthy characteristic of the fibre is its high percentage of mineral constituents. Subjoined are the results of complete analyses of the ash both from the original bast and the paper:—

#### ASH CONSTITUENTS OF ANDANSONIA DIGITATA.

	Raw Bast.	Paper.
	Aggregate Percentage 17.47, with 6.94 per Cent. CO <sub>2</sub>	Aggregate Percentage 5.35, with 1.72 per Cent. CO <sub>2</sub>
Lime .....	49.59	52.89
Magnesia .....	3.44	3.90
Alumina .....	0.11	0.18
Iron oxide .....	0.32	0.24
Potash .....	5.35	1.14
Soda .....	0.32	0.08
Silica .....	0.22	4.70
Phosphoric acid .....	0.54	2.97
Chlorine .....	1.03	1.23
Sulphuric acid .....	0.56	0.15
Carbonic acid .....	39.79	32.18
	99.61	9.86

It is interesting to compare with the above the subjoined results of analyses of cigarette papers prepared from hemp and flax fibre, A giving an aggregate of 0.35 per cent. and B, 0.44 per cent. of ash constituents.

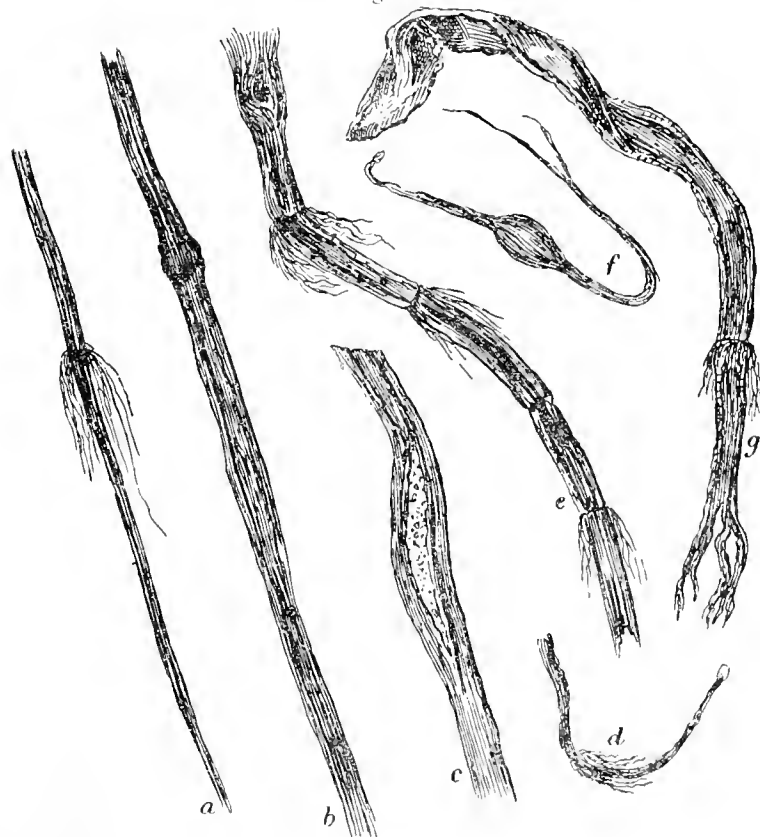
	Raw Bast.	Paper.
	Aggregate Percentage 17.47, with 6.94 per Cent. CO <sub>2</sub>	Aggregate Percentage 5.35, with 1.72 per Cent. CO <sub>2</sub>
Lime .....	10.45	21.35
Magnesia .....	5.53	13.51
Alumina .....	5.05	9.66
Iron oxide .....	1.82	3.91
Potash .....	2.83	2.13
Soda .....	0.85	0.11
Silica .....	17.73	37.76
Phosphoric acid .....	10.85	5.61
Chlorine .....	1.31	0.79
Sulphuric acid .....	0.91	0.94
Carbonic acid .....	12.59	2.84
	99.95	98.91

The characteristic features of the Andansonia ash are the preponderance of lime and the low percentages of silica and phosphoric acid.

*Structural and Microscopic Characteristics.*—Fig. 1 shows the general disposition of the fibres in the bast

Fig. 2.

Fig. 1.



ANDANSONIA PAPER.

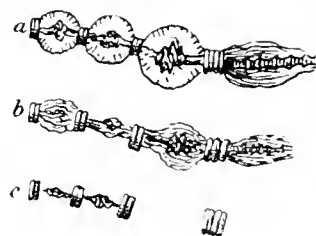
Fig. 3.



Fig. 4.



Fig. 5.



## ANDANSONIA PAPER.

complex. In Fig. 2—*a, b, c, d, e, f, g*—are reproduced the more important variations of the bast fibres, in point of microscopic structure. The cell walls and lumen show unusual irregularity; the ends of the entire fibres are mostly rounded, when fractured or drawn in the heating process there appears to be little tendency to resolve the cell wall into fibrillae. The length of the cells varies from 2.8 to 5.0 mm.; the average may be taken to be 4.2 mm. The brownish flecks in the paper before mentioned, when examined under the microscope, are seen to consist of groups of cells (Fig. 3), similar to the sclerenchymatous cells of straw. Occasionally also residues of vessels (Fig. 4) are to be seen. The structural changes observed during the progress of the solvent action of the ammonia cupric reagent are very characteristic. The successive stages are shown in Fig. 5, *a* to *c*. The result is obviously occasioned by the greater resistance of the outer portions of the cell wall to the action of the reagent. The particulars of the chemical treatment to which the fibre was subjected are not given.—C. F. C.

*Mineral Constituents (aggregate) of Paper-making Materials.* W. Herzberg. Mitt. k. Techn. Vers. Anstalt, Berlin, 1890, 89—92.

THIRTY-ONE samples of rags were investigated and found to contain on the average 3.06 per cent. of ash constituents. Twenty-two specimens of half-stuff, bleached and unbleached, prepared from rags, were found to contain on the average 0.74 per cent.

In unmanufactured fibrous raw materials the average lies between 1 and 2 per cent.; quantities in excess of 2 per cent., which may be taken as a maximum number, indicate the presence of adventitious mineral matters; in manufactured goods (rags, bagging, &c.) these may have been added in the form of mordants (dyed goods) or filling materials. It will be seen that they are for the most part removed in the process of boiling and washing.

Subjoined are the results of the author's determinations of "total ash" in commercial pulps:—

	Per Cent.
Sulphite pulp (1) .....	0.48
Sulphite pulp (2) .....	0.51
Sulphite pulp, bleached .....	0.42
Soda wood pulp, bleached .....	1.40
Soda wood pulp .....	1.34
Straw pulp .....	2.30
Straw pulp, bleached .....	1.22
Mechanical wood pulp (pine) .....	0.43
Mechanical wood pulp (fir) .....	0.70
Mechanical wood pulp (aspen) .....	0.44
Mechanical wood pulp (lime) .....	0.40

(Compare A. Frank on "Ash Constituents of Sulphite and other Pulps." Papier Zeitung, 1888, 893.)—C. F. C.

*Strength of Paper in relation to Thickness.* W. Herzberg. Mitt. k. Techn. Vers. Anstalt, Berlin, 1890, 92—94.

PAPERS were made containing 75 per cent. of linen, 10 per cent. of cotton, and 15 per cent. of wood cellulose, rosin sized; the weight of one (*a*) was 12 kilos. per 1,000 sheets,

of the other (*b*) 16 kilos. The mechanical tests gave the following results:—

—	Weight.	Tearing Length.		Elongation at Fracture.		Temperature and Moisture.
		Along Web.	Across Web.	Along Web.	Across Web.	
(a)	Grm. Sq. M. 87	Km. 4.85	Km. 3.48	Per Cent. 2.0	Per Cent. 3.4	15.8° C., 71 per cent.
(b)	117	4.58	3.23	2.2	4.4	17.5° C., 73 per cent.

Similar results were obtained with papers prepared from sulphite cellulose alone. Subjoined are the mean of the determinations: temperatures, 18.6°—18.8°; hygrometric state, 63—64 per cent.

—	Weight.	Tearing Length.	Elongation at Fracture.
(a)	Grm. Sq. M. 55	Km. 5.68	Per Cent. 2.1
(b)	90	5.40	3.0
(c)	170	5.17	3.7

With increase of thickness, therefore, there is a diminution of tenacity and an increase in elasticity.—C. F. C.

*Preparation of the New Russian Paper Money.* O. Hallsleben. Dingl. Polyt. J. 276, 181—183.

THE special feature which characterises the paper used for the preparation of the new Russian bank notes is that it contains an impression of a silk web, the meshes of which are of a definite form. This web is prepared upon a loom worked by a machine of special design, and is not removed from the rollers, but taken to the paper mills and worked into the paper during its manufacture, rollers of special construction being used, so that the original form of the silk meshes is retained.—D. B.

*Treatment of Ramie (Urtica Nivea).* J. Armandon. Monit. Scient. 1890, 401—402.

THE author has successfully cultivated the *U. Nivea* in his experimental garden at Saint-Maurice Canavere. The bast strips were treated with a solution of ordinary rosin in soda, containing 3—4 per cent. of the former. After boiling under slight pressure for some hours, the fibre was removed, boiled with dilute alkaline solution (2 per cent. Na<sub>2</sub>O), well washed with water, and bleached with hypochlorite. Thus prepared the fibre was brilliant white. The yield was 21 per 1,000 of the green stems. The fibre thus prepared appears to be easily dyed.—C. F. C.

## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

*Eucalyptus Oil.* Schimmel and Co. Zeits. Anal. Chem. **29**, 222—223.

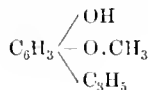
EUCALYPTUS oil, now in the market, contains 50 per cent. of eucalyptol, together with phellandrene. To recognise the latter constituent it is advised to mix 1 cc. of the oil with 2 cc. of glacial acetic acid, and to then add 1—2 cc. of a strong aqueous solution of sodium nitrite. On gently agitating, the separated oil almost immediately solidifies to a crystalline mass of phellandrene nitrite.

The highest boiling portions of eucalyptus oil (portions boiling from 220°—260° C.), contain euminal. To identify the pure compound, obtained from the sodium bisulphite compound, oxidation was performed by means of permanganate; the acid thus obtained possessed the melting point of euminal acid.

Good oil should contain from 50—70 per cent. of eucalyptol; the best kinds are so rich in eucalyptol that they may be semi-frozen by a freezing mixture.—T. L. B.

*The Phenol of Sassafras Oil.* C. Pomeranz. Monatsh. **11**, 101—103.

THE etheral oil of the American cinnamon tree *Laurus sassafras* has been shown by Grimaux and Ruott to contain 10 per cent. of a terpene,  $C_{10}H_{16}$ , 90 per cent. of saffrol,  $C_{10}H_{16}O_2$  (this Journal, 1890, 100), and a small quantity of a body possessing the characteristics of a phenol—probably eugenol. This last can be isolated by shaking the oil with dilute alkali, separating the alkaline liquid and acidifying with sulphuric acid, when a brown oil separates, the greater portion of which, when dried, distils over at 246°—247° C. The author obtained 7 grms. of the oil from 3 kilos. of the sassafras oil, and showed both by analysis and by the preparation of the characteristic benzoyl derivative that the phenol in question is undoubtedly eugenol—



—C. A. K.

*Lobeline.* H. Paschke and A. Smita. Monatsh. **11**, 131—132.

To obtain the alkaloid from *Lobelia inflata* the leaves are extracted with warm water acidified with acetic acid, the extract concentrated, made alkaline and the alkaline solution extracted with ether. The acid extract is then treated with water, rendered alkaline and again extracted with ether; after distilling off the latter the alkaloid remains as a viscous honey-coloured oil, with a smell akin to that of honey and tobacco. For further purification the oil is dissolved in ether, extracted with water acidified with hydrochloric acid, the acid extract then rendered alkaline and the resulting solution again extracted with ether. After distilling off the ether, the residue is dried over caustic alkali and distilled in a current of hydrogen. When lobeline or its sulphate is suspended in a 10 per cent. caustic potash solution and a 4 per cent. solution of potassium permanganate is gradually added, the whole being gently warmed on the water-bath, benzoic acid results, which can be extracted by means of ether after filtering off the manganic hydrate and acidifying the resulting solution. The identity of the acid formed with benzoic acid was proved by analysis and by its characteristic properties, whence it appears that lobeline contains an aromatic nucleus.

Further investigations on lobeline are in progress.

—C. A. K.

*Notes on some Pharmaceutical Preparations.* A. Weller. Chem. Ind. **13**, 162—165.

THE following notes are based on the report for the year 1889 of the "Vereinigte Fabriken chem.-pharm. Produkte Feuerbach," Stuttgart and Frankfurt a. M., Zimmer and Co.

*Arecoline.*—Jahns (this Journal, 1889, 210) and Marmé (Pharm. Zeit. **34**, 12) have studied the liquid alkaloid contained in areca-nut and assign to it the formula  $C_8H_{13}NO_2$ . It has an action on the heart similar to muscarine, slows the respiratory action, and strongly affects the peristaltic contraction of the bowels. Applied in small amount to the eye, it powerfully contracts the pupil.

The other alkaloid, arecaine, contained in areca-nut, is physiologically inactive.

*Atropine.*—Keen commercial competition has greatly lowered the prices of atropine salts. Ladenburg and Hundt (Ber. **22**, 2590) isolated from quinine tropate by fractional crystallisation dextro- and levo-rotatory quinine tropates and from these salts have further separated a dextro- and a levo-rotatory base, of which the latter resembles hyoscyamine, but is by no means identical with it.

*Caffeine.*—English competition in this article has reduced its price to such a degree that its production in Germany is out of the question. Amongst newer salts of this alkaloid is the phenate, which is also administered by injection.

*Quinine.*—The exports of cinchona bark from Ceylon and British India during the last eight years have been as follow:—

Year.	From Ceylon.	From British East India.
1881—82	Lb. 3,669,895	Lb. 428,497
1882—83	6,925,598	611,618
1883—84	11,492,917	396,019
1884—85	11,678,360	715,730
1885—86	15,364,412	857,040
1886—87	11,438,260	1,286,900
1887—88	11,704,332	1,449,315
1888—89	10,798,663	3,071,098

The proposed formation of a syndicate of quinine planters is rendered impossible by the present system of working, under which most of the planters are supported by loans of money from England, which are repaid in produce so that the planters have not a free hand in the disposal of the latter. The Dutch Government is largely encouraging the planting of cinchonas in Java. The exports from this source have risen from 1,104,534 Dutch lb. in 1883—84 to 4,415,631 lb. in 1888—89.

The price of quinine fell in June 1889 to 11d. per oz., the lowest price it has yet reached. It rose again, however, towards the close of the year, being quoted in London at 1s. 3d. per oz. The sales of this alkaloid have been very large, considerable quantities being required during the epidemics of *dengue* fever and influenza. As a prophylactic against malarial infections, quinine is still unequalled. The quinine-testing question is decided in favour of Kerner-Weller's ammonia test (Archiv. Pharm. (25), **3**, 16—17). Skrap and his pupils have shown (Monatsh. **10**, 39, 51, 65, 220; this Journal, 1889, 474, 815, 960) that the cinchona alkaloids are chemically closely related.

Of new salts of quinine, the bromide is of value in the treatment of neuralgia; the glycyrrhizinate very effectually masks the bitter taste of the alkaloid; quinine-hydroquinone hydrochloride and sulphate are types of a new class of double compounds of quinine and phenols, which are more rapid febrifuges than the corresponding simple quinine salts (Hesse, Pharm. Zeit. **34**, 24).

The consumption of cinchonidine, cinchonine, and quinoidine decreased; on the other hand quiniidine was in such demand that its price bordered on that of quinine.

Oudemans has worked on *cupreïne* (Rec. des trav. chim. des Pays-Bas, 8, 147).

The importation of coca leaves greatly diminished in consequence of the preparation of crude *cocaine* in South America. The price of this alkaloid has kept very steady.

Howard published (Pharm. Journ. and Trans. 969, 569) some interesting analytical results. He finds that species of erythroxylon, other than erythroxylon coca, contain only 0.02 to 0.03 per cent. of the alkaloid.

Numerous analytical tests for this alkaloid have been published during the past year. The chromate test of Metzger (Pharm. Zeit. 34, 92), despite the objections of Giesel (*ibid.*, 93), is very serviceable.

Liebermann, Hesse, Einhorn, and others, have pursued investigations in regard to the constitution of cocaine and its derivatives and associates.

*Cytisine*, according to Radziwillowicz (Arch. d. Pharm. Instituts zu Dorpat, 2), Prévost, and Binet, is intermediate in its physiological action between strychnine and curarine.

*Hydrastine* has been further investigated by Freund (this Journal, 1889, 412, 637, 1006).

*Hydrastinine*, a product of the oxidation of hydrastine, is a powerful base, whose salts, injected hypodermically, act as haemostats (Falk, Ther. Mon. 4, 19).

*Hysocine*.—The haloïd salts of this dye are largely used as soothing agents in cases of mental disease, for which purpose Kny (Münch. med. Woch. Nos. 45 and 46), Klinker, Dornblüth, and Rabow consider it unrivalled.

*Lobeline*.—The active constituent of *Lobelia inflata* gives excellent results in cases of dyspnea and spasmodic asthma, and is much to be preferred to the old Galenic preparation, as it does not induce vomiting or diarrhoea (Nunex, Brit. Med. J. 1889, 1051).

*Mandragorine*, prepared by Ahrens (Ber. 22, 2159), is possibly isomeric with the belladonna alkaloids. It is mydriatic. As yet it has not been employed.

*Opium alkaloids*.—The price of opium, and consequently of morphine, had a general upward tendency.

Flückiger (Archiv. Pharm. 27, 721, 769), Dieterich, Petersen, and Holdermann have described methods of estimating the alkaloids in opium.

Bardet and Adrian found morphine in *Escholtzia Californica*, previously recommended by Ter Zacarant (Bull. gén. de Thérap. 1889, 21) as a harmless hypnotic.

*Codeine* increases in importance. Dornblüth (Ther. Mon. 3, 363) recommends it as a hypnotic in cases of mental disease. For hypodermic administration codeine phosphate is preferred.

*Narcotine meconate* has recently appeared in commerce.

Roserehen continued his researches on *narcotine* (Annalen, 254, 334, 359).

*Pelletierine*.—Bérenger-Férard (Bull. gén. de Thérap. 1889, Feb.) recommends this alkaloid as being the most powerful and certain vermifuge known.

*Physostigmine* remained almost without change. The consignments being small in amount, the price of the raw material, Calabar beans, slightly advanced. This was also the case with Jaborandi leaves, the material from which *pilocarpine* is prepared.

*Quebrachine* hydrochloride is recommended as a specific for asthma and dyspnea.

Pawinski (Gaz. Hebdom. de méd. de Varsovie, 1889, June) finds the administration of *Sparteïne sulphate* of service in many cases of functional cardiac or valvular derangement.

*Strychnine*.—Jaroschewsky states that this alkaloid neutralises the toxic and narcotic effects of alcohol, and is, therefore, to be regarded as a powerful antidote to alcoholism.

*Theobromine* and, more especially, the easily soluble theobromine sodium salicylate, also known as *diuretine*, act on the kidneys (von Schröder, Archiv. f. exper. Path. u. Pharm. 24, 85), without exerting any toxic action like the closely-related caffeine does. Gram reports favourably on the diuretic action of this alkaloid (Ther. Mon. 4, 10).

—E. B.

## PATENTS.

*A Process for obtaining Carbon Acids of Meta-amidophenol, and of its Alkyl Derivatives.* C. Kolbe, Dresden, Germany. Eng. Pat. 12,191, August 1, 1889. 6d.

META-AMIDO-PHENOL is heated under pressure in a closed vessel to about 110° C. with about four times its weight of ammonium carbonate or other alkaline carbonate and five times its weight of water for 12 hours. The use of the carbonates of the alkaline earths is also claimed. The resulting solution is concentrated, the impurities separated by the addition of a little hydrochloric acid, and then the hydrochloride of the carbon acid obtained by the addition of an excess of hydrochloric acid. This is purified by dissolving it in soda and reprecipitating with acid. (The sulphate is less soluble than the hydrochloride.) To separate the free acid, the purified hydrochloride is treated with caustic soda or with sodium acetate and the solution then extracted with ether. The free acid melts with evolution of carbon dioxide at 148°, it dissolves readily in water, alcohol, or ether, but with difficulty in benzene, toluene, or chloroform. The alkaline salts are readily soluble in water, the hydrochloride and sulphate difficultly soluble.

Dimethyl- and diethyl-meta-amido-phenol are first converted into alkaline phenates, and the dried phenate treated under pressure with dry carbon dioxide at 120°–140° C. The free acids are separated from the sodium salts formed by addition of acetic acid, and can be purified by recrystallisation from hot toluene.—C. A. K.

*Improvements in Refining Apparatus or Furnaces for Camphor or similar Substances.* H. H. Lake, London. From W. V. McKenzie, New Jersey, and H. Braker, New York, U.S.A. Eng. Pat. 5367, April 8, 1890. 6d.

The apparatus consists of a furnace, refining pan and cover. The furnace is rectangular and is made of two casings of metal, between which asbestos is packed; in the bed of the furnace suitable openings for heating by means of gas are provided. The refining pan, made of suitable metal, extends nearly to the bottom of the furnace; the side walls of the pan converge downwards so as to allow the heat from the gas burners to be distributed all round the pan. The pan cover, also made of metal, rests upon suitable flanges of the refining pan, and is held firm by screws and luting; in the centre of the cover is an opening, which can be closed by a sliding cover or valve.

In refining camphor, &c., the crude material is placed in the pan, the cover fixed in position, and the heating started; at the end of the operation the opening in the cover is loosened to allow any evolved gases to escape, the cover removed and the camphor scraped off.—C. A. K.

## XXII.—EXPLOSIVES, MATCHES, Etc.

*Action of Fatty Substances on Gunpowder.* S. Bein. Zeits. f. angew. Chem. 1890, 217–222.

THE points considered are the detonating point, the rate of combustion, and the explosive force. The powders examined were treated with blubber, coal-tar, wood-tar, sweet oil, olein, linseed oil, and petroleum.

Of coarse grained powders those containing 1 to 15 per cent. of fatty matter seemed to the eye to burn in the normal manner; those containing 16–22 per cent. of fat burnt by no means normally, and those containing 23–25 per cent. could scarcely be burnt at all. After the samples had remained for a fortnight in stoppered bottles, it was found that even those containing 5–7 per cent. of fatty matter were harder to burn than before, and burnt slower.

The detonating points were next determined (this Journal, 1890, 211). Coarse-grained powders containing 1–2 per



cent. of fat had a detonating point approximately the same as the detonating point before treatment with oil, but when the grease rose to 3 per cent. the detonating point rose  $25^{\circ}$ — $26^{\circ}$ , and when to 1 per cent. the detonating point was raised as much as  $38^{\circ}$ .

The way in which gunpowder burns depends not only on its composition, but also on the character of the grain, fine-grained powder burning quicker than the coarser varieties. Fine powder containing 1—2 per cent. of fatty matter burns normally, whilst, with 3 per cent. and over, ignition is hard

to effect in normal manner. Fine-grained powder containing 3 per cent. of fatty matter ignited at a temperature  $16^{\circ}$  higher than the same powder free from fatty substance. A sample containing 4 per cent. was kept for six weeks, and at the end of this time its ignition point was  $362^{\circ}$ , that of the same powder free from fatty matter being  $305^{\circ}$ . The following table shows the detonating points of a powder after 10 weeks' contact with various oils. The average detonating point of the powder itself was  $355.5^{\circ}$ . It is here to be noted that a maximum distinctly exists, as shown in the table.

Percentage of Fat.	1.	2.	3.	4.	5.	6.	7.	8.	9.	10.	11.	12.	13.	15.
Character of fatty matter:—														
1 part of petroleum . . . . .	363	370	377.5	385	392.5	400.5	405	411	416	422	..	432	..	440.9
2 parts of sweet-oil . . . . .														
Coal tar . . . . .	360.5	365.5	366	366.5	366	366	366	366	366.7	367	366.7	369	369.5	369.7
Linseed oil . . . . .	380	387	386	383.5	381	..	361	..	..	360	..	..	..	..
Olein . . . . .	359	366	375	380	387	..	381	376	..	368	..	..	..	..
Sweet oil . . . . .	357.5	362	370.5	..	376	..	370.5	..	..	367	..	365	..	..
Wood tar . . . . . $\left\{ \begin{array}{l} 0.5\% \\ 347 \end{array} \right\}$	352.5	357	362	368	371	..	370	..	..	361.5	..	360	..	..
Blubber . . . . .	357	368	370	376	383.5	388	389	..	385	..	..	382	..	378.7

The rate of burning forms likewise an important feature in the character of a gunpowder. The interstices in the case of fine powder are very small, and hence the passage of the flame is considerably retarded. To investigate the rate of burning of greased samples, long cylindrical metal tubes, open at both ends, were used. These were filled with the powder and the time of burning was noted in seconds. It was shown that samples containing 2 per cent. of fatty matter required a longer time for burning than did the pure sample; with 4—5 per cent. of fatty matter double the time was required, and some samples containing still more fatty matter refused to burn completely. Similar results were obtained after allowing the samples to stand for three weeks. This may be explained by considering that the oil during combustion uses up a certain quantity of heat, which is abstracted from the heat of combustion of the powder. The powder is thus able to burn gradually only. Such action of fatty substances would, of course, enable the use of lighter gun-barrels on account of decreased expansive force of the gases, and would bring about the use of lighter weapons, and a consequent quicker movement of troops. It might be considered that a decrease in the expansive force of the gases in the barrel would bring about a decrease in the velocity of the shot. Against this the author advances the following:—At the moment of firing the velocity of the shot is 0; immediately after, a gas expansion takes place in the direction of the bore of the barrel, and the shot is driven towards the muzzle. If no new forces come into play the shot has now received the whole of its velocity imparted. If greased powder be used, however, this fires gradually, and fresh impulsive forces come into play. If  $p$  represent the acceleration of the shot, corresponding to the gas expansion at a particular point in the barrel,  $v$  the initial velocity, and  $V$  and  $V_1$  the velocity of the shot at two different points in the barrel, then—

$$p = \sqrt{\frac{n}{g}} [(V - V_1)^2 - (V - v)^2] \dots \dots (1),$$

when  $\frac{n}{g}$  is a constant determined for each weapon and shot.  $n$  is the sum of a coefficient depending on the density, elasticity, and other properties of the shot (determinable by experiment), and on the bore of the weapon. At first, when  $v = 0$ , we have:—

$$p_0 = \sqrt{\frac{n}{g}} (V_1^2 - 2 V V_1) \dots \dots \dots (2)$$

The maximum acceleration ( $p_m$ ) at a point  $m$  of the barrel is:—

$$p_m = \sqrt{\frac{n}{g}} (1 - V_1)^2 \dots \dots \dots (3)$$

$v$  being put =  $V$  in equation (1).

The difference of the squares of the greatest and smallest accelerations (equation 3)<sup>2</sup> - (equation 2)<sup>2</sup> is thus:—

$$p_m^2 - p_0^2 = \frac{n}{g} V^2 \dots \dots \dots (4)$$

If  $v = 0$  the velocity, at a point in the barrel, is from (2)

$$V = \frac{V_1^2 - \frac{n}{g} \cdot p_0^2}{2 V_1} \dots \dots \dots (5)$$

Then substituting in (4) we get:—

$$p_m^2 - p_0^2 = \frac{n}{g} \cdot \frac{(V_1^2 - \frac{n}{g} \cdot p_0^2)^2}{4 V_1^2}$$

and

$$V_1 = \sqrt{\frac{n}{g}} \cdot (p_m + \sqrt{p_m^2 - p_0^2})$$

$V_1$  is a maximum when  $p = 0$ , that is, the maximum velocity of a shot is attained when the gas evolution is gradual, and consequently the greasing of gunpowder has a very decided advantage and plays a very important part.

—T. L. B.

## PATENTS.

*Improvements in or relating to the Manufacture or Treatment of Explosives.* H. M. Chapman, Waltham Cross. Eng. Pat. 1115, January 21, 1889. (Second Edition.) 6d.

THIS is an invention for converting well-known explosives such as gun-cotton and other nitro-explosives into a gelatinised or horny mass, by treatment with suitable solvents and subsequent manipulation.

The patentee describes at great length, and illustrates by drawings, the special apparatus he uses for effecting the gelatinisation of the explosive, for removing the solvent, and, in certain cases, volatilising the agent which has been used for gelatinising the gun-cotton. He particularises the following four methods of gelatinising or agglomerating the explosive:—

“First; that in which the agglomerating agent, whether solid such as camphor, or liquid such as aldehyde or formic

ether, is evaporated or distilled or allowed to evaporate off completely. Second; that in which the agglomerating agents with boiling points too high to be safely volatilised off are left in the explosive, and partly or wholly fulfil the function of a 'moderator' or substance that reduces the rapidity of the explosion, such as acetate of amyl, the mono-di- and tri-acetins, oxalic ether, and the high-boiling point ketones and acetates. Third; that in which mixtures of the first and second, such, for example, as a solid, say camphor, and a liquid, say amyl acetate, are used together. Fourth; the employment of hydraulic or other pressure with or without the simultaneous application of heat in combination with any of the before-mentioned three methods, whereby the explosive under treatment can be gelatinised and rendered homogeneous with very much less of the agglomerating or gelatinising agent than would otherwise be required."

Nitro-starch can also be gelatinised by common ether or by any of the methods specified above. The use of "moderators" in nitro-explosives of the nitro-benzene and nitro-naphthalenes is one of the claims, and it is pointed out that although camphor has been used for hardening explosives, it has not been employed for gelatinising gun-cotton as described.—W. M.

*Manufacture of Explosives.* F. A. Abel, London, and J. Dewar, Cambridge. Eng. Pat. 8718, May 27, 1889. 4d.

THIS invention consists in the addition of about 10 per cent. of tannin to gun-cotton alone or mixed with nitroglycerin. Either the tannin is dissolved in such a solvent as acetone, and this solution is used for dissolving the gun-cotton, or the tannin is added to the solution of gun-cotton in acetone, and the solvent is eliminated in known ways. When tannin is added to gun-cotton alone the product is hard and can be granulated; when nitroglycerin is also used the product is gelatinous. The addition of tannin moderates the force of explosives such as gun-cotton and nitroglycerin, giving them a propulsive instead of a disruptive character.—W. M.

*Improvements in the Preparation of Explosive Compounds.* A. V. Newton, London. From A. Nobel, Paris, France. Eng. Pat. 9361, June 5, 1889. 6d.

THIS invention relates to the preparation of a similar explosive to that described in Eng. Pat. 1471, 1888 (this Journal, 1889, 214), without the addition of camphor.

The nitrocellulose is mixed with an equal weight of nitroglycerin until the latter is completely absorbed. This operation is conducted at the lowest convenient temperature (say 5° to 10° C.). It is advantageous to employ the nitrocellulose in the wet and pulped state, as the presence of water retards the dissolving action of the nitroglycerin and otherwise facilitates the intimate mixture of nitroglycerin and nitrocellulose. The mixing is continued until only water escapes on squeezing the material. The mass is next immersed in hot water of 80° C., and heated at that temperature for about one hour, until it has become gelatinised, so that nitroglycerin does not exude on pressure. The material is then rolled into plates or sheets as described in the specification referred to above.

The moist or dry nitrocellulose may also be allowed to absorb as much nitroglycerin as it can at, say, a temperature between 5° and 10° C., and the excess of nitroglycerin is removed by pressure. The mass is then gelatinised as before. Should nitrocellulose be used which is not quite insoluble in nitroglycerin under the above conditions, then the addition of about 20 per cent. of benzene to the nitroglycerin will prevent it from dissolving the nitrocellulose at this temperature. Another method consists in mixing nitrocellulose with two or three times its weight of nitroglycerin and gelatinising the mass as before and rolling it into sheets. The excess of nitroglycerin is removed by steeping the sheets in a solution of methyl alcohol to which 25 per cent. of water has been added, until sufficient nitroglycerin has been dissolved out. The material is then again rolled into sheets, &c. The chief advantage of this process is that the gelatinisation takes

place more quickly and at a lower temperature (50°—60° C.). The inventor, however, generally prefers the first described method of working.

The nitroglycerin can be easily recovered from the methylic alcohol solution, since, when the temperature of the solution is lowered to 5° C. the nitroglycerin separates out.—W. M.

*Improvements in Explosive Compounds.* W. B. McGavin, Woodford Green. From E. W. McGavin, Sydney, N.S.W. Eng. Pat. 9433, June 7, 1889. 6d.

ACCORDING to this invention an improved explosive compound is prepared by saturating sawdust with a solution of potassium picrate, drying the impregnated sawdust, and then mixing it with potassium and sodium nitrates and sulphur.—W. M.

*Improvements in the Manufacture of Dynamite.* C. O. Lundholm, Ardeer. Eng. Pat. 10,312, June 25, 1889. 4d.

THE inventor renders so-called black dynamite (this Journal, 1889, 818) flameless by kneading it with water, when a large proportion of water can be absorbed without causing exudation of the nitroglycerin, or nitrate of ammonia in the form of dry powder or strong solution is mixed with the dynamite.—W. M.

*Improvements in the Manufacture of Explosives.* C. O. Lundholm and J. Sayers, Stevenston. Eng. Pat. 10,376, June 26, 1889. 4d.

THE principal object of this invention is a means of combining large proportions of cellulose nitro-derivatives with nitroglycerin.

The inventors avail themselves of the fact that the cellulose nitro-derivatives have the property of taking up or absorbing the nitroglycerin when suspended in water.

According to one method of working the cellulose nitro-derivative in a fine state of division is made into a pulp with water, the nitroglycerin is made into an emulsion with water and this is poured into the pulp and the whole is vigorously agitated. All the nitroglycerin is uniformly absorbed by the cellulose nitro-derivative; the water can then be separated by straining and pressure, and the compound afterwards malaxated between heated rollers and made into suitable forms.—W. M.

*An Improved Machine for Compressing Gunpowder for Use in Cartridges.* J. Robb, Loanhead. Eng. Pat. 10,917, July 6, 1889. 8d.

"THE object of this invention is, by lateral and vertical pressure, to make the gunpowder of sufficient density and cohesion to be suitable for use in cartridges for small-bore or other arms and in accordance with the Government requirements." Drawings accompany the specification, which must be consulted for a description of the machinery employed by the patentee.—W. M.

*Improvements in Matches or Lighters of certain Form for Cigars, Pipes, and the like.* A. F. Hawksley, Altrincham. Eng. Pat. 11,320, July 15, 1889. 6d.

THIS invention relates to improvements on the previous patent No. 11,135, 1886, and consists essentially "in forming the match or lighter with a washer or filling of non-combustible material, such as asbestos, pipeclay, or the like between each mass of combustible composition" so as to prevent more than one mass of the composition becoming ignited at a time. The match thus consists of a series of alternating portions of combustible and non-combustible compositions formed into a rod which is kept in a suitable tube-like case.—W. M.

*Improvements in the Manufacture of Explosives.* F. A. Abel, London, and J. Dewar, Cambridge. Eng. Pat. 11,664, July 22, 1889. 4d.

THIS invention consists in making a gelatinous explosive by admixture of nitroglycerin with the highest nitrated cellulose or gun-cotton, instead of the soluble varieties hitherto used, effected with the aid of a solvent such as acetone.

Various substances may be added with the object of moderating the force of explosion.

It is stated that owing to the uniformity of the composition of the gun-cotton used, the manufactured product is also constant in composition and uniformly reliable in its action.

—W. M.

*Improvements in or relating to Explosive Cartridges.* J. C. Butterfield and T. C. Batchelor, London. Eng. Pat. 12,249, August 1, 1889. 6d.

THE object of this invention is to prevent explosive compounds containing a hygroscopic salt like ammonium nitrate from deteriorating by absorption of moisture. For this purpose the inventors, after carefully preparing the explosive so as to ensure its perfect dryness, fill it into a metallic case consisting of a hollow cylinder made of thin sheet metal, such as lead or other metal or alloys having the requisite ductility and toughness, the cylinder is closed at one end and has at the other a screw thread run upon the metal. A small metal cap, which has been dipped into some quick-drying waterproof paint, is slipped on to the screwed end, the paint inserts itself in the screw threads, and the excess exudes and forms a waterproof ring round the juncture of the cap and the cylinder.

When it is desired to fire the material, a hole can easily be cut in the end of the case and a detonator inserted. Two or more cartridges can be fired by one detonator by simply removing the caps and placing the cartridges together end to end.—W. M.

*An Improved Gunpowder Mixing Machine.* F. W. Barker, London. From P. A. Oliver, Pennsylvania, U.S.A. Eng. Pat. 18,916, November 25, 1889. 8d.

THIS invention consists principally in "the combination with a rapidly revolving tub balanced upon a central pivot of two agitators revolving rapidly in a contrary direction to the tub, and arranged upon opposite sides of its central axis." The tub is lined with wood, and a fine spray of water from a suitable pipe keeps the materials to be mixed moist, and thus ignition by friction is prevented.

The tub is driven at from 300—400 revolutions, and the agitators at 1,000 revolutions per minute. The object of the high speed is to throw the materials against each other with such violence that they are reduced to an impalpable powder and also thoroughly mixed. By opening an outlet door while the machine is still in motion the contents are discharged by centrifugal force.

Drawings accompany the specification.—W. M.

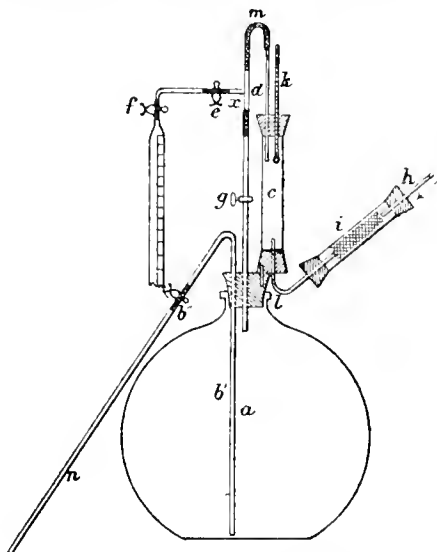
*Manufacture of Grained Smokeless Explosive.* W. Schückher, Vienna, Austria. Eng. Pat. 4243, March 18, 1890. 4d.

THIS explosive consists of a mixture of from 90 to 95 per cent. of explosive starch (xyloidine), and 10 to 5 per cent. of nitrobenzene. The ordinary machinery used in making gunpowder is employed. After long continued grinding in a ball-grinding mill, the mixture is in the form of an almost dry powder, it is then pressed into cakes, which are afterwards broken into grains, and the grains are coated with plumbago in the usual way.—W. M.

## XXIII.—ANALYTICAL CHEMISTRY.

*Sampling of Chimney Gases.* O. Schmidt. Zeits. Anal. Chem. 29, 136—138.

THE accompanying illustration shows an apparatus for taking samples of chimney gases and the like:—

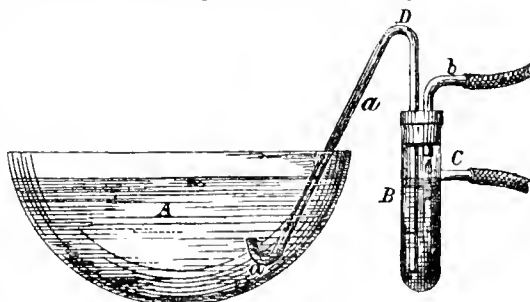


APPARATUS FOR SAMPLING CHIMNEY GASES.

*a* is an acid carboy, filled with water, and fitted with a doubly-bored rubber stopper, *b'* is a syphon, which may be closed by a pinch-cock at *b''*. *c* is a glass cylinder fitted with a rubber stopper at each end; one end is connected, through the cylinder *i* containing glass-wool, with the chimney. The T-tube *d* is connected with *c* by means of rubber tubing *m*, and on the other side with a tube which passes to the carboy and which is provided with a tap *g*. In using the apparatus, the pinch-cock *b''* is released, *g* is opened and *e* closed; suction at *n* sets the syphon working. The gases enter at *h*, are deprived of solid matter by the glass-wool in *i*, and pass through *l* and *c* into the carboy. When it is considered that all air has been displaced from *i*, *c*, and *d*, *g* is closed and *e* is opened. To *d* is connected, by means of a capillary tube, a Hempel's burette filled with water. The levelling portion of the Hempel is raised until the water reaches *x*, after which the gases to be examined may be drawn directly into the burette by lowering the levelling tube.—T. L. B.

*An Apparatus for retaining at a Constant Strength Salt Solutions, &c.* O. Foerster. Chem. Zeit. 14, 607—608.

THE vessel *A* in which the solution is heated, is connected by the tube *a* with the apparatus *B*. Through the tube *b* the water falls in drops into *B*, and the superfluous water



RETAINING SALT SOLUTIONS OF CONSTANT STRENGTH.

flows off at C. The lower end of *a* is bent upwards to prevent the liquid in A, when boiling, from rising in the tube *a*. In the same proportion as the liquid evaporates from A, the water is supplied from B through *a*.—H. S.

*An Impurity in Barium Chloride.* L. Blum. Zeits. Anal. Chem. 29, 139–140.

A sample of barium chloride was bought as “chemically pure.” Permanganate solution was, however, reduced by it. The author considers that the salt was probably obtained as by-product in the manufacture of hydrogen peroxide, and would therefore be liable to contain small quantities of enclosed hydrogen peroxide.—T. L. B.

*Detection of Nitrous Acid.* Curtmann. Pharm. Centralhalle, 29, 600.

THE green colouration obtained with antipyrine in presence of free nitrous acid is recommended.—T. L. B.

*Characteristics of the Alkaline Earths and of Zinc Oxide.* G. Brügelmann. Zeits. Anal. Chem. 29, 126–129.

WHEN these oxides are prepared from the nitrates by simple ignition, the following results are obtained. Calcium oxide, strontium oxide, and barium oxide are got in regular cubes, zinc oxide in hexagonal pyramids, often symmetric twin crystals, and magnesium oxide crystalline, not amorphous as hitherto considered. The specific gravities compared with water = 1 at 15° C., are CaO 3·25, SrO 4·75, BaO 5·72, ZnO 5·78, MgO 3·38.

Igniting the carbonates in a covered platinum crucible yielded:—CaO (3·26), MgO (3·48), ZnO (5·42), all amorphous; SrO 4·45, not amorphous as hitherto considered, but crystalline.

By heating the hydrates in a covered platinum crucible were obtained:—CaO (3·25), MgO (3·41), and ZnO (5·52) all amorphous, and here again, contrary to what is usually stated, SrO (4·57) crystalline. Barium oxide appears to form in graphite or earthenware vessels hexagonal needles (5·32), in platinum crucible it is regular (5·74). Barium oxide is thus dimorphous. Furthermore the strontium oxide obtained from both carbonate and hydrate was perfectly white; the barium oxide obtained from barium hydrate (the carbonate is too difficult to decompose by heating), was also perfectly white, hence it may be assumed that these oxides when pure are not grey but perfectly white as calcium oxide is. It was also shown that anhydrous barium oxide does not take up carbon dioxide within the limits of decomposition of the hydrate and carbonate. The specific gravity of strontium oxide stands almost midway between the specific gravities of the oxides of calcium and barium.

It is to be remarked that the crystalline oxides here obtained crystallised directly from the fused mass, and were not first obtained in the amorphous condition and subsequently transformed into the crystalline by continued heating.—T. L. B.

*Occurrence of Arsenic in Zinc Oxide.* W. Stromeyer. Archiv. d. Pharmacie, 27, 549.

A SAMPLE of zinc oxide bought as pure was shown to contain arsenic by dissolving in hydrochloric acid and adding a sulphuretted hydrogen solution. The percentage proved to be 0·088.—T. L. B.

*The Molecular Refractive Powers of Salts in Solution.* E. Doumer. Bull. Soc. Chim. 1890, 3, 200–206.

THE author has determined the refractive powers of 90 salts by means of Trannin's “identimètre” or differential refractometer. The results obtained with 78 salts are tabulated, and serve to furnish generalisations. Seven salts gave anomalous results, which may be due to the possible

presence of impurities in them, most of them being difficult to purify. From the results the following conclusions are drawn:—

1. All salts which contain the same acid have the same molecular refractive power, provided they be constituted on the same chemical type. Thus the molecular refractive power,  $\mu P_m$  ( $\mu$  = refractive power of the dissolved salt compared with water,  $P_m$  = molecular weight of the salt) of chlorides of the type  $M'Cl$  is approximately 21·5; that of chlorides of the type  $M'Cl_2$  is 42·8, and that of sulphates  $M'_2SO_4$  is 42·5. It may be stated, as a corollary to this law, that the various basicities of a polybasic acid may be satisfied with different metals without effecting the molecular refractive power, thus the salts  $K_2CO_3$  and  $KNaCO_3$  have the same molecular refractive power.

2. The molecular refractive powers of salts of different chemical types are approximately multiples of one number. Thus for the following salts,  $KCl$ ,  $K_2SO_4$ ,  $Na_3PO_4$ ,  $PtCl_4$ ,  $Al_2(SO_4)_3$ ,  $Fe_2K_2(C_2O_4)_4$ , the values of  $\mu P_m$  are respectively 20·7, 43·1, 64·3, 89·8, 130·5, and 170·7.

3. The molecular refractive powers of salts are functions of the valencies of the metals in combination.

In the last example the valencies of the metals are respectively 1, 2, 3, 4, 6, and 8, and the refractive powers are approximately  $1 \times 21·5$ ,  $2 \times 21·5$ , &c.

4. The molecular refractive power of a double salt is equal to the sum of the molecular refractive powers of the individual salts of which it is composed.

The molecular refractive power of the double salt,  $AuCl_3 \cdot NaCl$ , is 80, which is very nearly the sum of the respective molecular refractive powers of  $AuCl_3 = 57·6$  and  $NaCl = 23$ .—E. B.

*Analysis of Sodium Aluminate.* G. Lunge. Zeits. f. angew. Chem. 1890, 227.

THE value of sodium aluminate used, e.g., in soap manufacture, is generally ascertained by determining the alumina as well as the sodium protoxide by gravimetric analysis, whereby the determination of the insoluble residue and of silica (silicate being present) is also necessary. If silica is, however, present in but small quantities the following method can be applied quickly and easily for technical purposes. Weigh a quantity of sodium aluminate, dissolve in water to a specified volume (the insoluble residue may be ascertained if required), withdraw with the pipette measured portions, add phenolphthalein, and afterwards standard hydrochloric acid solution. The reduction is complete when the colouration has disappeared. The amount of  $Na_2O$  is hereby determined. To the same liquid a drop of methyl-orange solution is added and the titration continued until the yellow colour of the liquid has changed to a permanent red. This change of colour takes place rather slowly; a preliminary test is therefore recommended whereby the acid is added a cubic centimetre at a time. Having thus approximately ascertained the quantity of acid required, in the actual test almost sufficient hydrochloric acid is added at once, and then the reaction finished drop by drop. When the red colouration remains constant for five minutes the reaction is complete. The amount of  $Al_2O_3$  is hereby ascertained, the indicator only assuming a permanent red colouration after all alumina has been converted into aluminium chloride, and a little acid in excess remaining. *Key* made a comparative test of both methods (gravimetric and volumetric analysis) with the following results:—

	$Na_2O$ .	$Al_2O_3$ .
	Per Cent.	Per Cent.
Gravimetric analysis.....	26·42	25·92
Volumetric analysis.....	26·42	26·03

This shows that the volumetric method is sufficiently exact; but it must not be forgotten that this is only the case in presence of small quantities of silica; where a larger amount of silica is present the gravimetric method must be applied.—H. S.

*On the Volumetric Determination of Alumina in Sodium Aluminate and other Similar Compounds.* G. Lange. *Zeits. f. angew. Chem.* 1890, 293—300.

K. J. BAYER (*Zeits. Anal. Chem.* 24, 512; this *Journal*, 1886, 11) described this process, according to which normal soda solution is added to the aluminium solution until the precipitate which is first formed is redissolved and two portions of this solution titrated with sulphuric acid, one with litmus as an indicator until a red colouration is produced, and the other with "Tropäolin" until the citron-yellow colour changes to orange-yellow. The difference between the amounts of sulphuric acid required to produce these changes is such that three molecules of  $\text{H}_2\text{SO}_4$  are equivalent to one molecule  $\text{Al}_2\text{O}_3$ . R. J. Thomson, using methyl-orange as indicator, obtained the same result. Cross and Bevan (this *Journal*, 1889, 233) on the contrary find the difference in the amount of sulphuric acid is such that 5  $\text{SO}_3$  are equivalent to 2  $\text{Al}_2\text{O}_3$ .

The author has made experiments using phenolphthaleïn as the first indicator and methyl-orange as the second, and comes to the conclusion that Cross and Bevan are wrong, and that three molecules of  $\text{H}_2\text{SO}_4$  are equivalent to one molecule of  $\text{Al}_2\text{O}_3$ . His determinations agree very well with results obtained by a gravimetric process.

He considers that Cross and Bevan, in titrating with the methyl-orange indicator, did not wait long enough to see if the colour was really permanent. He finds that working at ordinary temperatures it is necessary to wait at least 10—15 minutes, but by working with the solution at blood heat the indication is perfectly sharp.—A. L. S.

*Tannin Matters as Anti-incrustators for Steam Boilers.* L. Vignon. *Bull. Soc. Chim.* 1890, 3, 410—413.

THE author, having occasion to examine a number of mixtures sold as boiler disinfectants, remarked the presence in many of them of tannin matters, along with sodium carbonate, &c. One such mixture, in particular, was composed of 260 cc. of sumac extract at 30° B., and 106 grms. of anhydrous sodium carbonate, per litre. With a view to ascertain whether the tannin matter was of use or otherwise, a series of experiments was instituted. Five pieces of clean boiler plate of the same size were weighed, and, one being kept for comparison, the others were boiled for eight hours separately in the following solutions:—

No. 1.—Water of the river Rhône .....	1,500 cc.
No. 2.—Sumac extract of 30° B. ....	35 cc.
Water, to make .....	1,500 cc.
No. 3.—Sodium carbonate .....	15 grms.
Water, to make .....	1,500 cc.
No. 4.—Sumac extract .....	35 cc.
Sodium carbonate .....	15 grms.
Water, to make .....	1,500 cc.

At the end of the time mentioned the samples were removed, rinsed, brushed, cleaned with fine sand, dried, and weighed. The effects of the various solutions are shown in the following table:—

Solution.	Weight of Iron-plate before Experiment, in Grms.	Weight after Experiment, in Grms.	Loss, in Grms.
No. 1	100.420	100.370	0.050
No. 2	94.450	93.507	0.943
No. 3	98.250	98.245	0.005
No. 4	103.450	103.070	0.380
Sample kept	98.600	98.590	0.010

With the Rhône water the iron was slightly rusted; a deposit of calcium carbonate, mixed with some ferric oxide, was left in the bath. Sumac extract, used alone, caused the iron to become covered with a black coating of iron tannate; the bath was deeply coloured. No trace of rust appeared on the iron when sodium carbonate alone was

used; in the bath there was a slight deposit of calcium carbonate of a pure white colour. With the mixture of sumac extract and sodium carbonate the iron was covered with a black film and the solution was much discoloured. It appears, therefore, that (1), free tannin matters attack boiler-iron; (2), excess of sodium carbonate does not prevent this objectionable action; (3), sodium carbonate has no injurious action on boiler-plates.—E. B.

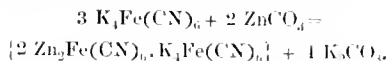
*Experiments on the Solubility of Minerals.* C. Dölter. *Monatsh.* 11, 119—150.

THE author has studied the action of distilled water on a number of minerals which are generally regarded as insoluble by heating them in sealed tubes to 80° C. for a number of weeks. Pyrites, Galena, zinc blende, stibnite, orpiment, bournonite, and copper pyrites, as well as rutile and cassiterite are more or less attacked, as much as 8 per cent. of the mineral being dissolved. Hematite and silicates such as heulandite, natrolite, anorthite and clabbsite are less readily acted on than the sulphides. Analysis shows that the minerals are dissolved as such, the dissolved portions having the composition of the original mineral. The quantity of water employed in the experiments is of necessity limited; the water was found to dissolve as much as 0.3 per cent. of its weight of substance. The addition of sodium fluoride to the water increases the solubility of rutile, cassiterite and hematite; sodium chloride has little effect. Carbon dioxide (not pumped in under pressure) increases the solubility of oxides and of silicates, but decomposition takes place during solution. Sodium sulphide or sulphuretted hydrogen considerably increases the solubility of sulphides, and stibnite is completely dissolved. Interesting cases of the reformation of crystals of the various minerals were observed in these experiments.

Gold heated to 200° C. in a closed tube with water containing 5 per cent. of sodium carbonate or silicate is dissolved in appreciable quantities to the extent of 1.5 per cent. of the weight of gold taken.—C. A. K.

*New Method of Estimating the Ferrocyanide in the Yellow Prussiate Melt.* R. Zaleski. *Zeits. f. angew. Chem.* 1890, 210—214.

TO the solution of the melt freshly precipitated zinc carbonate is added in excess; carbon dioxide is then passed through the liquid. After some time the clear solution is tested with potassium ferri-cyanide; the reaction is generally shown to be complete after about half an hour. The whole is then boiled for 10 minutes in order to decompose any dissolved zinc bicarbonate. After cooling and filtering an aliquot portion is titrated with sulphuric acid. The reaction taking place is represented by the equation:—



Sodium ferrocyanide is decomposed in an exactly analogous manner.—T. L. B.

*Determination of Ferrocyanides in the Residue from Gas Purifiers.* R. Zaleski. *Zeits. f. angew. Chem.* 1890, 301—302.

THE author has previously (*Zeit. f. angew. Chem.* 1890, 210, see preceding abstract) described a method for estimating ferrocyanides in crude potassium ferrocyanide, and he applies the same to the estimation of ferrocyanides in gas purifier residue.

Twenty grms. of the material are finely powdered, digested with 20 cc. of 10 per cent. potash solution, and the solution made up to 100 cc.; 45 cc. of the clear solution (containing half of the substance taken) is boiled in a 100 cc. flask until all ammonia is expelled, and then just neutralised with dilute acid. To this solution, which contains all the ferrocyanide as potassium ferrocyanide, 20 cc. of normal alkaline carbonate is added, and 5 grms. of moist zinc carbonate and a current of carbon dioxide passed through the hot solution for about half an hour. The solution is cooled, made up to

the 100 cc. mark, and 50 cc. of this titrated with decinormal acid, with methyl-orange as an indicator.

The 10 cc. of normal alkaline carbonate which has been added requires an equivalent quantity of acid, and if the acid be of such a strength that 1 cc. is equivalent to 0.001  $K_2CO_3$ , then the number of cc. of the excess of acid required multiplied by 0.23 and doubled gives the percentage of  $K_2FeC_4 \cdot 3H_2O$  in the material taken (this Journal, 1889, 732, and 1890, 111).—A. L. S.

*Quantitative Separation of Arsenic and Antimony.* O. Kochler. *Archiv. d. Pharmacie*, **227**, 106.

In his "Handbuch der Analytischen Chemie" (Sixth Edition, 2, 423), Rose mentions a method of separating arsenic and antimony by precipitating both with sulphuretted hydrogen and then gently warming with concentrated hydrochloric acid. The antimony trisulphide is alone dissolved. Some of the arsenic trisulphide is, however, apt to be dissolved as well. According to the present communication good quantitative results can be obtained by careful attention to concentration, and consequent prevention of the precipitation of the antimony. Small quantities of arsenic were taken in presence of large quantities of antimony. The antimony trichloride solution used had a specific gravity of 1.33, the concentrated hydrochloric acid was of sp. gr. 1.124 and the solution of arsenious acid contained 1 gm. of arsenious acid per litre.

It was found that to one part of the antimony solution at least two parts of the concentrated hydrochloric acid were necessary to prevent precipitation of the antimony by sulphuretted hydrogen from a hot solution.

For the separation 20 cc. of the antimony trichloride solution were mixed with 45 cc. of concentrated hydrochloric acid and 5 cc. of the arsenious acid solution; into the hot solution sulphuretted hydrogen was passed. The arsenic trisulphide was then filtered through a filter moistened with hydrochloric acid, and was subsequently washed with dilute hydrochloric acid (1:3). Finally, in order not to have to remove the hydrochloric acid with water, the precipitate was oxidised in a beaker with bromine water; the residue was filtered off and washed with ammonia, and to the filtrate ammonia was added. The arsenic was then precipitated with magnesia mixture and allowed to stand for some time (see Fresenius, Quant. Anal. Sixth Edition).—T. L. B.

*Volumetric Determination of Carbon in Iron.* J. Wiborgh. *Berg. u. Hüttenm. Zeit.* **46**, 223 and 233. (Compare this Journal, 1889, 726, and 1890, 16.)

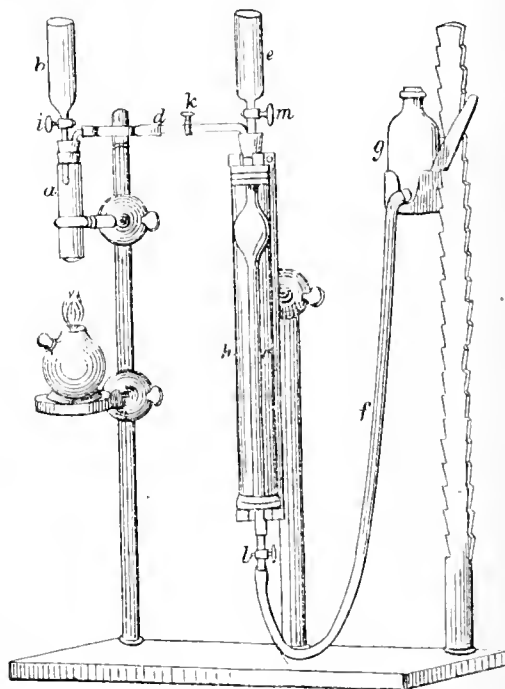
The iron is treated with copper sulphate, and the precipitated copper with the still undissolved iron is dissolved in a mixture of sulphuric and chromic acids. The carbon is there oxidised to carbon dioxide, the volume of which is measured in a burette. In order to obtain correct results it is imperative that the carbon be completely oxidised to carbon dioxide. Hence it is necessary that—

- (1.) In treating the iron with copper sulphate no carbon shall be lost as hydrocarbon.
- (2.) In treating the iron with the sulphuric and chromic acids no hydrocarbons shall be evolved.
- (3.) The iron shall be completely dissolved by the sulphuric and chromic acids.

To obtain complete oxidation without hydrocarbon formation, the solvent must consist of a sufficient quantity of chromic acid dissolved in sulphuric acid of requisite strength, and furthermore the iron must only be treated with such solvent after treatment with copper sulphate. If the iron be treated with sulphuric and chromic acid mixture without previous treatment with copper sulphate, then, be the concentration what it may, hydrocarbons will be evolved, the reason given being that the solution of the iron begins before the acid mixture has attained a temperature at which the chromic acid can complete the oxidation. If, however, the iron be completely disintegrated, the freed carbon is not oxidised until the liquid has reached a tolerably high temperature, near its boiling point; the oxidation is then complete.

The copper precipitated by the iron is not dissolved until the temperature of the acid mixture is sufficiently high to bring about complete oxidation of the carbon. Hence the iron need not be *totally* decomposed by the sulphate; it is only necessary that the coating of copper be sufficiently thick to protect the iron from the action of the acid mixture until a temperature sufficiently high for the oxidation has been attained. Finally, of course, all the iron must be dissolved. The iron used should be sufficiently fine to pass through a sieve of 1.5 mm. mesh.

The apparatus used for the determination is shown in the accompanying figure.



THE DETERMINATION OF CARBON IN IRON.

The test tube *a*, 140 mm. long and 20 mm. in diameter, is used for the solution of the iron. It is fitted with a rubber stopper, through which pass a tap-funnel *b*, and a delivery tube connecting *a* with the burette *c*; by means of the tap *k* communication between *a* and *c* may be shut off. The upper end of the burette is fitted with a rubber stopper, through which pass a tap-funnel *e* and the tube *d*. The distance between *a* and *c* is about 200 mm. The burette has a capacity of about 60 cc. and is conveniently made of the form shown; the lower portion is graduated into .1 cc. or .05 cc. It is provided with a tap *l*, and is connected by the rubber tubing *f*, with the movable flask *g*. The test-tube is enclosed in wire gauze, and may be heated by a burner or spirit lamp. The burette is provided with a water jacket.

Into *a* is introduced 0.2 gm. of wrought iron or steel, or 0.1 gm. of pig iron; 4 cc. of a saturated solution of copper sulphate are added, and action is allowed to proceed for 10 minutes, the mixture being stirred with a glass rod (in cases where an appreciable quantity of hydrocarbon is evolved only 3—4 minutes); 1.2 grms. of crystallised chromic acid is added, the whole is stirred, the glass rod washed with not more than 1 cc. of water, and the tube cooled; connection with the burette is made, the water in the burette being adjusted by the aid of *g* and *e*. From *b* now 8 cc. of sulphuric acid of sp. gr. 1.70 are added drop by drop, and the contents of *a* are carefully brought to the boil. Ten minutes' boiling suffices, after which the whole is allowed to cool for 15 minutes. By now pouring water through *b*, the whole of the gas may be transferred to the burette. The

carbon dioxide may be absorbed by caustic potash added from the tap-funnel *c*. This is the carbon dioxide corresponding to the carbon contained in the iron.

A determination occupies about 45 minutes. The results obtained are very satisfactory.—T. L. B.

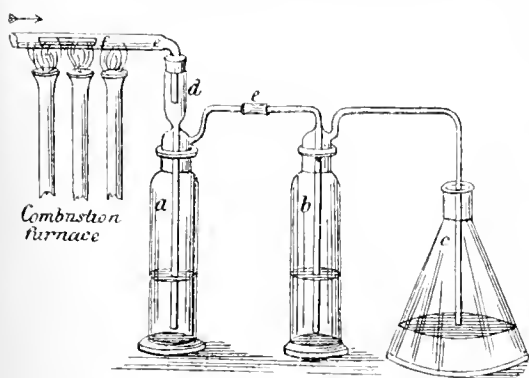
*Estimation of Sulphur in Iron.* L. Blum. Zeits. Anal. Chem. 29, 138—139.

It has been stated by Lucien (see this Journal, 1888, 401) that precipitation of sulphuric acid by barium chloride may be carried out in presence of free bromine, and that, therefore, there is no necessity to evaporate down previous to the precipitation. It is here pointed out, however, that in the case of some kinds of iron there is in reality no time saved by omitting the evaporation, for brominated organic compounds are formed, such as, for instance, bromo-propylene (Clötz, Ber. 7, 823), and these compounds separating in oily drops, stop up the pores of the filter and thus impede filtration. By evaporating to small bulk the organic compounds form a resinous film in the evaporating dish, and may be easily removed by ether. Further, it is usual in sulphur determinations to use not only bromine water, but even a solution of bromine in hydrochloric acid; and since Fresenius has shown that the solubility of barium sulphate is increased by the presence of acid, it is in such cases better either to evaporate down or else to neutralise the greater part of the acid by pure sodium carbonate.

—T. L. B.

*Determination of Sulphur in Inorganic Sulphides.* P. Jannasch. J. prakt. Chem. 1890, 41, 566—574.

THE author has already published (this Journal, 1890, 110) a method for determining sulphur in iron pyrites by burning the mineral in an oxidising atmosphere and absorbing the volatile products by bromine; he has now worked out the process for other metallic sulphides. The apparatus shown in the figure is designed to avoid the use of corks or rubber joints as far as is practicable, and to ensure perfect absorption. Not more than 0.6 grm. of the substance to be analysed is weighed out into a large porcelain boat and spread evenly therein so as to expose a large surface; the boat is heated in a combustion tube with an internal diameter of at least 15 mm. in



ESTIMATION OF SULPHUR IN INORGANIC SULPHIDES.

a stream of oxygen (150—200 moderate sized bubbles per minute), the heat being applied first at the end remote from the receiver *a* and regulated according to the rapidity with which the reaction proceeds as indicated by the tendency of the mass to glow spontaneously. *a* is about 203 × 36 mm. and has a capacity of 170 cc.; it contains about 60—70 cc. of bromine water; a like quantity is placed in *b*, and a little as a safeguard in the conical flask *c*. The end of the combustion tube leading into *a* is kept warm from the beginning of the operation to prevent condensation of sulphuric acid. The heating and passage of oxygen has to be continued for half to three-quarters of an hour, the temperature being considerably raised towards the last to decompose any sulphate that may have been formed. After oxidation is

complete, the stream of oxygen may be continued (or one of air substituted) until all cloudiness due to sulphuric acid vapour has disappeared. The contents of the absorbing vessels are then emptied into a roomy beaker, 1 cc. strong hydrochloric acid added and the liquid evaporated until all the bromine has been driven off; so important is this precaution that it is best when time permits to evaporate the whole of the aqueous liquid in a porcelain dish to ensure the absence of bromine. The removal of bromine by means of alcohol is not advisable, as small quantities of ethyl sulphuric acid are apt to be formed. The solution thus obtained, containing the sulphur of the substance analysed as sulphuric acid, is precipitated with a 10 per cent. solution of barium chloride in the slightest possible excess, and the barium sulphate washed by repeated decantation with boiling water. The filtrate is concentrated to precipitate any barium sulphate that may be held up by unremoved bromine.

After this description of the method, the author gives examples of its use with blende, copper pyrites, and antimony ore. With the first, particular care in powdering, and long continued heating are necessary to effect complete oxidation. The residue, consisting of zinc oxide contaminated with ferric oxide and gangue, is well fitted for analysis by the usual methods. The last two oxidise readily, and in the case of the copper pyrites, the rest of the analysis can be performed on the oxides left in the boat; but antimony is volatile to some extent, and a small quantity of the metal passes into the receivers. In consequence, a little tartaric acid, to keep it in solution, should be added before precipitating with barium chloride.

The method has the important advantage of allowing the precipitation of barium sulphate to take place in solutions free from salts of the heavy metals. Its accuracy is shown by the figures quoted in the original. Bromine is somewhat objectionable to work with, and the author hopes to replace it with hydrogen peroxide.—B. B.

*Determination of Cadmium as Sulphide by Precipitation with Sodium Sulphide.* W. Minor. Chem. Zeit. 14, 439—440.

THE substance to be analysed is dissolved in hydrochloric or nitric acid, and the lead precipitated by sulphuric acid. To the filtrate an excess of soda is added, which precipitates cadmium and iron. The precipitate is washed and treated with ammonia, which dissolves the cadmium, and this is precipitated from the filtrate by sodium sulphide. The precipitated sulphide takes some time to dry at 140° C. Or the cadmium may be estimated volumetrically by using a solution of sodium sulphide of known strength, and the hydrated oxide of iron as indicator. The least excess of sulphide is shown by the ferric hydrate turning black (see this Journal, 1890, 417).—A. L. S.

*The Citrate Method of Phosphoric Acid Estimation.* O. Reitmaier. Zeits. f. angew. Chem. 1890, 196—210. (See also this Journal, 1890, 218.)

SINCE this method has been doubtfully remarked on (this Journal, 1890, 219), and since conflicting statements as to the value of the method exist, the author has made a thorough examination of all the principles involved. His conclusions are that—

(1.) Solution in hydrochloric acid for citrate precipitation has the disadvantage that the precipitate obtained always contains silica, amounting to several mgrms.

(2.) Sulphuric acid gives a solution poor in silica, and the silica in this case may be taken as 1 mgrm.

(3.) When small quantities of calcium are contained in the solution (superphosphates), the ignited precipitate always contains several mgrms. of calcium as pyrophosphate, even though tolerable excess of ammonium citrate has been used.

(4.) In presence of manganese salts, gross errors, due to existence of manganese pyrophosphate in the ignited precipitate, may creep in.



(5.) Existence of iron and aluminium oxides in the precipitate is very slight if sulphuric acid solutions poor in silica be used. Even large quantities of iron and aluminium oxides may be present. Presence of these oxides in the solution only renders precipitation slower.

(6.) Slight admixture of magnesia must always be expected in the precipitate. Such admixture is lowest when precipitation is performed in ammoniacal solution (2.5 per cent.  $\text{NH}_3$ ). By precipitating in an approximately neutral solution, and in presence of large quantities of salts of the alkalis, the error may be great.

(7.) Against all these positive errors stands the error (equivalent to 1–2 mgrms. of magnesium pyrophosphate) due to incomplete precipitation of the phosphoric acid.

(8.) In controlling the citrate method by the molybdenum method, the means used for getting into solution must be taken into account.

In using the citrate method for determining in superphosphates the phosphoric acid soluble in water, variation from the result obtained by the molybdenum method should not exceed  $\pm 0.128$  per cent.

When Thomas slag and commercial phosphate are being examined in sulphuric acid solution, too high a result must be expected. This error in the case of solution containing very little silica and manganese may be very small, though no mean error can be given.—T. L. B.

*Detection of Benzoic Acid in Foodstuffs.* E. Mohler.  
Bull. Soc. Chim. 1890, 3, 414–416.

AN exceedingly small quantity of benzoic acid being sufficient for antiseptic purposes, the usual method of precipitating this substance as ferrie benzoate is not available. The following method, based on a colour reaction, is far more sensitive.

If a liquid such as beer for example, is to be examined, 100 cc. are taken, rendered alkaline, and evaporated to a syrupy consistency, then acidified with hydrochloric acid, mixed with clean sand, and extracted three times in succession with 20 cc. of ether; the ethereal solution is allowed to evaporate spontaneously, the residue moistened with 2 cc. of sulphuric acid, and heated to 240° till vapours of the acid are disengaged and the mixture blackens, owing to the destruction of the extractive matters dissolved by the ether; then a few crystals of sodium nitrate are gradually added until the liquid becomes colourless. The mixture is cooled and poured into excess of ammonium hydrate, the presence of benzoic acid being indicated by a yellow colouration, which, on the addition of a drop of ammonium sulphide, passes into brown-red. In this test symmetrical dinitrobenzoic acid is formed, which is reduced by the ammonium sulphide, first to amidonitro- and then to diamidobenzoic acid.—E. B.

*" $\alpha$ -Naphthol-Benzoin," a New Indicator.* R. Zolozicki.  
Chem. Zeit. 14, 605–606.

THE author has found that some of the benzeins (see Dobner, this Journal, 1890, 196) possess the qualities of indicators in a high degree, showing delicate colour-changes with acids and alkalis. The author has made experiments in this direction with  $\alpha$ -naphthol-benzoin which he prepares in a way somewhat different from Dobner, as follows:—2 mols. of  $\alpha$ -naphthol and 1 mol. of benzyltrichloride are diluted with benzene, allowed to stand for 24 hours, and then gently heated to 30–40° C. Benzyltrichloride in excess and benzene are subsequently evaporated. The substance is purified by dissolving it in dilute caustic soda, and afterwards precipitating it with hydrochloric acid. This has to be repeated several times, and the operation is finished by washing with water.  $\alpha$ -naphthol-benzoin, thus prepared, consists of a reddish-brown powder, soluble in very dilute alkalis with an intensely green colour, which changes to a reddish-yellow if dilute acids are added. Carbonic acid produces the same effect, the indicator being very sensitive to this gas. It is dissolved in alcohol, assuming a reddish-yellow colour. The author states that the results of his experiments show that  $\alpha$ -naphthol-benzoin belongs to the same group as

phenolphthalein, and that it is of similar sensitiveness as an indicator. Therefore, it can always be applied in volumetric analysis instead of phenolphthalein, carbonates only excepted, because its sensitiveness to the last-named salts is so great as to produce a change of colour before an acid salt is formed. But in most other respects,  $\alpha$ -naphthol-benzoin shows exactly the same properties as phenolphthalein. It may be applied in the following way: 10 to 20 drops of an alcoholic solution of 1 per cent. strength are added to the liquid to be analysed volumetrically; the titration is continued until a drop of the standard solution produces a change of colour from green to orange if standard acid, and from orange to green if standard alkali is added. Acids give a more precise reaction than alkalis. It may be mentioned as an advantage of this new indicator, that the colour-changes are also discernible in artificial light.—H. S.

*Pyrrrol, a Sensitive Reagent for a Group of Etheral Oils.*  
A. Ihl. Chem. Zeit. 14, 438.

THE author has previously observed that pyrrrol is a sensitive reagent for lignified tissue, and he finds that it also gives characteristic colours with such derivatives of allylbenzene, as cinnamic aldehyde, eugenol, safrol, anethol.

A dilute alcoholic solution of cinnamic aldehyde gives, with an alcoholic solution of pyrrrol and concentrated hydrochloric acid, first a yellow-red colouration, changing to dark red, and ultimately a dark precipitate.

In the same way melkenol gives a carmine-red colouration, pimentol also a carmine-red, sassafrasol a rose-red, esdragonal a carmine-red, and anisol and sternalisol a yellow. (This Journal, 1889, 421; and 1890, 555.)—A. L. S.

*The Detection of Olive Waste in Pepper.* Pabst. Monit. Scient. 1890, 470.

THE author recommends for this purpose either dimethyl-para-phenylenediamine which has been proposed by Würster for the detection of ground wood in paper, or thalline, which is used to stain the ligneous tissues of plants. The former reagent is conveniently prepared as follows:—Ten grms. of commercial dimethylaniline are mixed in a porcelain dish with 20 grms. of pure strong hydrochloric acid. 100 grms. of broken ice are next added, and then, little by little, with constant stirring, a solution of 7 grms. of sodium nitrite in 100 cc. of water. After half an hour 30–40 grms. of hydrochloric acid and 20 grms. of tin-foil are added. The reduction is allowed to go on for an hour, when the tin in solution is precipitated by means of metallic zinc. The decanted and filtered liquid is treated with a slight excess of sodium or potassium carbonate, and the turbidity thus produced is redissolved by the addition of acetic acid. Finally, 10 grms. of sodium bisulphite are added, and the whole is diluted to 2 litres. In testing pepper, 1–2 cc. of the above solution are placed in a shallow dish, and a pinch of the pepper is sprinkled into the solution. In a few minutes the particles of olive waste assume a splendid red-carmine colour; whilst the grains of pepper remain unaltered or are coloured only a faint pink. If some water be now added, the heavy particles of olive fall to the bottom, and collect together, when they may be detected with the greatest ease. Of course, any other ligneous tissue present in the pepper, such as ground nut-shells, will also be coloured red by this reagent. Thalline sulphate, in  $\frac{1}{2}$  per cent. solution, stains the olive particles a beautiful orange, easily detected. The colour, however, takes longer to develop than the red produced by the preceding reagent, and the solution of thalline sulphate does not keep well. The colours given to olive waste by the above reagents are soluble in water after some time, the waste itself assuming a brownish or blackish colour.—H. T. P.

*Experiments on Gum Arabic and Gum Senegal.* L. Liebermann. Chem. Zeit. 14, 665–666.

GUM arabic is often adulterated with gum Senegal, the latter being sometimes sold in its stead. If the gum has not been powdered, the appearance is sufficient to show

which variety is chiefly present. Gum arabic forms round or irregular masses, varying in colour from colourless to brown, which reflect light strongly, almost appearing to be crystalline. Gum Senegal is usually colourless or pale yellow, with a surface resembling ground glass, the pieces cylindrical or worm-like. It occurs occasionally in round masses resembling a mulberry.

On dissolving either gum in water, only small particles of wood are left, which, according to Kramsky, are usually red in the case of gum arabic, but black with gum Senegal. Other gums, cherry, for example, are only partially soluble in water, leaving a swollen residue, which only dissolves after long digestion.

With potassium hydrate, and a few drops of copper sulphate solution, both gums give a blue precipitate, which, in the case of gum arabic, is larger in quantity, coherent, and rises to the surface, while the other is more flocculent and remains diffused in the liquid. The precipitates are only slightly soluble on heating, and are not reduced even by boiling. Dextrin yields a similar precipitate, easily soluble on warming, and which is completely reduced on boiling for some time. On prolonged heating with dilute potassium hydrate, solutions of gum arabic and of dextrin become amber coloured, those of Senegal are only slightly coloured.

Mixtures of gum arabic and Senegal behave to the copper test like Senegal alone, but with potassium hydrate they assume the amber colour. Mixtures of gum arabic or Senegal and dextrin with the copper test behave like gum arabic alone, and if the amount of dextrin is not too small, reduction takes place on boiling. When only small quantities of dextrin are present, the copper precipitate after thorough warming may be filtered off, and the filtrate boiled; reduction will then take place. When all three substances are present, the precipitate should be washed, dissolved in

dilute hydrochloric acid and a large excess of alcohol added. The precipitated gum is allowed to settle for a day, washed with alcohol, dried, and examined as above.

Gum arabic may be systematically examined in the following manner:—The powdered substance is treated with luke-warm water; a gelatinous residue indicates other gums. The aqueous solution is mixed with an excess of potassium hydrate and a few drops of copper sulphate, warmed and filtered, and the filtrate examined for dextrin by boiling, the precipitate being tested as above.

It is usually stated that gum Senegal is more hygroscopic than gum arabic, but when dried at 105° C., gum Senegal lost 13.39 per cent. of water and gum arabic 14.56, and on exposure to moist air for 24 hours, the former gained 6.15 per cent., the latter 6.34.—F. H. L.

#### *Thermo-Chemical Investigations on Silk.* L. Vignon. Bull. Soc. Chim. 1890, **3**, 165—110.

SIXTEEN skeins of silk (cultivated), of the same quality, reeled and spun under precisely similar conditions, were dried and weighed, and then half of them boiled-off, re-dried and weighed, the average loss in weight being 22.91 per cent. The ungummed skeins were left exposed to the air in the laboratory to cause them to regain their normal amount of moisture; then they were immersed in certain solutions in a calorimeter, the skeins of raw silk being similarly treated. The liquids employed in the calorimeter consisted of seven normal solutions as follow: potassium, sodium, and ammonium hydrates, sulphuric, hydrochloric, and nitric acids, and potassium chloride, along with distilled water, in each case 500 cc. being used.

The results are shown in the table below:—

Reagents.	Raw Silk.		Ungummed Silk.	
	Calculated for 100 Grms.	Calculated for the Mol. Wt. $(C_{10}H_{12}N_4O_6)$ in Grms.	Calculated for 100 Grms.	Calculated for the Mol. Wt. $(C_{10}H_{12}N_4O_6)$ in Grms.
Heat Units evolved. Temperature about 12°.				
Water.....	0.10	3.50	0.15	5.29
Potassium hydrate*.....	1.35	47.00	1.50	45.25
Sodium hydrate*.....	1.55	53.95	1.30	43.25
Ammonium hydrate*.....	0.65	22.65	0.50	17.40
Sulphuric acid.....	0.95	33.10	0.90	31.35
Hydrochloric acid.....	0.95	33.10	0.90	31.35
Nitric acid.....	0.90	31.35	0.85	29.60
Potassium chloride.....	0.20	6.95	0.19	3.59
Total.....	6.65		6.00	

\* Alkalies act on raw silk, removing some of the silk-gum.

The total numbers of heat units evolved are 6.00 in the case of ungummed silk and 6.65 in that of raw silk; sericin, consequently, causes the evolution of more heat than does fibroin, although the two bodies are chemically closely related; on calculation, it is found that sericin evolves 1.47 times more heat than fibroin. The author hopes that further investigations in this field may be of use in extending our knowledge of the theory of dyeing.—E. B.

*Eucalyptus Oil.* Schimmel and Co. Zeits. Anal. Chem. **29**, 222—223.

See under XX., page 761.

#### *On the Examination of Wax.* H. Röttger. Chem. Zeit. **14**, 606—607.

THE determination of the specific gravity of wax may in some cases be used as a preliminary test, pure bees-wax having an average specific gravity of 0.960. If the substance in question differs considerably from this, an adulteration is proved. Hübl's test (Chem. Zeit. **13**, 1375) is the most reliable for the quantitative and qualitative examination of wax, and the author having most carefully examined the various other methods, has come to the conclusion that the result in most cases is very unsatisfactory. Two methods only are mentioned which give good results, if the adulteration of wax with stearic acid has to be proved.

1. Fehling's method (Dingl. Polyt. J. **147**, 227). He recommends boiling the substance four or five minutes with

20 times its weight of alcohol, and afterwards letting it stand for several hours. Then it is filtered and the filtrate mixed with water. If the wax was pure, the liquid remains perfectly or almost transparent, but if it was adulterated with stearic acid, it loses its transparency, a reaction which is plainly perceptible even if the adulteration amounts to as little as 1 per cent. of stearic acid.

2. Benedikt (*Analyse der Fette*, 291) advises boiling the substance with sodium carbonate. The solution on cooling becomes solid when sodium stearate is present. This method is also reliable, but not so exact as that of Fehling.

The two methods just mentioned, together with Hübl's and the specific gravity test, seem to be all we have practically to rely upon, to prove adulterations in wax.—H. S.

#### Determination of Mineral Oil in Fatty Oils. A. Grütner. *Zeits. f. angew. Chem.* 1890, 261.

Houx's method (this *Journal*, 1888, 696), consisting in saponifying the mixture of mineral and fatty oil and extracting the dried soap with chloroform, has given excellent results in the hands of the author save when the proportion of mineral oil is large, in which case a pasty mass is formed, difficult to extract. He accordingly modifies it thus: 2—3 grms. of the oil are saponified in a porcelain basin 8—10 cm. in diameter with 20 cc. of alcoholic soda containing 20—25 grms. of caustic soda in a litre of 95 per cent. alcohol. After the alcohol has been driven off, sand (purified by treatment with hydrochloric acid) is stirred in, and the mass wrapped in filter-paper and extracted with chloroform in a Soxhlet's apparatus. The purification of the sand is necessary, as any lime present in it would form a lime soap which is soluble in chloroform. The chloroform is best purified by distillation with strong sulphuric acid. The results of the method are accurate.—B. B.

#### Determination of the Solidifying Point of Lubricating Oils.

A. Martens. *Mitt. k. tech. Versuchst.* 1890, 53—71.

A COMPARISON was instituted between the various forms of apparatus in use by several Government railways for determining the solidifying points of lubricating oils, and the method adopted by the Versuchsanstalt (this *Journal*, 1889, 423).

(1.) The oil is cooled by exposing the test tube containing it to a bath of ether through which a stream of air can be drawn, and its condition judged by inclining the tube. The method is unsatisfactory, the cooling with ether being a grave defect, as it is costly, dangerous, disagreeable, and, unless the air be previously dried, liable to cause stoppage by blocking the tubes with ice. The time commonly allowed for cooling is also insufficient (the importance of this condition is shown in the preceding abstract), and results compare unfavourably with the standard method of the Versuchsanstalt.

(2.) The oil is solidified in a freezing mixture, removed therefrom, and the temperature at which it begins to flow under a head of 30 mm. of oil noted. This also met with disapproval, wide discrepancies occurring with slight alterations of the conditions of experiment; besides it does not determine the *solidifying* point but that of incipient fusion.

(3.) The apparatus and method are of a character similar to those of (2), and the same objections hold good. The use of ether as a cooling agent leads to the presence of the disadvantages mentioned under (1).

(4.) Since the description of the standard method of the Versuchsanstalt appeared, a new process has been devised, the apparatus for which is illustrated herewith. It may be briefly described thus:—The oil is placed in a U-tube (Fig. 1) and exposed to a known and constant low temperature by the use of a bath of one of the frozen salt solutions prescribed for the standard method (this *Journal*, 1889, 423), and its condition ascertained after a sufficient period of cooling by exposing it to a pressure equal to a column of water 30 mm. in height. Fig. 2 shows a convenient method of obtaining a constant pressure for this purpose. A gas-holder *a* is connected with a tube (having its end

turned up and rounded as shown in Fig. 3) immersed to such a depth in any suitable fluid (e.g. a non-volatile oil) that air from the gas-holder can escape from it when the water-manometer *c* shows a greater pressure than 30 mm.; this device is necessary to prevent fluctuations in the pressure due to alterations of temperature. A water pump may be substituted for the gas-holder. In carrying out a number of determinations a set of U-tubes may be held by the stand shown in Fig. 4, the design of which is obvious. The frame carrying them can be lifted bodily out of the containing vessel. The whole of the metal parts of the apparatus are nickel-plated to decrease their liability to corrosion from the salts used. The concordance of the results obtained by this method and its ease of working make it preferable to those previously described.

Fig. 1.

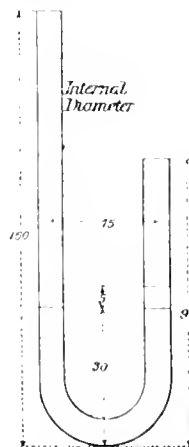


Fig. 3.

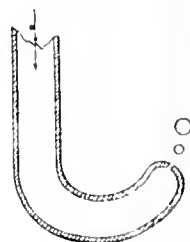


Fig. 2.

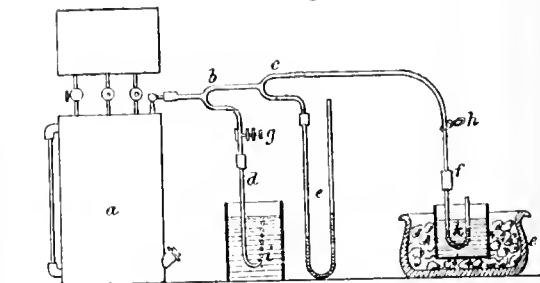
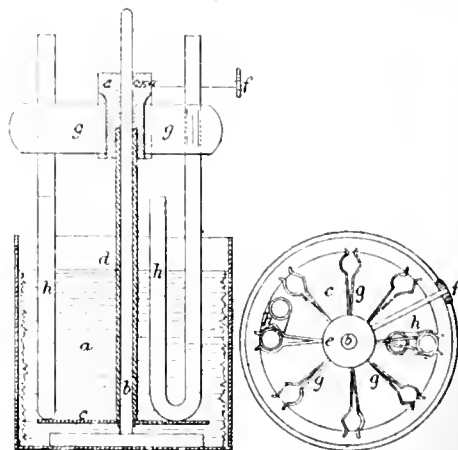


Fig. 4.



(5.) The simple plan of cooling the oil in a capsule immersed in a freezing mixture, stirring it with a thermometer, and noting the change of state it undergoes, is used, and has little to recommend it, the serious personal equation (to get rid of which is largely the object of most of the more complex methods) being particularly objectionable.

(6.) The standard method of the Versuchsanstalten has already been described (this Journal, 1889, 423), and does not need repetition. It has been found lately that a large increase in the width of the vessel in which the oil is cooled causes it to remain fluid at a lower temperature than under normal conditions.—B. B.

*The Melting and Solidifying Points of some Fatty Bodies and Mixtures.* A. Terrell. Bull. Soc. Chim. 1890, 3, 195—200.

See under XII., pages 743—744.

*The Density of Resin Oil and of Essence of Turpentine.* A. Renard. Monit. Scient. 1890, 469.

The author has determined the density and coefficient of expansion of these oils, so that their density observed at any convenient temperature may be reduced to their density at 15° C. by means of the formula—

$$D_{15} = D \mp Ct$$

in which  $D_{15}$  = density at 15° C.;  $D$  = density observed;  $t$  = difference between the temperature of observation and 15° C.

$Ct$  is added to  $D$ , when the temperature of observation exceeds 15° C.; and it is subtracted from  $D$  when the temperature is below 15° C.

#### *Pale Resin Oil (First Distillation).*

Average density at 15° C. .... 0.9823  
Coefficient of expansion ..... 0.000663  
 $D_{15} = D \mp 0.00061 t$

#### *Rectified Resin Oil (Second Distillation).*

Average density at 15° C. .... 0.9712  
Coefficient of expansion ..... 0.000673  
 $D_{15} = D \mp 0.00065 t$

#### *Green Resin Oil.*

Average density at 15° C. .... 0.9901  
Coefficient of expansion ..... 0.00066  
 $D_{15} = D \mp 0.00065 t$

#### *Blue Resin Oil.*

Average density at 15° C. .... 0.9810  
Coefficient of expansion ..... 0.000717  
 $D_{15} = D \mp 0.00070 t$

#### *Essence of Turpentine.*

Average density at 15° C. .... 0.8690  
Coefficient of expansion ..... 0.000918  
 $D_{15} = D \mp 0.000789 t$

—H. T. P.

*Estimation of Anhydrous Glycerol in Commercial Glycerol.* E. Deiss and C. Deiss. Les corps Gras Industr. 16, 293.

THE method depends upon the fact that the absorption of water by a constant weight of a mixture of glycerol and pure phenol is proportional to the degree of concentration of the glycerol. Into a flask of about 100 cc. capacity are introduced exactly 10 grms. of the glycerol under examination, and 6 grms. of pure crystallised phenol, which has been melted; the whole is well mixed and allowed to cool. A solution is made up containing 50 grms. of pure crystallised phenol to 1,000 grms. of water. This solution is carefully run from a burette into the flask containing the above mixture of the glycerol and phenol until permanent milkiness is just attained. In the

case of anhydrous glycerol 28.15 cc. of this phenol solution are required; and for every per cent. deficiency in the glycerol at the same temperature (not stated) 0.39 cc. less is required, as experiments have conclusively shown.

If  $q$  be the number of cc. of phenol solution used, the percentage of glycerol contained in the sample examined is—

$$100 = \frac{28.15 - q}{0.39}$$

For a temperature of 11° C. the percentage is —

$$100 = \frac{21.40 - q}{0.28}$$

In order not to have to find the equations corresponding to various temperatures the authors recommend that all experiments should be carried out at a temperature of 11° C. by surrounding the flask with water kept at this temperature. Presence of extractive substances, salts, and colouring matter are said not to affect the result. The method gives very good results with commercial glycerol.

—F. L. B.

*Estimation of Starch-Flour in Grain.* Z. von Milkowski. Zeits. Anal. Chem. 29, 134—136.

ASBÖTH first removes fat by extraction with ether (this Journal, 1889, 574), and then estimates the starch in the product by the baryta method (this Journal, 1887, 608). The author has made comparative experiments by extracting with carbon bisulphide, and his results agree perfectly with those of Asböth.

Mäcker's method is as follows:—3 grms. of finely powdered substance are made into a paste with 50 cc. of water in a water-bath at 100° C.; the whole is cooled to 70° C., and 5 cc. of malt extract (100 grms. of green malt to 500 cc. of water) are added, the temperature being kept at 70° for some minutes. To prevent darkening of the solution a 1 per cent. solution of tartaric acid must be added. The liquid is next subjected to a pressure of three atmospheres, is cooled and has 5 cc. of malt extract added to it, after which it is again warmed to 70° C., and at the end of about 20 minutes the starch is completely dissolved. A portion of the filtered solution is now inverted with hydrochloric acid (sp. gr. 1.125), and in this by means of Fehling's solution the sugar, and indirectly the starch, is estimated. Allowance must be made, of course, for the 10 cc. of malt extract added.

Results obtained by the author by this method and by Asböth's method agree exceedingly well together. Methods which depend on direct inversion by acids cannot be reliable, since other substances, such as cellulose and pectin, are calculated as starch in such cases.—T. L. B.

*Determination of Nitrogen in Manures.* E. Aubin and J. Quenot. Bull. Soc. Chim. 3, 1890, 322—326.

THE authors first cite experiments to prove that, whether sugar, or a mixture of sulphur and calcium oxalate, be added to the soda lime, the whole of the nitrogen of nitrates is not obtained as ammonia by the combustion process, and that the addition of oil in the Kjeldahl process is open to the same objection. A mixture of sulphuric acid, phenylsulphuric acid, and zinc gives a much better result; but still there is a loss, 0.0667 grm. N being obtained from potassium nitrate instead of 0.0692 grm. If the Kjeldahl process be employed on an organic manure containing nitrates, some of the nitrate is reduced; and if a mixture of ammonium salt and nitrate only be analysed by this process, some of the ammoniacal nitrogen is lost by oxidation.

The best way to obtain the total nitrogen (organic, ammoniacal, and nitric) by the Kjeldahl process is to use a mixture of 30 cc. sulphuric acid, 3 cc. phenylsulphuric acid, 1 grm. zinc, and 0.5 grm. mercury with the manure; but even then there is a slight deficiency in the nitrogen obtained. Attempting to first eliminate the nitric nitrogen, by digestion with ferrous chloride, the authors find the ammoniacal nitrogen rendered too low. Ferrous sulphate gives better results.

The authors recommend, however, the following process, which permits of the determination of the organic and ammoniacal nitrogen, together or separately, the nitric nitrogen being determined in a fresh portion by Schloesing's process (see this Journal, 1884, 379, and 1888, 692). One gram. of the manure is exhausted on a filter by 30--40 cc. of a 2 per cent. solution of tannin, by which the nitrogenous organic matters are rendered insoluble, if not already so. The residue and filter, deprived of nitrates, and of most of the ammonium salts, are attacked by the Kjeldahl method, and when ready for the final distillation, the filtrate is remixed with the contents of the flask, and the organic and ammoniacal nitrogen determined together.

To determine them separately, it suffices to remove the ammonium salts entirely by the tannin solution; and this can be done even when ammonio-magnesium phosphate is present, by digesting 1 gram. of the manure for 15 hours with 0.7 gram. tannin and 150 cc. salt-water. The residue is treated by Kjeldahl's process for the organic nitrogen, the filtrate by distillation with sodium hydrate for the ammoniacal nitrogen, and the nitric nitrogen is determined in a fresh portion by Schloesing's process.—J. M. H. M.

*The Vacuum Method of Drying Barley and Malt, with a Direct Determination of the Extract in Beer and Worts.*  
C. N. Rieber. Zeits. f. d. ges. Brauw. **13**, 97.

See under XVII., page 756.

#### PATENT.

*Apparatus for Estimating the Quantity of Glucose or Grape Sugar in Fluids.* A. W. Gerrard, London, Eng.  
Pat. 18,856, November 25, 1889. 4d.

The apparatus consists of a burette, from which the sugar solution is run in the ordinary way into Fehling's or any other copper solution. In working with this apparatus it is necessary always to use such an amount of Fehling (say 10 cc.) or other copper solution as is exactly equivalent to 0.05 gram. of glucose; and further, the sugar solution must be diluted to 20 times its volume. Under these conditions, the correct percentage of sugar in the original solution may be read off direct from the burette.—A. J. K.

### New Books.

CHEMISCH-TECHNISCHE ANALYSE. HANDBUCH DER ANALYTISCHEN UNTERSUCHUNGEN ZUR BEACHTSICHTIGUNG DES CHEMISCHEN GROSSRETRIEBES UND ZUM UNTERRICHTE. HERAUSGEGEBEN VON DR. JULIUS POST. Zweite vermehrte und verbesserte Auflage. Zweiter Band. Zweite Lieferung. 1890. Braunschweig: Friedrich Vieweg und Sohn. London: H. Grevel and Co., 33, King Street, Covent Garden.

This volume forms part II. of the second volume of Post's great work on Chemical-technological Methods of Analysis. (See this Journal, 1888, 872.) The work is illustrated with 77 excellent engravings and the volume is mainly devoted to the subjects of Starch, Dextrin, Sugar and the Fermentation Processes, and products such as Wine, Beer, Spirits, and Vinegar.

Under Spirits and Vinegar, also Wood Spirit appears and receives full treatment. The methods of Krell, Krämer and Grodzki, and Bardey and Bordet, are described, for estimating the Methyl Alcohol, Acetone, and other constituents.

The Chemical and Chemico-physical Methods for Testing Sugar-juices, Syrups, &c., are very fully given and well illustrated, and the best methods for estimating the Constituents of Wines and Spirits are described in considerable detail.

### Trade Report.

#### TARIFF CHANGES AND CUSTOMS REGULATIONS.

##### PHILIPPINE ISLANDS.

##### Customs Tariff.

*Note.*—Kilog. = 10 Hectogrammes = 2.204 lb. avoird. ;  
litre = 22 Imp. gallons.

The following is a statement of the rates of Customs duty now levied on imports into the Philippine Islands:—

Classification of Articles.	Rates of Duty now leviable.	
	Pes. Cts.	
Mineral oils .....	100 kilos.	1.10
Wax .....	Kilo.	0.04
Wax, worked .....	"	0.12
Copper, brass, zinc .....	"	0.05
Matches of any kind .....	"	0.05
Caoutchouc, manufactured .....	"	0.22
Perfumery of any kind .....	"	0.10
Hides tanned .....	"	0.12
Pharmaceutical and chemical products not prohibited by sanitary regulations .....	8 % ad val.	
Candles .....	Kilo.	0.05

The importation of the following articles is prohibited:—

(a.) Articles of food containing materials injurious to health (fuchsine, salicylic acid, &c.).

(b.) Pharmaceutical products the composition of which is kept secret (such as Holloway's pills).

For the importation of explosives, such as gunpowder, dynamite, &c., a special permit is required.

The importation of the following is only permitted to certain individuals:—

Opium imported by the farmer of this tax or by chemists in small quantities and for medicinal purposes.

The following are free of import duty:—Manures, lime, cement, plaster.—*Board of Trade Journal.*

#### EXTRACTS FROM DIPLOMATIC AND CONSULAR REPORTS.

##### BRITISH GUIANA.

##### Drug Exports.

Among the exports from the colony during the years 1887 and 1888 were the following articles of interest to druggists:—

	1887.	1888.
Gum (Balata) .....	£ 3,428	£ 14,069
Gum (other) .....	2,021	479
Isinglass .....	338	107
Arrowroot .....	153	25

*Balata and India-rubber.*

It is gratifying to record the growth of another industry in the collection of balata. The legislation of 1887, placing the collection of this and other gums on a proper footing, has been eagerly taken advantage of, and 25 balata licences were issued during the year, the result of which has been to increase the export of this useful article from 80,942 lb. in 1887 to 248,484 lb. in 1888.—*Chemist and Druggist*.

## CAPE COLONY.

*Drugs and Chemicals.*

In 1888, drugs and chemicals were imported into the Cape Colony to the value of about 69,760/.

*Soap.*

There is a duty of 4s. 2d. per cwt. on foreign soap imported into the Cape, and this protection accounts for the existence in the colony of 10 soapworks, of which the two most important ones are those of Messrs. Low and Smithers. Notwithstanding the existence of this industry, the colony imports a considerable quantity of common soap, especially for wool-washing. The indigenous product sells at 9s. to 10s. per  $\frac{1}{2}$ -cwt. box, while the foreign article is slightly higher in price. The imports in 1888 were:—From England, 5,261,354 lb.; from Natal, 19,560 lb.; from the United States, 35,650 lb.; from South Australia, 10,046 lb.; from Germany, 13,524 lb.; from the Argentine Republic, 80 lb.—*Ibid.*

## CHINA.

*Aniline Dyes.*

It is worthy of remark that no aniline dyes were imported into Newchwang last year, says the British Consul in that city. It is said that the Chinese have come to dislike these dyes, and to find that those which they make themselves are more serviceable and cheaper in the end. So they are returning to the use of sapan-wood and their own dyestuffs. On the other hand, the Consul at Kewkiang observes that dyes, principally of German manufacture, continue to form a considerable article of trade. Many of the colours chosen are not in the best taste, magentas and aniline greens, blues, and pinks being the favourite shades.

*Chinese Camphor.*

The camphor trade of Formosa does not show any signs of improvement. The restrictions formerly imposed by the Government monopolists are nominally removed, but the difficulties and risks of getting the camphor down to the coast are so great that foreigners are not inclined to engage in the trade, notwithstanding the very high prices that have ruled in the Hong Kong market lately. Chinese traders decline positively to deliver camphor at the port, and do not care much about touching the business at all. Practically, it may be said, the monopoly still exists, and the entire export of 1889 was only 709 cwt.

*Turkey Opium.*

The consumption of Turkish opium in certain parts of China is largely on the increase. In the island of Formosa, for instance, it has almost entirely replaced Indian opium, as the following figures will show:—

—	Indian.	Persian.	Turkey.
	Cwt.	Cwt.	Cwt.
Imports in 1889 .....	445	25	1,987
„ 1888 .....	618	24	1,707

Eight years ago Indian opium from Benares, with a small quota of Malwa, took about 50 per cent. of the trade, the remaining moiety being divided between Persian and Turkey. Persian and Malwa have now practically disappeared; Turkey monopolises about 80 per cent. of the demand, leaving only 20 per cent. to Benares.

*Native Opium.*

It is also certain that the native-grown drug is steadily gaining a footing in Formosa, but as it does not pass through the foreign Customs statistics are not obtainable; but it is well-known that there is a steady re-export of foreign opiums in native junks to the mainland, where they are exchanged for the opiums of Szechuan and Yunnan, which are then brought back to Tamsui for local consumption.

It is estimated that 80,000 lb. of Chinese-grown opium were imported in that city during the year 1889; the most popular coming from Tang Shan Hsien and Feng Hsien, in the neighbouring province of Kiangsu. A Szechuan firm has just set up business in Waku as well, and there is every probability of the native drug, which is now far better prepared than formerly, besides being only about half the price of the Indian, coming more and more into favour.

*Drug Exports.*

The following figures show the export of drugs during the years 1888 and 1889 from Ichang, the Chinese inland trade centre on the Yangtze river, which is the principal place at which goods brought from the interior pass into commerce for foreign countries. The question of the establishment of regular steam navigation with Ichang recently led to the conclusion of a special treaty with China:—

—	1889.		1888.	
	Lb.	£	Lb.	£
Medicines .....	..	46,613	..	79,380
Musk .....	4,056	71,856	4,269	85,537
Nuttsalls .....	907,708	12,786	371,656	7,911
Rhubarb .....	308,118	13,460	260,227	11,065
Safflower .....	104,423	11,062	117,556	12,431
Wax, white .....	1,543,115	95,211	2,081,041	117,540

*White Wax.*

The diminished export of white wax is due to low prices ruling in Shanghai for most of the year; and still it hardly pays to export it. Over 40,000 lb. of this substance was sent to Peking for use at the Emperor's marriage, tallow and colours being the other contributions from Szechuen.—*Ibid.*

## FRANCE.

*Dyewoods in Havre.*

The large decrease in the quantity of dyewoods imported into Havre in 1889 must be put down to the disturbed state of the island of Hayti, and to the great difficulty of transport from the interior where the wood is now procured. Thus fustic showed a decrease of 11,500 tons. It must also be noted that three Havre manufacturers have erected works at Riga for extracting dyes, and that they receive the raw material there direct. This step has been necessitated owing to the increase in the Russian Customs duties on dyewood extracts.

*Oils and Oil Seeds in Marseilles.*

The imports of oleaginous seeds into Marseilles for the year 1889 amounted to 314,265 tons, against 278,527 tons in the previous year. The crops of ground-nuts in the French colonies (Coromandel coast) were very large, about 110,000 tons, and the imports of this article alone attained 97,374 tons, against 73,061 tons in the previous year. Likewise of coprah (produced chiefly in the South Sea Islands, Straits Settlements, and Java) Marseilles received 46,000 tons, against 35,500 tons in the preceding year. The whole 314,265 tons of oleaginous seeds have been crushed by the local mills, and the oils produced met with a ready and remunerative sale to local soapmakers, as well as for export, showing a very prosperous condition of this great industry.

The coprah and palm-kernel trade continues to increase steadily, and the quantity of coprah and palm-kernels crushed in Marseilles in 1889, excluding the old stocks lying over from 1888, exceeded 80,000 tons, yielding upwards of 10,000 tons of concrete oil, chiefly consumed by local soap-mills. Imports of palm oil amounted to 12,039 tons, against 11,551 tons in 1888. The French Government has granted a large subsidy to one of the leading companies of steamers to run a regular line to and from the different ports on the West Coast of Africa, which is a further facility in the way of extending the direct imports of palm-oil and palm-kernels, the chief articles exported from that country.

The oil-seed crushers also had a sound and profitable trade throughout the year, their export trade having attained 13,110 tons against 36,783 tons in 1888.

In soap also there was an increase of 1,700 tons, the year's exports being 10,184 tons, against 8,470 tons in 1888. The quantity of candles exported from Marseilles amounted to 4,100 tons, against 3,967 tons in the previous year. This industry is very prosperous, and tends to increase further.—*Ibid.*

#### ITALY.

##### *Chemicals.*

Acids and chemicals used for the extermination or prevention of phylloxera, such as sulphate of copper, are purchased in large quantities. Pigments for house-paintings are also in demand, as well as a few products for leather dressing and dyeing, of which branches of industry the city of Cagliari is an important centre; but these goods are mostly furnished by other parts of the island, where the principal raw materials are found in abundance.

#### JAPAN.

Consul Playfair, of Hakodate, reports that last year's exports of sulphur amounted to 14,421 tons, valued at 34,630*l.*, as against only 3,609 tons in the previous year. Prices showed no material change. One of the causes of the smallness of the exports in 1888 was that freights were low, and in consequence tonnage was not available.

#### NORWAY.

##### *Cod-liver Oil.*

The total exports of cod-liver oil have been :—

	1884.	1885.	1886.	1887.	1888.
Tons.....	12,500	14,700	16,900	16,000	20,200
Value..... <i>£</i>	317,000	290,000	285,000	256,000	312,000

#### RUSSIA.

##### *The Petroleum Trade.*

The exports of petroleum products from Batoum, the port through which the output of the Baku petroleum wells is brought into commerce, has grown to 649,085 tons (value, 3,023,300*l.*) in 1889, from 450,326 tons (value, 1,724,446*l.*) in 1888. All other branches of business, especially those relating to imports, occupy a very subordinate rank in the local market. On the other hand, the import of such foreign goods as are absolutely required for the petroleum trade—as, for instance, tin plates, caustic soda, &c.—has become a business of considerable importance. If it were not for the demand for such articles there would be scarcely any cargoes for ships arriving in Batoum to load petroleum. The imports of caustic soda are about 4,000 tons per annum. Although the aggregate quantity and value of the petroleum trade considerably surpassed that of any preceding year, the business, so far as profitability goes, was upon the whole less satisfactory. Such firms trading in petroleum who at the same time are owners of productive petroleum wells must have found the year an exceptionally profitable one, because of the great rise in the price for crude oil; but

the commercial community as a body have not been able to realise profits in any way proportional to the expansion of business.

#### SPAIN.

##### *Quicksilver.*

The shipments of quicksilver (all of which goes to England) from Corunna, in 1889, show a large increase over the previous year, the value being returned as respectively 5,864*l.* and 11,600*l.* This quicksilver is produced by the Asturian mines in the north of Spain. These are much less important than the Almaden mines in the south; but, as the Asturian mines are being exploited much more extensively now, the quantity they produce is likely to be larger in future.

##### *Drugs for Barcelona.*

The total weight of manufactured goods imported by sea into Barcelona in 1889 was 52,742 tons, against 51,862 tons in 1888. There was an increase in the importation, among other goods, of colouring extracts, chemical and pharmaceutical products, soap, and india-rubber goods. About 17,790 tons, or one-third of total amount, came from Great Britain, and consisted, to a great extent, of drugs and chemicals. The imports by land included 10,361 tons of drugs, chemicals, and colours.

##### *Sulphate of Copper.*

Great attention has been given to the question of the use of sulphate of copper in vine disease in the autumn of 1889, and a committee of investigation arrived at the conclusion that the poor results shown for the use of sulphate of copper had been owing to the inferior character of the article that had been employed. It was resolved that the committee should import a quantity of pure sulphate of copper, and supply it to municipalities in the province at cost price, import duty being remitted by the Government. The total amount asked for by the entire province, however, was only 45 tons, and a contract was made for the supply of this quantity to the committee by Messrs. Vivian and Sons, of Swansea. It is thought that other municipalities will avail themselves of this system of obtaining pure sulphate of copper at a low rate as soon as its advantages come to be known.

#### UNITED STATES.

##### *Californian Quicksilver.*

The total production of quicksilver in California in 1889 amounted to 25,650 flasks, as against 33,250 flasks in 1888. The average price for the year is 9*l.* per flask.—*Chemist and Druggist.*

#### WESTERN AUSTRALIA.

##### *Sandalwood.*

The exports of sandalwood from the colony in 1889 were 4,470 tons, valued at 33,525*l.*—*Ibid.*

## MISCELLANEOUS TRADE NOTICES.

#### HENEQUEN PRODUCTION IN YUCATAN.

In the State of Yucatan, in consequence of stagnation in the henequen industry, owing to the conflicting interests of producers, the *Mexican Financial Review* reports the existence of distress among the poorer classes. The large profits obtained by the growing of henequen had induced land-owners and others to neglect the production of cattle wheat, and other primary necessities of life, with the result that food supplies had been greatly enhanced in price to the inhabitants of that part of the Republic.

Of the 240,000 packages of henequen sent to the United States during the past year, about 225,000 went into consumption, and the balance remained on hand for the



present year. In view of the increased area of cultivation which has since been developed, the supply during the present year cannot fall far short of 260,000 packages. During the months of January and February only about 35,000 were exported, which, added to the surplus from the previous year, would make a total of about 50,000 on hand in the United States at that time. According to data received up to March 15th, the stock on hand in the United States was about 30,000, so that only about 20,000 had gone into consumption. There is, therefore, every prospect of an increased supply being encountered by a considerably diminished demand.—*Board of Trade Journal*.

#### GALICIAN PETROLEUM INDUSTRY.

*Bradstreet's* for the 31st May has the following:—

Although still of very moderate proportions, and but little heard of outside of Austria, the petroleum industry of western Galicia is said to be older than the Pennsylvania industry by nearly 10 years. The advance in the world's markets of the American product and the extraordinary growth of the supply acted as a discouraging feature in the development of the Austrian deposits just as the use of petroleum as an illuminating material began to be understood there. A State investigation, made public in 1880, according to a writer in the Vienna *Neue Freie Presse*, showed that the Galician oil-producing region embraces almost the whole northern slope of the Carpathian Alps from New Sandee to the Bakovina, and is of far greater extent than that of Pennsylvania. The strata are pretty regular, and the surface indications of the presence of oil are well marked. Two American explorers, Messrs. Macgarvey and Berghien, who discarded the old methods of trenching and adopted the most modern American tools for boring, seem to have met with considerable success, and to have trained the native workmen till they are in every way capable of carrying out the work on the most approved lines; but in spite of this capital seems to have fought shy of the industry, and only some small works, but nothing on any large scale, has been attempted. The quality of the oil raised in Galicia varies with the strata containing it.—*Ibid.*

#### BAKU PETROLEUM TRADE.

Official statistics published by the Russian Ministry of Finance show, says the *Levant Herald* for the 20th June last, that in the first quarter of this year the total shipments abroad of petroleum and its products derived therefrom, from the Baku district, amounted to 12,733,432 poods, against 9,512,517 poods during the corresponding period of 1889, the increase being, therefore, 3,220,885 poods, or over 30 per cent. In these totals refined petroleum figured for 9,413,281 poods in 1890, and 7,917,268 poods in 1889, or an increase of 1,496,013 poods, being about 20 per cent. During the same time 17,431,476 poods of petroleum of all kinds, 2,018,653 poods refined, and 14,041,242 poods residual products were despatched to European Russian markets; whereas in the same period last year this movement amounted only to 7,617,073 poods of which 980,867 poods refined petroleum, and 6,106,108 poods residual products. To the Transcaucasus 773,754 poods of petroleum of all kinds were also sent, against 477,453 poods in the first quarter of 1889, residual products accounting for 758,206 poods this year, and 470,104 poods last year. The quantity of naphtha extracted and supplied to the factories during the quarter reached 52,744,124 poods, compared with 34,985,713 poods in 1889, the difference in favour of the present year being 17,758,411 poods, or more than 50 per cent.—*Ibid.*

#### PERUVIAN PETROLEUM.

The *Journal de la Chambre de Commerce de Constantinople* for the 14th June says that the discovery of very important petroleum wells in the north of Peru, in the region comprised between Payla and Tumbes (Department of Pima) will be the means of creating a new source of prosperity for Peru. Already for several years past an Italian, M. Paggio, has been working four wells at Zorritos, near Tumbes, but the petroleum was of an inferior quality,

and the refined petroleum was only sent to Lima in small quantities. An American, M. Tivfield, already well known by his works at Baku and on the Red Sea, purchased a few months ago vast deposits situated near Tatará (to the south of Tumbes). Owing to the large amount of capital at his disposal, the workings have assumed considerable importance. Eight wells are at present opened, and the supply of petroleum would appear to be inexhaustible. A factory for refining the petroleum, and another for manufacturing the tin boxes, have been erected.

A joint stock company, with a capital of 200,000*l.*, has just been formed for the working of the Talará deposits, and those which may be discovered in the future. M. Tivfield has ordered from the United States six fast steamers containing tanks for distributing the petroleum on the Pacific coast, and even sending it to Europe. Besides kerosene, the Talará company also exports unrefined petroleum, which is used for the heating of locomotives. The wells have the advantage of being situated near to the port, which is an excellent one.—*Ibid.*

#### DISCOVERY OF MINERAL OIL IN INDIA.

The *Calcutta Englishman* (overland mail) for the 13th May has the following:—

"A discovery of mineral oil has recently been made in the Suliman hills in the district occupied by the Tser Khoyl tribe of Shiranis. The story of its discovery is decidedly interesting. About five years ago, it appears, the Rev. W. Thwaites, a missionary at Dera Ismail Khan, had in his employment a Shirani chowkedar. On one occasion when the regular house servant was away the boy undertook to trim the wall lamps for night use. The smell of the oil struck him as familiar, and he asked what oil was put into these lamps as its smell was exactly like that of an oil that issued from the ground in his own country. He was accordingly told to get some, and in the course of a few days some oil was brought from the hills by one of his relatives. On being burned in a lamp it gave a good light, though it was somewhat darker-tinted than the ordinary kerosene procurable in the bazaar and gave off more smoke. In the pass from whence the oil was brought there is a stream of water running between two hills, and from small springs in the ground on one side the oil issues and is collected in small holes dug in the ground. The Shiranis use it for outward application to sores on animals, and among them it goes by the name of Water of Shkormá. Attention was drawn to the fact of the existence of this oil during the Viceroy's late visit to the Gualal, and its quality has since been declared by an expert to be very good. As regards the practicability of working the oil on any large scale nothing can be said until a further examination of the locality has been made."—*Ibid.*

#### THE USE OF KEROSENE OIL IN INDIA.

A striking illustration of the increasing use of kerosene oil as an illuminant among the natives is to be found in the returns of the rail-borne trade of Bengal for the last quarter of last year. It is there shown, says the *Pioneer Mail*, that whereas in December 1887 only 46,000 maunds of the oil were transported from Calcutta up-country by rail, in the corresponding month last year the amount was no less than 131,000 maunds. The chief obstacle in the way of the extended consumption of the oil has hitherto been the difficulty of carriage, as it is an article which cannot be easily conveyed without loss over rough roads and paths into country districts; but the figures given show that these obstacles are being gradually surmounted. Patna has become a great centre of distribution, principally by means of country boats, and similar centres will in time be established elsewhere.—*Ibid.*

#### A NEW COCOA-NUT PRODUCT.

The *Ceylon Observer* for the 10th April, quoting from the *Ceylon Advertiser*, says:—

Paragraphs have been going the round concerning a new manufactured product of the cocoa-nut, so much the

reverse of correct, and so calculated to leave a wrong impression as to the future development of the new industry, that it may be well to correct these errors.

The product to which we refer is what has been termed "cocoa nut butter," a name which is incorrect, and is probably accountable for much of the ill-repute into which it has fallen. Being regarded as a "butter," and produced at about half the market price of ordinary butter, those who have written on the subject raise a cry condemning it as a new adulterant of the dairy produce, and a rival of margarine. In the first place, it is not a butter in any sense of the word, and is not at all likely to be employed as an adulterant by reason of its peculiar flavour and colour. It is, in fact, a vegetable lard, and as such it is intended to be used, and is so employed in German kitchens in all cooking processes.

We think it necessary to remove the wrong impression created by allusion to this new article as an "adulterant," because we believe it will build up a future trade in the eastern and western worlds. The natives of India use a large quantity of fat, under the name of "ghee," answering in colour and quality to our "lard." It is the product of buffalo milk, and, inasmuch as Hindoos will not use any animal fat and the Mahomedans avoid the use of hog's lard, this ghee is the only article in use throughout India for culinary purposes, and being in such large demand it is

by no means cheap when purchased in the bazaars. Now, in the newly introduced cocoa-nut fat, misnamed butter, there is at once a better and a cheaper article for Hindoo and Mussulman use. Being a purely vegetable product, it is commended to the scrupulous Hindoo and the prejudiced Mahomedan, and cannot fail to come into extensive use throughout India. If it can be produced by German chemists at Mannheim at half the cost of butter, after incurring the charges on importing the bulky raw material from the East, how much more can it be manufactured in localities where the cocoa-nut is produced.—*Ibid.*

#### MINERAL WEALTH OF AUSTRALASIA.

The following table gives, in a succinct form, the total value of the principal minerals raised in Australasia from their first discovery to the end of 1888, and it will present to the reader a fair idea of the mineral wealth of these colonies. With the exception of coal the development of other minerals in Australia was the outcome of the discovery of gold. The figures given below would, therefore, practically represent the mineral production of Australasia from 1852 to 1888 inclusive, if the value of the coal extracted up to the end of the year 1851 is deducted:—

Colony.	Gold.	Silver and Silver Ore.	Copper.	Tin.	Coal.	Shale.	Total.
	£	£	£	£	£	£	£
New South Wales .....	37,189,817	2,948,754	5,438,262	8,510,372	21,154,307	1,156,782	76,389,294
Victoria .....	222,543,836	87,618	191,022	665,710	25,278	..	223,543,464
Queensland .....	21,319,747	358,572	1,655,894	5,438,509	903,941	..	22,967,083
South Australia .....	891,612	2,800	19,186,163	1,057	..	..	20,081,632
Western Australia .....	219,492	320,381	544,695	..	..	..	1,054,478
Tasmania .....	2,031,520	800	..	4,366,483	179,659	..	6,578,455
New Zealand .....	44,843,642	124,792	17,862	..	2,698,480	..	47,653,776
Australasia .....	329,021,666	3,843,717	27,093,718	18,682,541	24,936,736	1,156,782	444,639,180

It is difficult to obtain much information with regard to other minerals raised in most of the colonies, but if to the total just given be added some 405,167*l.* worth of sundry minerals produced in New South Wales, and 4,857,866*l.*, nearly the whole of which represents the total value of Kauri gum extracted in New Zealand up to the end of 1888, the grand total will be raised to 409,902,213*l.* Deducting therefrom the sum of 279,923*l.*, which represents the value of coal raised in New South Wales prior to the year 1852, the difference, 409,622,290*l.*, practically shows the total value of the mineral wealth produced by the Australasian colonies from 1852 to 1888 inclusive; and during a period of 37 years the average yearly production amounted to 11,070,873*l.*

In the year 1888 the total value of minerals raised in each colony and in Australasia was as follows:—New South Wales, 3,856,187*l.*; Victoria, 2,503,925*l.*; Queensland, 2,066,979*l.*; South Australia, 369,014*l.*; Western Australia, 206,847*l.*; Tasmania, 520,878*l.*; and New Zealand, 1,531,614*l.* Total for Australasia, 11,055,444*l.*—*Ibid.*

Articles of interest on "The Cultivation of Cochineal in the Canaries" and on "Compressed or Tablet Tea" will be found on pages 90 and 96 respectively of the *Board of Trade Journal* for July.

#### THE NEW FERTILISER LAW.

The following Act relating to the fertiliser trade in the State of New York passed the Legislature of this State at its recent session, and in order to give information to all interested in the business, the New York Fertiliser and Chemical Exchange decided to publish a copy of the Bill for gratuitous circulation.

SECT. 1. All commercial fertilisers which shall be offered for sale to be used in this State shall be accompanied by an analysis stating the percentages contained therein of nitrogen or its equivalent of ammonia, of soluble and available phosphoric acid, the available phosphoric acid either to be soluble in water or in a neutral solution of citrate of ammonia as determined by the methods agreed upon by the American Society of Agricultural Chemists, and of potash soluble in distilled water. A legible statement of the analysis of the goods shall be printed on or attached to each package of fertilisers offered for sale for use in this State; and where fertilisers are sold in bulk to be used in this State, an analysis shall accompany the same, with an affidavit that it is a true representation of the contents of the article or articles.

SECT. 2. Manufacturers residing in this State and agents or sellers of fertilisers made by persons residing outside the limits of this State shall, between the 1st and 20th days of July in each year, furnish to the Director of the New York State agricultural experiment station at Geneva a list of the commercial fertilisers they manufacture or offer for sale for use in this State, with the names or brands by which they are known on the market, and the several percentages of nitrogen or its equivalent of ammonia, of phosphoric acid soluble and available, and of potash, either single or combined, contained in said fertiliser, as called for in section 1 of this Act. Whenever any fertiliser or fertilising ingredients are shipped or sold in bulk for use by farmers in this State, a statement must be sent to the Director of the New York State agricultural experiment station at Geneva, giving the name of the goods so shipped, and accompanied with an affidavit from the seller, giving the analysis of such percentage guaranteed.

SECT. 3. Whenever a correct chemical analysis of any fertiliser offered for sale in this State shall show a deficiency of not more than one-third of 1 per cent. of nitrogen or its equivalent of phosphoric acid, or one-half of 1 per cent. of ammonia soluble or available, and one-half of 1 per cent. of potash soluble in distilled water, such statements shall not be deemed false within the meaning of this Act. This Act shall apply to all articles of fertilisers offered or exposed for sale for use in the State of New York, the selling price of which is 10 dols. per ton or higher, and of which they are part or parcel, and of any element into which they enter as fertilising material, among which may be enumerated nitrate of soda, sulphate of ammonia, dissolved bone black and bone black undissolved, any phosphate rock, treated or untreated with sulphuric or other acids, ashes from whatever source obtained, potash salts of all kinds, fish scrap, dried or undried, also all combinations of phosphoric acid, nitrogen, or potash, from whatever source obtained, as well as all and every article that is or may be combined for fertilising purposes.

SECT. 4. All manufacturers or dealers exposing or offering for sale in this State fertilisers containing roasted leather or any other form of inert nitrogenous matter, shall, in legible print, state the fact on the packages in which the fertilisers are offered or exposed for sale.

SECT. 5. Every person, firm, or corporation violating any of the provisions of this Act shall, upon conviction thereof, for the first offence be punished by a fine not less than 50 dols. and not more than 200 dols., and for the second offence by double the amount in the discretion of the court, such fines to be paid to the officer whose duty it is to enforce the provisions of this Act, to be used by him for that purpose, and to be accounted for to the Comptroller.

SECT. 6. The Director of the New York State agricultural experiment station at Geneva is charged with the enforcement of the provisions of this Act, and shall prosecute in the name of the people, for violations thereof; and for that purpose he may employ agents, counsel, chemists, and experts, and the court of special sessions shall have concurrent jurisdiction to hear and determine charges for violating the provisions of this Act committed in their respective counties, subject to the power of removal provided in chapter 1 of title 6, of the code of criminal procedure.

SECT. 7. And the said Director of the New York State agricultural experiment station at Geneva, or his duly authorised agents shall have full access, egress and ingress to all places of business, factories, buildings, cars, vessels, or other places where any manufactured fertiliser is sold, offered for sale, or manufactured. Such Director shall also have power to open any package, barrel, or other thing containing manufactured fertiliser, and may take therefrom sufficient samples; and whenever any such fertiliser is so taken for samples, it may be divided into different portions, and one or more portions sealed in such a way that it cannot be opened without upon examination giving evidence of having been opened to the person sealing the same, and delivered to the person from whom said sample is taken, or any other person that may be agreed upon by the said Director or his agents who takes the same, and the person from whom it is taken, which portion so delivered may, upon consent of the parties, be delivered to a chemist for the purpose of being analysed other than the chemist employed by said Director.

SECT. 8. The sum of 20,000 dols., or so much thereof as may be necessary, is hereby appropriated out of any money in the Treasury not otherwise appropriated, to be used by said Director of the New York State agricultural experiment station at Geneva, as shall be authorised by the board of control thereof, in enforcing the provision of this Act. Said sum shall be paid to said Director by the Treasurer upon the warrant of the Comptroller, upon vouchers to be approved by the Comptroller, in such sums and at such times as said Director may require, who shall file a statement for what purposes he desire the same.

SECT. 9. Agents, representatives, or sellers of manufactured fertiliser, or fertilising material made or owned by parties

outside of this State, and offered for sale for use in this State, shall conform to the provisions of this Act, and shall be subject to its penalties, and in all particulars shall take the place of their non-resident principal.

SECT. 10. Chapter 222 of the laws of 1878 is hereby repealed.

SECT. 11. This Act shall take effect immediately.—*Chem. Trade Journ. from Oil Paint and Drug Reporter.*

#### THE GERMAN DRUG TRADE.

The German commercial statistics for 1889 have just been published, and show the value of all imports into the country to have been 4,087 millions of marks, that of the exports being 3,256 millions. Compared with the previous year, these figures show that the imports have increased by 6.51 million marks, or nearly 16 per cent., while the exports have diminished by 3 per cent. (96 million marks). Drugs and chemicals show a very marked increase, both in imports and exports, on the preceding year, the following being the values, in thousands of marks:—

	Imports.		Exports.	
	1889.	1888.	1889.	1888.
Raw materials for chemical industry	74,819	65,241	8,356	7,707
Drugs for medicine and perfumery..	17,946	15,594	6,845	5,760
Raw dyestuffs .....	13,998	13,299	5,439	5,111
Simple chemicals, bases, acids, and salts.....	25,805	27,715	63,569	60,328
Ether, essential oils, medicaments, and perfumeries .....	10,385	9,913	20,737	27,241
Paints, varnishes, lacquers, and glues	10,875	9,717	9,632	9,505
Pitch, tar, and tar distillates .....	19,152	16,713	10,487	8,910

The largest increase in value among the imports is shown by coal-tar oils, carbolic and picric acids, carbonate of ammonia, camphor and dyewood extracts, and under exports by soda and potash salts, albumen, carbolic acid. The principal decreases are, in exports—cinchona salts and quinine preparations, glycerin, iodides, salicylates, tartar materials, aniline dyes, and other coal-tar dyes; in the imports—bichromates of soda and potash, gums, cinchona, indigo, iodine, and phosphorus. Camphor was imported to the extent of 1,414,000 marks, while the exports were 921,000 marks. The values of the exports of some of the principal medicinal preparations shipped from Germany last year were:—

	Marks.
Salicylates.....	2,613,000
Iodine.....	315,000
Iodides.....	2,367,000
Alcoholic tinctures .....	624,000
Essential oils.....	1,050,000
Cinchona salts and preparations.....	6,864,000
Alkaloids and their salts.....	3,720,000
Toilet soaps .....	2,030,000

—*Chemist and Druggist.*

#### EXPLOSION IN A PICRIC ACID WORKS.

About midday on June 27th a terrific explosion wrecked the greater part of the works of the Chemische Fabrik Rhein in Germany. The cause of the disaster was a fire, which had originated in the drying-rooms of the picric-acid

department, and exploded part of the stock of that material. The picric-acid department itself is completely destroyed, but a store of 60 tons of picric acid kept at another part of the works was saved. No lives were lost, and the acid manufacture will be taken up again as soon as possible in another part of the factory.—*Ibid.*

#### "BENZOL" MANUFACTURE IN BAKU.

The Rothschilds are building works in Baku (Russia) for the manufacture of benzol from naphtha residue. The experiments have shown that there is every probability that this industry will be successful. Schibajew's oilworks in Baku have been manufacturing both benzene and anthracene for many years.—*Ibid.*

#### THE ALKALI TRADE.

The efforts which are being made to syndicate the Leblanc alkali works in England, and the probability of the union including, within a short time, the ammonia soda works, have raised hopes in Germany of increasing the export trade. A considerably enhanced cost of British alkali will not only help German competition in foreign neutral markets, but will also stimulate the trade with Great Britain. The quantity of soda now annually shipped to England amounts to about 16,000 cwt., that of potash being about 28,000 cwt. These are not large quantities; but a small addition to the cost of English-made alkali would enable the German manufacturers to increase their exports very greatly.—*Industries.*

#### MINERAL PRODUCE OF THE UNITED KINGDOM.

Description of Mineral Raised.	1888.		1889.	
	Quantity.	Value at Mines.	Quantity.	Value at Mines.
	Tons.	£	Tons.	£
Alum clay (bauxite).....	9,666	4,893	9,150	5,490
Alum shale.....	1,384	248	4,188	520
Antimony ore.....	7½*	7	67*	900
Arsenic.....	1,624	35,497	4,758	38,260
Arsenical pyrites.....	5,325	1,210	7,688	7,310
Barytes.....	25,191	26,117	24,849	28,230
Bor ore.....	10,927	5,463	14,002	7,000
Clays (excepting ordinary clay).....	2,562,792	653,419	3,036,253	828,170
Coal.....	160,395,219	12,971,276	176,916,724	56,175,120
Cobalt and nickel ore.....	152	746	155	960
Copper ore.....	15,132½	60,980	9,029	26,580
Copper precipitate.....	418	6,559	281	3,110
Fluorspar.....	140	153	297	410
Gold ore.....	3,844	27,300	6,226	10,740
Gypsum.....	130,082	58,998	132,357	53,810
Iron ore.....	11,590,713	3,591,317	11,516,165	3,848,260
Iron pyrites.....	23,507	11,392	17,719	8,110
Jet.....	2,217½	332	618*	120
Lead ore.....	51,259	438,383	48,465	429,640
Lignite.....	971	437	947	280
Manganese ore.....	4,812	1,034	8,852	6,470
Ochre,umber, &c.....	7,573	13,387	10,194	15,530
Oil shale.....	2,076,469	519,126	2,014,860	503,710
Petroleum.....	35		30	40
Phosphate of lime.....	22,500	43,312	20,000	38,200
Salt.....	2,305,569	700,829	1,946,496	800,700
Slates and slabs.....	471,788	1,057,535	458,436	1,048,100
Stone, &c.....	....	8,624,697	....	8,670,900
Sulphate of strontia.....	7,064	3,532	5,576	2,900
Tin ore.....	14,370	891,665	13,809	729,200
Tungstate of soda.....	2½	54	....	....
Wolfram.....	60	1,025	½	....
Zinc ore.....	26,408	95,984	23,202	96,900
Total values.....	....	59,834,997	....	73,176,000

\* Cwt.

† Lb.

—Ironmonger.

## METALS OBTAINABLE FROM ORES PRODUCED IN THE UNITED KINGDOM.

Description of Metal.		1888. Metals obtainable by Smelting.		1889. Metals obtainable by Smelting.	
		Quantity.	Value at the Average Market Price.	Quantity.	Value at the Average Market Price.
Aluminium.....	Lb.	5,000	£ 5,000	12,000	£ 6,000
Antimony.....	Cwt.	3½	18	666	1,998
Copper.....	Tons	1,156	115,849	905	49,948
Gold.....	Oz.	8,745	29,582	3,800	13,227
Iron.....	Tons	5,120,861	9,492,092	5,181,733	12,595,246
Lead.....	"	37,578	322,804	35,004	164,342
Magnesium.....	—	....	1,000	....	....
Silver.....	Oz.	321,125	57,121	305,149	54,453
Sodium.....	Lb.	20,040	6,000	63,726	9,445
Tin.....	Tons.	9,241	1,083,700	8,912	869,342
Zinc.....	"	10,002	191,155	9,392	192,145
Total values.....		....	11,595,321	....	14,346,846

—*Ibid.*

## COPPER.

We have again to report an enormous delivery to consumers out of stores. The total so stated is 18,781 tons, making, with the 17,030 tons in May, a delivery not only unexampled in the trade, but nearly 9,000 tons in excess of any two previous consecutive months. Time must show whether any portion of this is due to consumers stocking in their own warehouses in anticipation of their future requirements, or whether it is a purely natural flow of business.

Meantime it has reduced the stocks of copper in warehouse in this country to 38,521 tons, a quantity so small that traders here are justified, and, in fact, bound to exercise unusual caution in all their dealings for forward delivery. The market has had an upward tendency with a large daily turnover on change. The highest figure for G. M. B. being 59*l.* 10*s.*, and the lowest 57*l.* closing firm at 58*l.* 2*s.* 6*d.*

## STOCKS AND QUANTITIES AFOAT FOR ENGLAND AND FRANCE.

	1890, May 31.	1890, June 30.	1889, June 30.	1888, June 30.	1887, June 30.	1886, June 30.	1885, June 30.
Liverpool and Swansea stocks of Chili bars, ores, and regulus, reduced to the standard of fine copper.....	Tons. 16,744	Tons. 16,406	Tons. 28,259	Tons. 33,692	Tons. 37,493	Tons. 48,506	Tons. 30,249
Other furnace material, same ports.....	21,057	17,161	32,538	11,937	3,093	4,761	4,783
Continental stocks and afloat.....	34,758	30,969	41,321	15,979	1,589	1,203	2,561
London stock, chiefly Australian.....	5,535	4,957	6,860	4,255	3,747	7,030	5,496
Chilian produce afloat, per mail and wire advices.	3,850	2,304	1,900	500	5,100	9,200	9,711
Australian do. per mail and wire advices.....	500	700	550	1,200	1,250	850	1,337
Total tons.....	82,044	72,493	111,689	72,443	51,972	61,550	54,137
Price of Chili bars per ton.....	54 <i>l.</i> 10 <i>s.</i>	58 <i>l.</i> 10 <i>s.</i>	41 <i>l.</i> 10 <i>s.</i>	81 <i>l.</i> 5 <i>s.</i>	40 <i>l.</i>	39 <i>l.</i> 12 <i>s.</i> 6 <i>d.</i>	44 <i>l.</i> 10 <i>s.</i>

The quantity chartered at West Coast during June was 1,300 tons.

From January 1 to June 30 in the respective years mentioned below it was :—

	1890.	1889.	1888.	1887.	1886.	1885.	1884.
Estimated fine copper in round numbers.....	Tons. 11,950	Tons. 11,800	Tons. 15,200	Tons. 11,500	Tons. 18,600	Tons. 19,350	Tons. 21,100

## DELIVERIES.

	1890.	1889.	1888.	1887.
	Tons.	Tons.	Tons.	Tons.
From England and France, January to June .....	76,087	42,551	33,163	50,435

## SUPPLIES TO ENGLAND AND FRANCE.

	1890.	1889.	1888.	1887.
From North American sources.....	13,400	12,253	18,306	7,096
Other sources.....	39,080	39,522	44,500	32,021
January to June, Total tons .....	52,480	51,785	63,406	39,117

—W. T. Sargant and Sons.

## QUICKSILVER.

In the early part of June importers made sales at 10*l.* 5*s.*, and raised their price to 10*l.* 10*s.*, but as the demand has been small the outside market has not followed, and we quote 10*l.* 2*s.* 6*d.* sellers, as nearest rate to day.

The receipts of Californian at San Francisco during June were 885 bottles, making from January 1 to June 30 as follows:—

1890.	1889.	1888.	1887.	1886.	1885.
Bottles.	Bottles.	Bottles.	Bottles.	Bottles.	Bottles.
6,139	7,300	17,234	10,186	9,765	12,220

## IMPORTATIONS INTO LONDON FOR THE SEVEN MONTHS OF EACH SEASON TO JUNE 30.

	1890.	1889.	1888.	1887.	1886.
Government Spanish .....	50,242	69,334	52,013	50,853	44,015
Other Spanish .....	..	254	1,145	156	346
Italian.....	8,200	5,700	6,160	3,900	5,196
Austrian .....	600	1,300	1,694	1,100	1,800
Californian, Borneo, and indirect import .....	50	545	3,800	..	..
Total bottles.....	59,092	57,103	64,842	56,009	51,357

## EXPORTS FROM LONDON FOR THE SEVEN MONTHS OF EACH SEASON TO JUNE 30.

	1890.	1889.	1888.	1887.	1886.
Bottles .....	37,824	36,289	30,097	35,791	43,820

—*Ibid.*

## CERTIFICATES OF ORIGIN IN FOREIGN COUNTRIES.

The following summary of replies received from Her Majesty's Representatives in the principal commercial countries respecting Customs requirements in the matter of certificates of origin has been received at the Board of Trade from the Foreign Office:—

*Belgium.*—Only required in exceptional cases, such as for animals imported from infected countries, vines, &c.

*Brazil.*—None required.

*Bulgaria.*—Certificates required; those issued by Chambers of Commerce will be accepted, and they should be in the French language.

*Denmark.*—None required.

*Ecuador.*—None required.

*France.*—Required for some articles, such as live plants and meat. Certificates issued by Chambers of Commerce would not be accepted.

*Germany.*—Only required for pork (including bacon and sausages).

*Mexico.*—None required.

*Netherlands.*—Only required for articles the importation of which is prohibited, but which may be admitted by exception on certain conditions.

*Norway and Sweden.*—None required.

*Portugal.*—None required.

*Romania.*—Certificates required; those issued by Chambers of Commerce in their corporate capacity will be accepted.

*Russia.*—None required.

*Serbia.*—Certificates required; those issued by a Chamber of Commerce at the port of despatch will be accepted. They must be attested on arrival at Belgrade by the British consular or diplomatic representative.

*Spain.*—Certificates required; they are to be signed before mayors, Chambers of Commerce, police authorities, or public notaries, and must be visé before the Spanish Consul.

*Switzerland.*—None required.

*United States of America.*—None required.

*Uruguay.*—None required.

—Board of Trade Journal.

## THE CONDITION OF GERMAN CHEMICAL INDUSTRY SINCE 1881.

From the Report to the Bureau des Deutschen Handelstages by Consul Annacke. Chem. Ind. **13**, 159—162.

*Hydrochloric acid* and *bleaching powder* have both been the subjects of a great deal of fluctuation since 1881. The high price of bleach in England, maintained by the Bleach Association, had a favourable effect on the German market, the Leblanc soda manufacturers being enabled in some measure to recoup themselves for the losses incurred on their alkali. In 1881 the demand for salts exceeded the supply, the price in consequence rising 10 per cent. Further advances were effected in 1886 and 1887, hydrochloric acid thus attaining a price hitherto unprecedented, certain qualities in particular, notably that free from iron, being sold at specially good rates. Bleach towards the beginning of this period was in great request, the more so as an outbreak of cholera occurred in South France. Later, the manufacturers, by utilising to a fuller extent their existing works and erecting fresh plant, were able to supply in full the home demand, the deficiency of about 50,000 double-ctr. being previously imported. (The editor of Chem. Ind. adds that Germany will shortly export bleaching materials, as bleaching powder is being made from the Stassfurt magnesium chloride; and chloride of lime, made electrolytically from potassium chloride, is already in the market.)

The manufacture of *chlorate of potash* has long been unprofitable in Germany, as the production of hydrochloric acid is only sufficient for use as such and for manufacturing bleach, whilst in England there is an excessive over-production. Last year the English make chiefly was imported.

Towards the end of 1883 a syndicate of Stassfurt firms was formed for the sale of *potash salts* through one channel. Good business was done for some time, but in 1888 the price had fallen so much that scarcely 100 M. per 100 kilos. could be realised.

The manufacturers of *oxalic acid* and *salts of sorrel* were in a prosperous position at the beginning of the last decade. Then came a period of adversity. America, in consequence of bad trade, reduced its import, the result being that the English output, previously relieved by export to America, was thrown on the European market. This exportation to Germany annually increased although prices continued to fall. In 1888, however, a convention was arranged between the English and German manufacturers of the acid, and fairer prices were obtained. The existence of the convention has been prolonged owing to the English manufacturers recognising the fact that their German competitors can produce this article at a lower cost. (The Editor of Chem. Ind. contradicts the last statement, adding, that several German firms have latterly been compelled to discontinue the manufacture.)

Great changes have taken place since 1884 in the *tartar* trade. Formerly this article was imported into Germany from France, Italy, and Austria. Since 1884, however, the supply from France has entirely ceased, and that from the other two countries has greatly diminished. The Austrian tartar would have been replaced by the superior quality received through London from the Cape, if the American market had not forced up the price of the same. As it was, the Italian manufacturers, being excluded from France by the increased import duty on Italian tartar, sought a market in Germany, and by underselling their Austrian rivals were enabled to obtain a monopoly of the trade.

The condition of the *tartaric acid* trade has not been satisfactory. Germany buys the raw material for the manufacture of the acid from abroad, and exports 90 per cent. of its manufacture, so that the increased foreign duties and larger production in England led to a glut in the market. To remedy this state of things a convention was formed in 1884 and a diminished output resolved upon. Two years later English speculators bought up the German stocks and raised the price, an over-production on the part of the manufacturers again ensuing, which greatly injured the trade of the following year. After 1888, prices were reduced, the English price falling 10—20 per cent., and the Austrians selling in Germany at 10—15 per cent. less than in their own country.

The consumption of *acetic acid* constantly increases, larger quantities being employed both in the textile industries and for vinegar manufacturing. Unfortunately the German export trade is hampered with duties imposed by Italy and Russia.

The quality of *citric acid* made in Germany is so excellent that almost the whole consumption is supplied by this country.

*Borax*, formerly a product of chiefly English manufacture, is now largely sold by the Hamburg factories.

In 1881—83 the enormous quantities of double distilled *glycerin*, consumed by the American dynamite manufacturers, led to an increase of 100 per cent. in the price. In 1884, however, the price had re-assumed its normal level, and, although an American syndicate bought one million kilos., the market remained unaffected. The American manufacturers in 1886 began to buy crude glycerin, and the price speedily rose 140 per cent., but fell again two years later, and only partially recovered in the following year.

The *charcoal-manufacturing* and *wood-distillation* industries have not been in the flourishing condition that the natural advantages in the shape of abundant forests, cheap transport and fuel, and low rates of wages, should warrant. The export trade has decreased in consequence of the higher duties levied abroad.

*Wood-tar* has fallen considerably in price since 1884 through the importation of the same from Russia and Austria.

The stocks of *coal-tar* continued to increase until 1888, but began to diminish again towards 1889, and the price at the present time is more profitable.

Better business has been done in *wood-pitch*. Although the exportation to Austria has been cut off, the home market has not been affected thereby, and the price has even slightly advanced.—E. B.

## BOARD OF TRADE RETURNS.

### SUMMARY OF IMPORTS.

	Month ended 30th June	
	1889.	1890.
	£	£
Metals.....	1,450,566	2,031,964
Chemicals and dyestuffs.....	567,433	642,632
Oils.....	483,526	603,721
Raw materials for non-textile industries.....	3,422,480	3,603,421
Total value of all imports ....	29,326,994	32,926,295

### SUMMARY OF EXPORTS.

	Month ended 30th June	
	1889.	1890.
	£	£
Metals (other than machinery) ....	3,193,849	3,775,311
Chemicals and medicines .....	641,174	732,523
Miscellaneous articles.....	2,501,830	2,718,070
Total value of all exports.....	13,696,717	21,532,817

### IMPORTS OF METALS FOR MONTH ENDED 30TH JUNE.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
			£	£
Copper:—				
Ore ..... Tons	8,213	16,130	54,341	95,798
Regulus ..... "	3,777	11,031	98,193	315,993
Unwrought .... "	1,991	4,013	87,886	223,177
Iron and steel:—				
Iron ore ..... "	301,221	406,446	215,729	323,697
Iron bolt, bar, &c. "	9,551	7,799	86,239	69,387
Steel, unwrought.. "	1,521	746	11,923	9,126
Lead, pig and sheet ..	9,917	13,375	127,792	173,871
Pyrites ..... "	59,738	58,060	111,361	109,383
Quicksilver ..... Lb.	1,111,915	132,667	133,501	57,716
Tin ..... Cwt.	41,319	38,066	199,852	178,465
Zinc ..... Tons	3,545	3,144	62,055	69,949
Other articles ...Value £	..	..	270,634	405,160
Total value of metals	..	..	1,450,566	2,031,964



IMPORTS OF CHEMICALS AND DYESTUFFS FOR MONTH  
ENDED 30TH JUNE.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Alkali..... Cwt.	3,899	4,006	£ 3,497	£ 3,667
Bark (for tanners, &c.) .....	77,173	93,131	40,460	46,574
Brimstone .....	96,541	64,494	19,350	14,136
Chemicals..... Value £	..	..	110,253	132,261
Cochineal .....	384	700	2,293	4,368
Cutch and gambier Tons	3,417	2,033	88,673	55,946
Dyes:—				
Anilino .....	..	..	24,217	18,671
Alizarine .....	..	..	23,175	25,923
Other .....	..	..	795	2,443
Indigo .....	3,024	1,976	40,484	26,308
Madder .....	922	728	1,050	903
Nitrate of soda....	31,599	111,527	13,186	45,728
Nitrate of potash .	7,586	26,111	7,148	24,253
Valonia .....	4,270	2,622	60,472	52,579
Other articles... Value £	..	..	132,380	188,873
Total value of chemicals	..	..	567,433	642,632

## IMPORTS OF OILS FOR MONTH ENDED 30TH JUNE.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Cocoa-nut..... Cwt.	3,461	11,376	£ 4,823	£ 15,775
Olive .....	2,404	4,132	84,536	160,010
Palm .....	79,957	73,349	79,789	77,828
Petroleum .....	7,237,349	8,645,981	165,947	181,514
Seed .....	1,373	1,440	34,492	37,298
Train, &c..... Tons	2,035	3,373	42,027	61,702
Turpentine .....	12,252	35,757	19,860	54,399
Other articles .. Value £	..	..	52,052	81,195
Total value of oils ...	..	..	483,526	669,721

IMPORTS OF RAW MATERIALS FOR NON-TEXTILE  
INDUSTRIES FOR MONTH ENDED 30TH JUNE.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Bark, Peruvian .. Cwt.	8,770	10,737	£ 29,590	£ 32,186
Bristles..... Lb.	353,297	234,341	55,869	36,610
Caoutchouc..... Cwt.	9,876	14,529	99,266	166,846

## IMPORTS OF RAW MATERIALS, &amp;c.—cont.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Gum:—			£	£
Arabic..... Cwt.	3,812	6,006	14,155	16,704
Lac, &c..... "	2,942	4,454	10,063	19,067
Gutta-percha ....	2,928	3,134	27,580	26,909
Hides, raw:—				
Dry..... "	39,777	34,095	118,385	84,765
Wet .....	45,126	62,079	104,632	142,240
Ivory .....	384	1,771	17,705	90,413
Manures:—				
Guano .....	1,629	5,257	18,883	31,830
Bones..... "	3,235	4,519	13,833	23,495
Paraffin..... Cwt.	22,790	35,206	27,219	43,024
Linon rags..... Tons	4,025	3,753	35,717	33,812
Esparto, &c. ....	17,710	19,543	84,526	89,278
Pulp of wood ....	9,355	10,806	51,992	61,369
Rosin..... Cwt.	89,672	216,657	22,890	50,000
Tallow and stearin "	123,488	161,122	165,764	191,306
Tar .....	2,689	6,563	1,743	6,596
Wood:—				
Hewn .....	179,733	199,229	373,464	433,100
Sawn .....	563,698	500,913	1,388,830	1,182,411
Staves .....	11,818	18,981	46,036	70,553
Mahogany .....	1,118	2,177	11,428	18,648
Other articles... Value £	..	..	702,946	812,462
Total value .....	..	..	3,422,480	3,663,424

Besides the above, drugs to the value of 68,704*l.* were imported as against 53,578*l.* in 1889.

EXPORTS OF METALS (OTHER THAN MACHINERY) FOR  
MONTH ENDED 30TH JUNE.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Brass..... Cwt.	10,120	9,075	£ 42,393	£ 42,617
Copper:—				
Unwrought .....	92,437	69,720	200,258	199,805
Wrought..... "	27,661	25,117	77,469	84,908
Mixed metal ....	19,683	38,334	47,310	105,845
Hardware .....	..	..	220,799	231,967
Implements..... "	..	..	100,792	105,290
Iron and steel.... Tons	332,297	346,704	2,249,173	2,616,964
Lead .....	3,775	5,276	54,393	74,952
Plated wares... Value £	..	..	30,299	20,623
Telegraph wires... "	..	..	45,189	152,228
Tin .....	7,796	8,547	37,050	41,964
Zinc .....	15,717	18,697	11,160	18,230
Other articles .. Value £	..	..	77,564	80,488
Total value .....	..	..	3,193,849	3,775,311

EXPORTS OF DRUGS AND CHEMICALS FOR MONTH ENDED  
30TH JUNE.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Alkali..... Cwt.	469,984	483,176	123,882	137,348
Bleaching materials ..	119,922	169,430	43,692	30,829
Chemical manures. Tons	22,912	24,788	175,580	162,466
Medicines..... Value £	..	..	72,816	88,229
Other articles ... ..	..	..	225,504	263,656
Total value ..... ..	..	..	641,474	732,528

EXPORTS OF MISCELLANEOUS ARTICLES FOR MONTH  
ENDED 30TH JUNE.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Gunpowder..... Lb.	749,400	839,200	18,224	18,697
Military stores.. Value £	..	..	101,559	86,859
Candles..... Lb.	1,006,900	1,139,900	17,594	22,181
Caoutchouc ..... Value £	..	..	82,375	92,495
Cement..... Tons	47,963	50,954	91,945	104,489
Products of coal Value £	..	..	77,129	87,817
Earthenware ... ..	..	..	150,160	171,286
Stoneware ..... ..	..	..	15,808	16,827
Glass:—				
Plate..... Sq. Ft.	301,428	303,729	19,812	20,542
Flint..... Cwt.	8,929	8,224	19,662	20,285
Bottles..... ..	67,449	70,201	31,508	33,993
Other kinds..... ..	16,973	17,592	12,628	14,841
Leather:—				
Unwrought .... ..	10,916	11,514	93,969	107,385
Wrought ..... Value £	..	..	23,556	23,621
Seed oil..... Tons	5,496	4,782	120,375	115,219
Floor cloth ..... Sq. Yds.	1,171,700	1,298,600	57,554	65,614
Painters' materials Val. £	..	..	131,315	126,372
Paper ..... Cwt.	73,467	86,337	136,086	141,544
Rags..... Tons	4,621	4,429	37,367	33,713
Soap..... Cwt.	41,715	42,763	39,524	45,122
Total value ..... ..	..	..	2,501,830	2,718,979

TRADE BETWEEN SPAIN AND THE  
UNITED KINGDOM.

IMPORTS INTO THE UNITED KINGDOM FROM SPAIN FOR  
THE THREE MONTHS ENDED 30TH JUNE.

Principal Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Chem. products unenumerated Value £	..	..	16,330	13,288
Copper ore and regulus ..... Tons	16,095	19,800	285,313	385,196
Iron ore ..... ..	917,699	1,090,768	656,818	871,503
Lead ore..... ..	141	415	1,470	2,924
Lead, pig and sheet ..... ..	10,889	19,449	213,912	248,255
Pyrites ..... ..	154,739	154,498	289,573	265,290
Quicksilver..... Lb.	2,017,265	1,923,222	224,136	254,013
Rags, esparto ... .. Tons	15,957	14,726	96,343	84,397
Zinc ore ..... ..	351	176	1,688	465
Total value ..... ..	..	..	2,679,995	2,997,549

EXPORTS OF HOME PRODUCE FROM THE UNITED KINGDOM  
TO SPAIN FOR THE THREE MONTHS ENDED 30TH JUNE.

Principal Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Alkali..... Cwt.	71,902	63,159	29,498	26,246
Caoutchouc manufactures .... Value £	..	..	2,485	3,368
Cement ..... Tons	1,758	1,510	3,417	2,400
Chemicals (including dye-stuffs) ..... Value £	..	..	19,095	44,287
Products of coal .. ..	..	..	7,394	12,847
Glass manufactures ..... ..	..	..	2,098	2,436
Grease, tallow, &c. Cwt.	17.2	4,993	1,654	4,709
Manure..... Value £	..	..	51,973	87,053
Oil, seed ..... Tons	73	94	1,191	2,126
.. other sorts.. Value £	..	..	2,739	2,964
Oil and floor cloth ..... Sq. yds.	87,000	127,100	3,511	4,609
Painters' colours &c. .... Value £	..	..	8,516	8,204
Paper of all sorts Cwt.	1,570	1,593	3,702	3,674
Soap..... ..	555	531	737	634
Total value ..... ..	..	..	194,751	1,276,769

## Monthly Patent List.

\*The dates given are the dates of the Official Journals in which acceptances of the Complete Specifications are advertised. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the said dates.

### I.—GENERAL PLANT, APPARATUS, AND MACHINERY.

#### APPLICATIONS.

9285. G. Stevenson. Improvements in apparatus for automatically measuring defined quantities of solid or liquid substances. June 16.

9312. G. Horner. Improvements in surface condensers. Complete Specification. June 16.

9326. J. O. Woods. Improvements in vaporisers. Complete Specification. June 17.

9354. C. H. Huntley. Improvements in recovering saline and other substances from solution. June 17.

9379. W. B. Bull. Improvements in means for purifying liquids for the prevention of steam boiler incrustation, ageing liquors, &c. Complete Specification. June 17.

9416. A. Anderson and J. Wilson. Apparatus for burning liquid fuel. June 18.

9518. J. Foster. Improvements in and relating to apparatus for evaporating solutions containing salts and for other liquids. June 19.

9554. L. H. A. W. Sack. Apparatus for automatically drawing off viscous substances. Complete Specification. June 20.

9604. W. E. Smith. Improvements relating to apparatus for burning liquid fuel. June 20.

9685. G. Cox. Improvements in the manufacture of tin, iron, and other metal drums for carrying oils and other liquids. June 23.

9703. E. F. Edgar. An improved furnace. June 23.

9941. R. Haddan.—From E. Passburg, Germany. An improved method and apparatus for drying aqueous matter. Complete Specification. June 26.

10,116. F. B. Pemberton. An improved composition for preventing or obstructing the passage of heat through bodies, and for the deadening of sounds, and methods of applying the same. June 20.

10,133. P. Abrahamson. Certain improvements in oven and other heating and fire-chamber doors. Complete Specification. July 1.

10,172. H. von Bayer. Improvements in and relating to a method of and apparatus for producing cold, and for freezing liquid and other substances. Complete Specification. July 1.

10,374. J. Gamgee. Improvements in condensing or liquefying vapours or gases, and in apparatus therefor. Filed July 4. Date applied for December 9, 1889, being date of application in Belgium.

10,380. J. Murrie. Improvements in and relating to pyrometers or thermometers. July 4.

10,398. L. P. Cohen and E. Hermann. Improvements in furnaces. Complete Specification. July 5.

10,524. T. Gaunt and E. H. Clapp. Improvements in evaporating apparatus. Complete Specification. July 8.

10,539. E. Holt. Improvements in automatic apparatus for regulating the supply of air to furnaces. July 8.

10,592. M. Honigsmann. Improvements in heating gaseous, liquid, and solid substances. July 8.

10,679. E. Fahrig. A press for impregnating liquids with gases. July 9.

10,721. W. L. Wise.—From Solvay and Co., Belgium. Improved method of, and means for charging materials into apparatus, such as cupolas, blast furnaces, lime kilns, scrubbers, and absorption towers. July 10.

10,724. W. A. Carlyle. An improved compound for manufacturing crucibles or the like vessels or utensils. July 10.

10,788. H. M. Saint Denis. Improvements in centrifugal drying machines or hydro-extractors. July 11.

10,856. A. Silverberg and C. Detering. Improvements in carbonising apparatus. Complete Specification. July 12.

10,888. J. H. R. Diasmore. Improvements in or connected with apparatus for drying granular substances. July 12.

#### COMPLETE SPECIFICATIONS ACCEPTED.\*

1889.

11,925. C. A. Bempen and E. Andre. Valves for regulating the pressure of gas and other fluids. June 25.

12,885. M. Miller. Steam or air superheater. July 16.

13,814. W. Young. Apparatus for evaporating brine, &c., under a vacuum. July 9.

13,816. W. Young. Apparatus for boiling brine, &c., in a vacuum. July 2.

14,416. P. Radcliffe. Furnaces for melting, &c. July 2.

1890.

6864. J. J. Shedlock and J. J. Meldrum. See Class X.

8568. W. P. Thompson.—From J. A. Bowden. Filters. July 16.

8915. O. Guttman. See Class VII.

### II.—FUEL, GAS, AND LIGHT.

#### APPLICATIONS.

9320. T. Travers. Improvements in gas burners, and apparatus for naphthalising gas. June 16.

9331. J. A. Whitby. The magic firelighter. June 17.

9394. J. C. Chandler. Improvements in charging gas retorts, and apparatus therefor. June 17.

9521. J. C. Mewburn.—From O. W. Ketchum, Canada. Improvements in and connected with gas producers, or apparatus for producing gas to be used as fuel. June 19.

9529. C. Herzog. An improved gas generator to be independent of climatic variations. June 19.

9540. C. Herzog. An improved method of and means for the regulation of the quality of an artificial gas. June 19.

9642. G. Horsley and A. C. Wilson. Improvements in or relating to the manufacture of coke. June 21.

9651. W. Loé and C. Gumbart. Improvements in or relating to the treatment of peat for the production of fuel and by-products. June 21.

9733. S. Pitt.—From E. J. Jerzmanowski, United States. Improvements in the manufacture of illuminating gas. Complete Specification. June 23.

9786. L. Hendle and E. Schuberth. An improvement on electric incandescent lighting, called "The non-consuming glow light filament." June 24.

9956. R. Kennedy. Improvements in heating by electricity. June 27.

10,155. J. Ruseoe. Improvements in means or appliances for facilitating the operations of charging and drawing the charges of certain gas retorts. July 1.

10,329. J. Love. Improvements in and relating to apparatus for the manufacture of gas for illuminating and other purposes. July 3.

10,713. W. Hughy. An improved portable apparatus for charging inclined gas retorts. July 10.

10,831. L. Van Vestrant. Improvements in means and apparatus for charging inclined gas retorts. July 12.

10,869. G. F. Priestly. Automatic machinery for charging and discharging gas retorts. July 12.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

10,660. W. P. Thompson.—From J. Wilson and A. Mason. Appliances for burning hydrocarbons. July 2.

10,661. W. P. Thompson.—From J. Wilson and A. Mason. Process for burning petroleum and other hydrocarbons, and apparatus therefor. July 2.

11,459. J. Dheyne, Comte de Nydprick, and F. de la Hault. Apparatus for generating gas or vapour from combustible liquids. July 9.

11,855. J. Morris. Artificial fuel. July 2.

12,043. J. F. L. Monier. Manufacture of gas from atmospheric air. July 2.

12,655. J. Watson. Heating apparatus. July 16.

13,653. W. P. Thompson.—From P. Marix and M. Notkin. Process for purification of mineral oils, and production of alcohol. July 2.

13,933. R. B. Helliwell. Firelighters. July 9.

14,715. E. Mansfield. Oil-gas apparatus. July 2.

14,717. G. W. Macalpine. Coke-oven valves. July 9.

19,256. G. C. Trewby. Method and means for charging gas retorts. July 2.

1890.

260. J. Stears. Preventing deposit of carbonaceous matter in gas ascension pipes. July 9.

1556. W. C. Owston. Coal saving composition. July 9.

6800. W. C. Andrews. Purification of illuminating or heating gases. July 9.

6804. W. C. Andrews. Apparatus for purification of illuminating gas. July 9.

### III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

#### COMPLETE SPECIFICATION ACCEPTED.

1889.

19,183. J. A. Yeadon and W. Adgie. Retorts or furnaces for the distillation of coal, or other analogous purposes. June 25.

### IV.—COLOURING MATTERS AND DYES.

#### APPLICATIONS.

9322. R. Holliday and Sons, Lim., and T. Holliday. Improvements in the formation of new colouring matters in or upon cotton or other fibre. June 17.

9530. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. Improvements in the production of blue-green colouring matters. June 19.

9537. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik, Germany. Improvements in and relating to the manufacture of substantive dyestuffs, and of new materials therefor. June 19.

9676. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik, Germany. Improvements in and relating to the manufacture of substantive dyestuffs, and of materials therefor. June 21.

9768. J. Dawson and R. Hirsch. The production of a new  $\alpha$ - $\beta$ -naphthylendiammonosulpho acid. June 24.

9825. A. J. Boulton.—From G. Boulet, fils, Donard, and Contamine, France. See Class XII.

9923. O. Imray.—From C. F. Boehringer, Sohn, Germany. Manufacture of dehydromethylphenyl-pyrazine. June 26.

10,047. O. Imray.—From The Society of Chemical Industry, Switzerland. Improvements in the production of colouring matters. June 28.

10,095. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik, Germany. Improvements in the manufacture of trioxy-benzo-phenone, a colouring matter of the oxyketone group. June 30.

10,101. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. Improvements in the production of azo-colouring matters. June 30.

10,237. R. Holliday and Sons, Lim., and P. R. E. Deidler. Improvements in the manufacture of mono-sulpho acid of alpha naphthol. July 2.

10,240. R. Holliday and Sons, Lim., and P. R. E. Deidler. Improvements in the manufacture of azo colours. July 2.

10,401. W. S. Conrad. An instantaneous copying ink. July 5.

10,448. Brooke, Simpson, and Spiller, Lim., and A. G. Green. The production of new azo-colouring matters. July 5.

10,470. J. Dawson and R. Hirsch. Process for the manufacture of new basic blue colouring matters. July 7.

10,509. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik, Germany. The manufacture and production of artificial indigo, and of a new material suitable for use therein. July 7.

10,599. H. H. Lake.—From K. Oehler, Germany. Improvements relating to the manufacture of colouring matters. July 8.

10,802. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. Production of novel colouring matters for wool dyeing and printing. July 11.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

13,558. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik. Production of a new colouring matter suitable for dyeing and printing cotton without the aid of a mordant. June 25.

13,710. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning. Production of azo-colouring matters from dioxynaphthalinmonosulphonates. June 25.

14,222. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik. Production of diazo colouring matters. July 16.

14,388. R. Ashton. Method of preparing colours for making writing inks. July 16.

15,706. M. Lange. Production of thioxydiphenylamine. July 2.

### V.—TEXTILES, COTTON, WOOL, SILK, Etc.

#### APPLICATIONS.

9430. T. Mellor. Improvements in the method of and apparatus for steaming, boiling, and disinfecting fabrics or fibres. June 18.

9643. A. Hodge. Improvements in the treatment of ramie or rena fibre, or China grass and such like fibrous growths, and in the appliances or mechanism connected therewith. June 21.

9711. J. Miller, sen., and J. Miller, jun. Improvements in the method of and apparatus for rendering textile fabrics waterproof by one treatment or process. June 23.

10,285. T. J. Hutchinson. Improvements in the method of and means for removing grease and fatty matters from textile fabrics. July 3.

10,517. E. Dezwarte and The General Financial and Industrial Corporation, Limited. Improvements in the treatment of ramie, hemp, flax, and other fibrous plants. July 7.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

13,626. A. S., and F. Ambler. Cleansing wool and like animal fibres and apparatus therefor. July 2.

15,333. K. T. Sutherland and G. Esdaile. Treatment of fibres to promote easier cleansing or bleaching. July 9.

18,932. K. T. Sutherland and G. Esdaile. Method of treating vegetable fibres to produce easy spinning and greater strength. July 16.

1890.

7389. J. Longmore and W. L. Watson. Decorticating rheia, jute, and other vegetable fibrous stems. July 16.

8520. P. A. Fayler. Machines for decorticating ramie or other textile plants in a dry or a green state. July 9.

#### VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

##### APPLICATIONS.

9322. R. Holliday and Sons, and T. Holliday. See Class IV.

10,323. W. Hutchinson. Improvements in apparatus for boiling, scouring, or cleansing textile fabrics. July 3.

10,324. W. Hutchinson. Improvements in apparatus for use in the treatment of cloth with bleaching and other liquors. July 3.

10,396. F. A. Blair. Improvements in and connected with vats or apparatus and machinery for washing, bleaching, scouring, mordanting, and drying yarns or stubbing in the bank. July 5.

#### COMPLETE SPECIFICATION ACCEPTED.

1890.

5051. C. Kellner. See Class XI.

#### VII.—ACIDS, ALKALIS, AND SALTS.

##### APPLICATIONS.

9670. J. C. Steele. Improvements in the packing or putting up of chloride of lime and other substances. Complete Specification. June 21.

9793. F. W. A. Frerichs. Improvements in and relating to the manufacture of carbonates of alkalis and acetone. Complete Specification. June 24.

9929. A. G. Haddock and J. Leith. Improvements in the manufacture of alkaline carbonates and by-products, and in arrangement of plant therefor. June 26.

9997. F. Ellershausen. Improvements in the manufacture of sodium ferride and caustic soda. June 27.

10,115. H. Precht. Improvements in the process of and apparatus for purifying double carbonate of potassium and magnesium. June 30.

10,194. O. Murray.—From J. Storer, Australia. A process for producing alkaline carbonates from sulphates. July 1.

10,399. J. Barlow and H. T. Everist. Improvements and apparatus in connexion therewith for the crystallisation of salt from brine. July 5.

10,451. C. G. Moor. See Class XVIII., B.

10,737. A. P. J. Archbold. Improvements in the manufacture of salt for domestic purposes. July 10.

10,790. H. J. Haddan.—From C. Negrier, France. Improvements in apparatus for the concentration of sulphuric acid. July 11.

10,906. F. Ellershausen. Improvements in the manufacture of caustic soda. July 12.

10,910. F. Ellershausen. Improvements in the manufacture of sulphide of sodium. July 12.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

11,975. E. Augé. Manufacture of soda ash. July 2.

13,575. M. N. D'Audria. Treating liquid acid residues containing iron to obtain products therefrom. July 2.

13,726. E. de Cuyper. Recovering valuable substances from the mother-liquor of copper ores. July 16.

16,609. W. Wolters. Recovery or regeneration of nitric acid employed in the manufacture of chlorine. July 9.

1890.

8090. J. D. Pennoek and J. A. Bradburn. Manufacturing bleaching powder and caustic soda. July 2.

8915. O. Guttmann. Apparatus for condensation of nitric acid and other distillation products. July 16.

#### VIII.—GLASS, POTTERY, AND EARTHENWARE.

##### APPLICATIONS.

9867. W. H. Hilde. Improvements in the manufacture of hollow glass ware, and in apparatus therefor. June 25.

9942. O. Dillmann. An improved method for producing coloured designs on glass. June 26.

10,018. D. Rylands. Improvements in the appliances, processes, and heat producers used for annealing or toughening bottles or other ware. June 28.

10,795. A. Bevington. An improved process for the decoration of china and earthenware. July 11.

10,847. D. Rylands and R. Morant. Improvements in the manufacture of glass tubing. July 12.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

13,036. A. Taylor. Method and apparatus for building up inside of kilns for burning enamelled bricks and tiles. July 16.

13,994. S. Washington. Finishing glass ware, and apparatus therefor. July 2.

14,156. The Coalport China Co. (J. Rose and Co.) and T. J. Bott. Ornamenting china, earthenware, glass, &c. July 16.

1890.

1820. H. Gurney. Method and apparatus for burning bricks and tiles. July 2.

6058. G. Scott. Method and apparatus for manufacturing roofing plates from glass, &c. July 2.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

### APPLICATIONS.

9306. C. J. Howe. An instrument for testing the setting properties of cements and plasters. June 16.

9381. D. H. Close. Improvements in the manufacture of bricks. June 17.

9618. J. C. Bloomfield. Enamelled bricks, blocks, slabs, or tiles. June 21.

10,002. T. Breakell and J. W. Breakell. A method of colouring and polishing calcareous and other stones, and producing variegated effects thereon in one or more colours. Complete Specification. June 28.

10,036. J. P. Bayly. — From W. Lenderoth, Canada. Improvements in and relating to the manufacture of porous earthenware building material. June 28.

10,427. W. Darling. Improvements in and relating to the calcining of carbonaceous minerals for cements and other purposes. July 5.

10,548. L. Wilson. Improvements in blocks and tiles for paving, flooring, facing walls and ceilings, and for similar purposes. July 8.

### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

13,008. J. Wilson. Construction of fire-resisting flooring, decking, &c. July 9.

13,749. H. Aitken. Treating timber. July 9.

1890.

3184. A. Rovedins. Improvements in and connected with paving. July 9.

6058. G. Scott. See Class VIII.

## X.—METALLURGY, MINING, Etc.

### APPLICATIONS.

9684. C. T. J. Vautin. Improvements in the recovery of gold from solutions. June 21.

9746. A. Jones and J. W. Sheel. Safety appliance for Bessemer converters. June 24.

9830. D. Rennie. Improvements in furnaces for melting crucible steel and other metals. June 25.

9835. R. T. Cooke. Improvements in ingot heating furnaces. June 25.

9900. A. Philip. Improvements in the manufacture of sodium and potassium. June 26.

9906. C. A. Burghardt. An improvement in the manufacture of tin andterne plates. June 26.

10,029. J. Ward. Improvements in and relating to the manufacture of compound armour plates. June 28.

10,062. J. Bradbury. A new or improved process for the utilisation of oxide of iron obtained as a by-product in the smelting and extraction of copper from pyrites, or from spent oxide produced in the manufacture of sulphuric acid. June 30.

10,175. J. B. Torres. Certain new or improved refractory compounds and mixtures. July 1.

10,510. C. G. Cunningham. Improvements in the reduction of ores containing lead, zinc, silver, or gold. July 7.

10,537. C. J. Ellis and A. Dickie. Improvements in re-converting for re-use residuary cyanides such as are obtained in extracting gold from ores. July 8.

10,555. E. Regnander and P. A. Waller. Improvements in moulding ingots of phosphor tin, phosphor bronze, and the like, and in apparatus therefor. July 8.

10,620. W. Withington. Improvements in the manufacture of steel castings. July 9.

10,625. J. E. Stead. Improvements in the manufacture of basic phosphatic slag. July 9.

10,711. G. A. Herdman. Improvements in treating waste products resulting from the smelting of ores, and in producing a valuable product therefrom, or from analogous compounds. July 10.

10,783. A. McDougall. A new process for recovering tin from tin plate in which zinc white is produced as a product. July 11.

### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

12,556. W. P. Thompson. — From A. A. L. Levy. Coating or plating metal articles. July 16.

14,226. A. J. Boulton. — From M. Castelnaud. Treatment of ores. July 2.

14,357. E. Dodé. Refining and separating precious metals. July 2.

18,985. C. A. Piat. Metallurgical furnaces. July 16.

1890.

1733. J. F. Spence. Apparatus for charging pig iron and scrap metal into open-hearth furnaces. July 9.

4675. R. T. Tiedford. Welding metals. July 2.

6864. J. J. Shedlock and J. J. Meldrum. Apparatus for production and application of gases for metallurgical and other purposes. July 9.

7733. H. H. Lake. — From G. W. Cummins. Process and apparatus for preventing access of air to iron and steel during annealing. June 25.

7820. F. G. Bates. Preparation of materials for converting low steel or iron into high grade steel by the cementation process. June 25.

8655. E. C. Miller. — From The Magnolia Anti-friction Metal Co. Compositions or alloys for anti-friction purposes. July 16.

8989. H. H. Lake. — From G. W. Cummins. Treating copper and its alloys to prevent oxidation and deoxidation during heating and annealing. July 16.

8991. H. H. Lake. — From J. D. Brown. Manufacture and treatment of iron and steel, and compounds therefor. July 16.

## XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

### APPLICATIONS.

9330. W. H. Dowland and W. Mills. Improvements in the electrolysis of chlorides, and apparatus therefor. June 17.

9361. T. L. Willson. Improvements in electric reduction of metals, and in apparatus therefor. June 17.

9857. M. W. Dewey. Improvements relating to welding, soldering, brazing, and otherwise working metals by electricity, and to apparatus therefor. Complete Specification. June 25.

9861. A. J. Boulton. — From D. Tommasi and C. Therye, France. Improvements in electrical storage batteries. June 25.

9956. R. Kennedy. See Class II.

9991. T. Parker. Improvements in or connected with the manufacture of iron by electrolysis. June 27.

10,038. A. Watt. Improvements in the electrolytic treatment of metals. June 28.

10,044. Sir C. S. Forbes. Improvements in primary batteries, and in apparatus connected therewith. June 28.

10,123. M. W. Dewey. Improvements relating to welding, soldering, and otherwise working metals by electricity. Complete Specification. Filed June 30, date applied for December 11, 1889, being date of application in United States.

10,125. M. W. Dewey. Improvements in electric heating apparatus. Complete Specification. June 30.

10,181. T. M. Foote. Improvements in secondary batteries. Complete Specification. July 1.

10,211. V. Fabris. Improvements in electric primary batteries. July 2.

10,515. A. J. Jarman. An improved connector for the electrodes and cells of primary and secondary electrical batteries. Complete Specification. July 7.

10,527. E. S. D'Odiardi and A. Thompson. Improvements in magneto-voltaic and static electrodes. July 8.

10,530. E. S. D'Odiardi and A. Thompson. Improvements in liquid electrodes. July 8.

10,538. G. J. Wier. Improvements in galvanic batteries. July 8.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

10,822. G. Button and W. E. Wyeth. Apparatus for extracting gold and silver from their ores by electrical amalgamation. July 9.

11,242. Siemens Bros. and Co., Lim.—From Siemens and Halske, Germany. Dynamo-electric, magneto-electric, and electro-dynamic machines. July 16.

12,772. A. de Castro. Means or apparatus for generation and conversion of electric energy. July 16.

1890.

1347. T. Coad. Fluid for primary batteries. June 25.

4191. T. D. Farrall. Method and mechanism for generating heat, steam, and light by electricity. July 9.

4949. L. Weigert. Electrolytic solution for galvanic batteries. July 9.

5054. C. Kellner. Bleaching fibrous material by aid of electricity. July 9.

5420. C. Kellner. Process and means for manufacture of cellulose by aid of the electric current. July 16.

6177. G. Barker.—From H. Woodward. Secondary batteries. July 16.

6236. A. J. Jarman. Improved storage battery. June 25.

7185. W. P. Thompson.—From C. L. Coffin. Method and apparatus for welding metals electrically. July 2.

7853. W. P. Thompson.—From C. L. Coffin. Method and apparatus for welding metals electrically. July 9.

7876. H. H. Lake.—From E. A. Colby. Methods and apparatus for melting, refining, and casting metals, &c., by electricity. July 16.

8578. J. Y. Johnson.—From C. Hering. Secondary batteries and plates or elements therefor. July 9.

8703. W. P. Thompson.—From C. L. Coffin. Electric welding. July 9.

#### XII.—FATS, OILS, AND SOAP MANUFACTURE.

##### APPLICATIONS.

9614. P. A. Newton.—From The Chesebrough Manufacturing Co., Consolidated, United States. Improvements in the manufacture of compressed cakes of soap. June 20.

9659. J. S. Fairfax.—From F. Crane, United States. Improvements in or relating to the preparation of oils for compounding purposes. June 21.

9825. A. J. Boulton.—From G. Boulet fils, Donard, and Contamine, France. Improvements in or relating to apparatus applicable for the extraction of fatty substances from oil, sulphur, and other material, and for manufacturing extracts for dyes, and other purposes. June 24.

9856. G. Anderton, G. T. Barlow, and T. G. Marsh. Improvements in or connected with the bleaching or extracting colour from wax. June 25.

10,173. J. N. Zeitler. Process for producing hard and soft potash soaps and hard soda and potash soda soaps by spontaneous heating. July 1.

10,590. L. Roumieu. Improved process and apparatus for the extraction of oils from oleaginous seed or grain by means of volatile solvents. July 8.

10,753. A. F. St. George. Treatment of linseed and other oils employed in varnishes. July 11.

10,848. T. G. Croker. An improvement upon Brooke's soap. July 12.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

11,292. H. Enrich. Producing hard potash and similar soap from solid or liquid fats. July 16.

11,817. C. R. Huxley. Ammoniated soaps. July 2.

13,023. J. L. Moret. A solid product for removing grease, bleaching textile materials, fulling cloths, and removing hair from skins. July 2.

1890.

7854. A. W. Rhenstrom. Manufacture of soap. June 25.

8077. G. Allen. Composition of soap. July 2.

9062. C. D. Heustrom. Separation of fats from emulsions. July 16.

#### XIII.—PAINTS, PIGMENTS, VARNISHES, AND RESINS.

##### APPLICATIONS.

9822. A. J. Boulton.—From W. Kiel, United States. Improvements in and relating to the manufacture of vulcanised plastic compounds. Complete Specification. June 24.

9897. J. Gordon. Improvements in making white pigments of lead. Complete Specification. June 26.

10,130. M. Alsberg. A new or improved process for producing minium. Complete Specification. July 1.

10,616. G. R. B. Kempton. An improved blue for laundry and other colouring purposes. Complete Specification. July 9.

#### XIV.—TANNING, LEATHER, GLUE, AND SIZE.

##### APPLICATIONS.

9402. O. Potelune and O. Potelune and Co. New or improved machinery or apparatus for treating skins. June 17.

10,337. A. S. F. Grunbaum and T. B. Wood. An improvement in the manufacture of blood albumen. July 3.

10,706. S. Thorn. The production from raw hides of an article in substitution of what is commonly known as tanned leather. July 10.



## XV.—AGRICULTURE AND MANURES.

## COMPLETE SPECIFICATION ACCEPTED.

14,719. A. Manbré, sen. See Class XVII.

## XVI.—SUGARS, STARCHES, GUMS, Etc.

## APPLICATIONS.

10,331. A. Lefranc, L. Lefranc, and A. Vivien. Improvements in and connected with the manufacture of sugar. July 3.

10,449. W. R. Hutton. Improvements in saccharate of lime. July 5.

10,893. R. Pzillas. Apparatus for drying sugar or like substances. Complete Specification. July 12.

## COMPLETE SPECIFICATION ACCEPTED.

1889.

11,136. S. Pitt.—From L. W. Tracey. Apparatus for the treatment of cane juice. July 9.

## XVII.—BREWING, WINES, SPIRITS, Etc.

## APPLICATIONS.

9442. L. C. Best. Improvements in apparatus to facilitate the fining of beer. June 18.

9910. D. A. Bouallanger and A. Scheunert. Improvements in the process of and apparatus for purifying alcoholic liquids. June 26.

10,160. F. X. Kleinpeter. A new process for producing extract of malt or malt sugar in the malt grain. July 1.

10,436. W. P. Thompson.—From T. Bolland, Venezuela. Improvements in or relating to distilling apparatus for spirits and the like. July 5.

10,845. A. Boake and F. G. A. Roberts. An improved means of applying certain antiseptic agents to beer, wine, or other fermented drinks. July 12.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

10,667. W. W. Mutter and W. Dawson. Improvements in distillers' safes, also applicable for check-sampling spent wash at outlets of "patent" stills. July 2.

13,653. W. P. Thompson.—From P. Marix and N. Notkin. See Class II.

14,406. J. Hutchison. Treatment of alcoholic liquors. July 16.

14,719. A. Manbré, sen. Manufacture of spirit from grain and cereals, and treatment of by-products therefrom for production of cattle-food, charcoal, and manure. July 16.

## XVIII.—CHEMISTRY OF FOODS, SANITARY CHEMISTRY, AND DISINFECTANTS.

## APPLICATIONS.

*A.—Chemistry of Foods.*

9701. E. W. Hewett, jun. Bread-making with potatoes as the principal ingredient, and improvement therein. June 23.

10,004. E. McSwiney and S. J. Cluff. An improvement in preserving meat and other perishable goods by an antiseptic gas. June 18.

10,563. F. A. Raedler. Improved process for sterilising milk. July 8.

10,663. W. W. Bull and J. B. Baylis. New or improved manufacture of desiccated potato. July 9.

10,798. T. H. Poupon. An improved method of and apparatus for preserving meat, game, fish, vegetables, and the like. July 11.

*B.—Sanitary Chemistry.*

10,157. F. W. Shorey and C. R. Hudson. Separating the liquids from the solids in sewage and other like bodies. July 1.

10,229. A. Green. Improved means for decomposing sewage water by oxidising zinc by electric current. July 2.

10,313. F. Scudder. Improvements in the treatment of refuse material, such as town refuse, for obtaining valuable products therefrom. July 3.

10,451. C. G. Moor. An improved process for the recovery of ammonia from sewage sludge. July 5.

*C.—Disinfectants.*

9681. H. D. Booth. Disinfecting sinks, urinals, and the like with perforated disinfectant soap. June 21.

10,144. T. Sheppard and W. Sheppard. Preserving organic fluids. July 1.

## COMPLETE SPECIFICATIONS ACCEPTED.

*A.—Chemistry of Foods.*

1889.

11,806. W. Palmer. Preparation of a solid extract of meat and a preservative coating for same and other preserved foods. July 2.

13,438. C. A. Coombe. Treating brewers' grains, and manufacturing feeding cakes therefrom. July 2.

13,439. C. A. Coombe. Cattle-feeding cake. July 9.

1890.

8018. J. White. Jelly powder. July 2.

8354. W. C. H. Jessen. Preserving eggs. July 9.

*B.—Sanitary Chemistry.*

1889.

13,746. W. P. Buchan. Deodorising or disinfecting sewage and other polluted water. July 9.

1890.

7500. L. Hesse. Destructors for town and other refuse. July 9.

*C.—Disinfectants.*

1889.

13,203. J. T. MacMahon. Composition for arresting or preventing decay in wood. July 2.

## XIX.—PAPER, PASTEBOARD, Etc.

## APPLICATIONS.

9953. A. Gray. The manufacture of a new material to be called "Lacrepap," consisting of paper, wool, or cotton, separately or together, hardened or solidified by a process given herewith. June 27.

10,259. J. F. N. B. Simons and S. Smith. Improvements in the manufacture of paper pulp. July 2.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

- 13,657. M. Frisch. Press copying paper. June 25.  
 14,508. J. A. Causton and J. Challis. Process for applying luminous preparation to the surface of paper, &c. June 25.

1890.

5053. C. Kellner. Softening brittle paper pulp. July 9.  
 6348. H. H. Lake.—From J. H. Carpenter and C. E. O'Hara. Manufacture of coloured paper. July 16.  
 8422. O. Evanstad and O. Senstad. Apparatus for manufacture of wood-wool. July 9.

## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES, AND EXTRACTS.

## APPLICATION.

- 10,432. H. Lütke, L. Scholven, and L. F. Riedel. The manufacture of salicylate of phenyldimethylpyrazolone. July 5.

## COMPLETE SPECIFICATION ACCEPTED.

1889.

- 13,027. A. M. Clark.—From the Firm of F. von Heyden's successor. Separation of the isomeric substances produced in the manufacture of dithio-salicylic acid. July 2.

## XXI.—PHOTOGRAPHIC PROCESSES AND MATERIALS.

## APPLICATIONS.

9538. W. Rebikow. Improvements in the manufacture and production of emulsions and other materials sensitive to light. June 19.  
 9893. J. W. Swan and J. Leslie. Improvements in the manufacture of transparent flexible films for photographic and other purposes. June 26.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

- 10,353. J. S. Fairfax.—From F. Crane. Films or supports for photographic negatives or prints. July 2.  
 12,309. J. Williams. Treating paper, &c., to render it suitable for photographic purposes. July 2.

## XXII.—EXPLOSIVES, MATCHES, Etc.

## APPLICATIONS.

9315. E. N. Todd. Improvements in the process of manufacturing thin sheets of nitro-cellulose. Complete Specification. June 16.  
 9350. J. A. Goudge. The protection of gunpowder cartridges, fireworks, fire signals, and all solid explosives used for a similar purpose to gunpowder, from absorbing moisture. June 17.  
 9660. J. S. Fairfax.—From F. Crane, United States. Improvements in or relating to pyroxylic compounds. June 21.  
 10,025. W. M. Mordey. Improvements in electric safety fuses. June 28.  
 10,263. K. R. Malstrom. Improvements in percussion fuses. July 2.  
 10,321. A. J. Boulton.—From H. H. Rosenfeld, France. Improvements in the manufacture of matches. July 3.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

5614. Sir F. A. Abel and J. Dewar. Manufacture of explosives for ammunition. July 9.  
 11,500. P. Ward and E. M. Gregory. Fuses and detonators. July 9.  
 11,501. P. Ward and E. M. Gregory. Construction of electric fuses, and method and appliances for manufacturing same. July 16.  
 12,307. A. V. Newton.—From A. Nobel. Preparation and application of explosive compounds. June 25.  
 12,338. C. O. Lundholm and J. Sayers. Manufacture of explosives. June 25.  
 12,763. J. Lutz and L. Oltósy. Manufacture of lucifer matches. June 25.  
 13,486. C. O. Lundholm and J. Sayers. Making and applying explosives. July 16.  
 14,073. W. Clayton. Production of whistling fireworks. June 25.  
 14,804. H. Stephenson. Manufacture of certain fireworks for display purposes. July 16.

1890.

5364. J. R. France. Manufacture of nitro-cellulose, and apparatus therefor. July 9.

# THE JOURNAL

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## Journal and Patent\* Literature.

### I.—GENERAL PLANT, APPARATUS, AND MACHINERY.

#### PATENTS.

*Improved Apparatus for regulating the Flow of Compressed Gases.* A. T. Clarkson and J. B. Spurge, London. Eng. Pat. 11,518, July 18, 1889. 8d.

This invention relates more particularly to apparatus used for gases contained under pressure in vessels for transport, but it can also be used for other purposes where constant pressure is required to be obtained from a source sometimes greatly in excess, and variable in amount, and can be applied to the regulation of nitrous oxide for anæsthetic purposes, in the preparation of aerated waters, or for transporting or elevating liquids. Two sets of apparatus are used, which, though differing slightly in construction are the same in action, and are placed in communication with each other.

The first is intended to reduce any pressure ranging up to 150 atmospheres to any other that may be required, and consists of an expanding box having a flexible side or diaphragm pressed inwards by steel springs, which can be adjusted to counteract any pressure in the opposite direction, and has two openings communicating with the interior. To the diaphragm is also attached "scroll or cam-shaped pieces," actuating a valve and conical plug. The second apparatus is attached to the first by one of the openings alluded to, and differs from it mainly in the mode of fixing the valve. The effect of these improvements is said to "ensure efficient action over a greater range of pressures than hitherto, and at a less outlet pressure, the supply being maintained until the inlet pressure has fallen to one-third of an atmosphere."—E. S.

*Improvements in Filtering Apparatus.* J. M. Smales, Leavesden. Eng. Pat. 12,162, July 31, 1889. 8d.

A SOLID disc of metal or other material has radial or circular grooves on each side, and is covered with cotton cloth or other filtering medium. A circular opening is made in the centre, and round it is a boss having radial internal openings. A series of these discs is mounted on a hollow shaft having slots or holes, the discs being separated from each other by collars, and the whole set is placed in a cistern. The liquid to be filtered is admitted to the cistern, and passing through the filtering medium follows the course of the grooves to the holes in the boss, and into the hollow shaft by means of the holes therein, and flows out of the cistern. By communicating motion to the series of discs the deposit can be more readily washed off than when they are stationary.

—E. S.

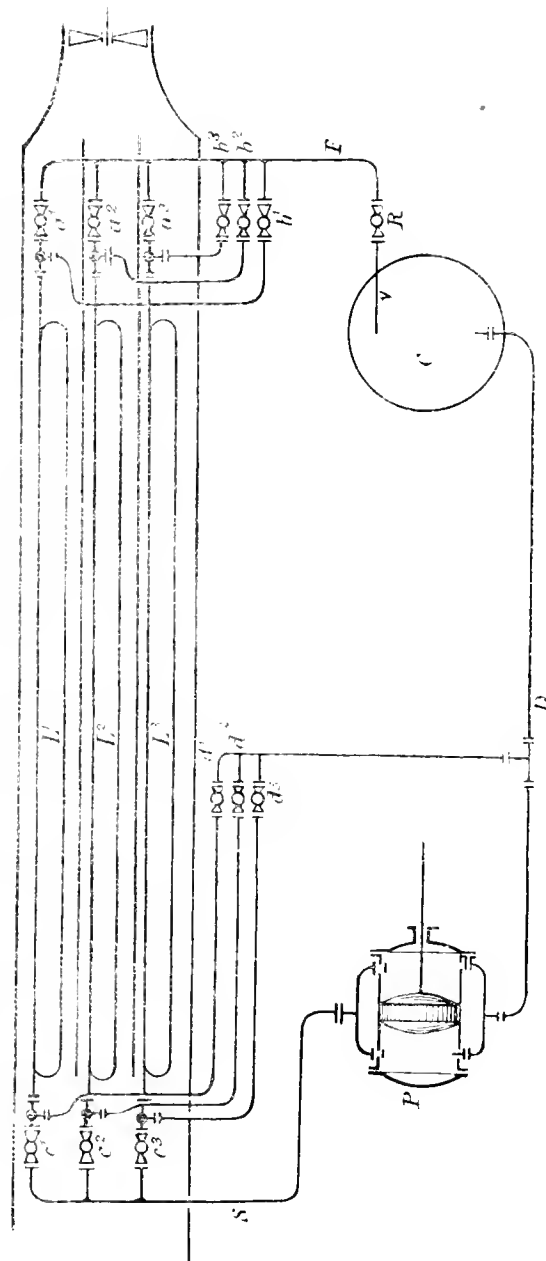
*Improvements in Multiple-Effect Evaporating Apparatus for Evaporating or Concentrating Sugar Juice and other Liquids.* W. Maxwell, Djoeclja, Java. Eng. Pat. 12,809, August 14, 1889. 8d.

As compared with evaporators of ordinary construction the improvements consist (1) in substituting for the central internal tube two or more external circulating tubes connecting the upper and lower chambers, and passing outside

\* Any of these specifications may be obtained by post, by remitting the cost price, plus postage, to Mr. H. Reader Lack, Comptroller of the Patent Office, Southampton Buildings, Chancery Lane, London, W.C. The amount of postage may be calculated as follows:—

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TO BE SUBSTITUTED FOR THE FIGURE ON PAGE 594 IN THE JUNE NUMBER OF THIS JOURNAL.





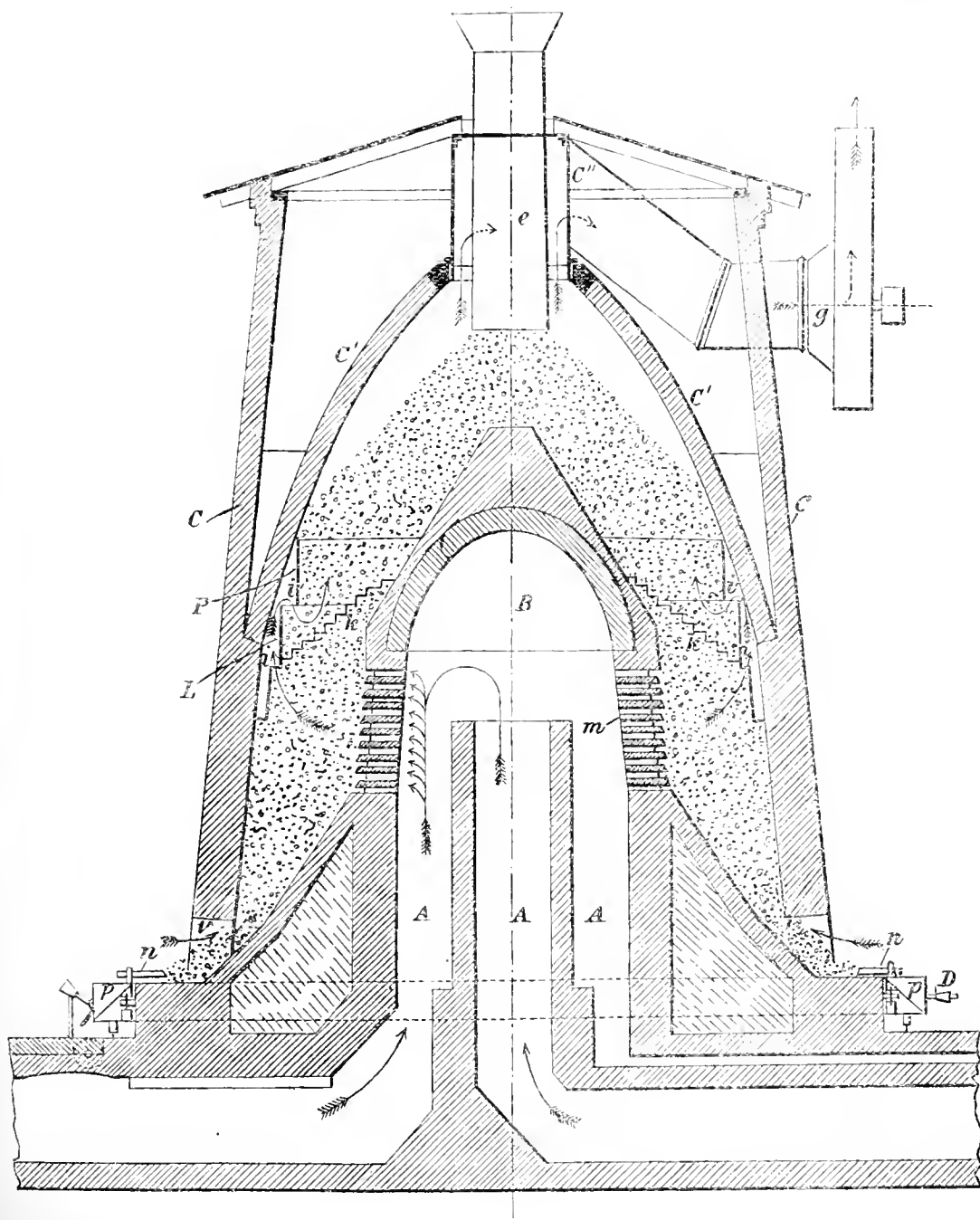
the tube chamber, thus allowing the space so obtained in the tube chamber to be utilised for additional heating surface; (2) in constructing the connecting pipe from the upper chamber of each evaporator to the tube chamber of the next, so that the inlet to the latter is deep in proportion to its breadth, and extends nearly the whole depth of the tube chamber; and (3) in arranging the tubes in the tube chamber so as to form one or more passages between them diverging from the inlet, and thus allowing the vapour entering by this inlet to have free access to the external surface of all the tubes. By these improvements it is stated that the heating medium readily reaches every part of the heating surfaces, while at the same time the greater rapidity with which the heating vapour is condensed, and the

diminished resistance to its flow, allow the evaporation in the liquid from which it arises to go on under a lower pressure and temperature.

There are five claims.—E. S.

*Improvements in Apparatus for Drying Earthy, Disintegrated, or Similar Materials.* V. F. L. Smidth, Copenhagen, Denmark. Eng. Pat. 12,863, August 14, 1889. 11d.

THE invention relates to apparatus for drying large quantities of small or disintegrated materials by means of hot air, and it may best be explained by reference to the accompanying drawing, which shows one modification of the apparatus.





It consists of a circular building or tower C of suitable height, closed at the top and fitted with a funnel *e* in the centre of the roof, through which the materials are fed. The funnel is surrounded by a shell C' open at the lower end and resting air-tight on the dome C' whilst connected by a flue with the exhaust fan *g*. From the floor of the tower rise one or more flues A to a convenient height, through which the hot air enters. They are surmounted by the truncated, spaced rings *m* and closed by the small dome B, which causes the hot air to pass through the spaces between the rings and through the body of the materials to be dried, assisted and directed by other rings and projections specially provided for that purpose in the annular space, as indicated by the letters L P k i and the arrows. The lower part of the walls of the tower C is perforated with openings *v* through which the dried material issues, and is thence removed mechanically in the following manner:—At the foot of the tower is a projecting ledge or step below the said openings *v*, which is encircled by a revolving ring D carried on and guided by suitable wheels or rollers. Attached to the ring are a number of scrapers *n*, and it is fitted with corresponding pockets *p* into which the material falls as it is brought forward by the continuous action of the scrapers. The pockets have side doors, closed by weights, which are raised by means of catches when they reach the point where the dried material is to be discharged. The material falls into a worm below, for conveyance to any desired point, whilst the vapour and gases are removed by the fan *g*.

The patentee makes two claims, and there are four sheets of drawings.—B.

*Improvements in and Relating to Vacuum Evaporating and Distilling Apparatus.* J. Foster, Sunderland. Eng. Pat. 18,153, November 13, 1889. 1s. 1d.

This invention consists of improvements in apparatus for evaporating, concentrating, and distilling sugar, caustic lyes, and other liquors in a vacuum (more or less) in vessels of the character of "triple or multiple effects." The improvements are specially applicable to the horizontal arrangements of apparatus described in Eng. Pat. 7343 of 1889. For the mode of construction and operation, the three sheets accompanying the specification must be consulted.

There are eight claims.—E. S.

*Improvements in or Appertaining to Pulverising Machines.* W. P. Thompson, Liverpool. From J. J. Bordinan, Brooklyn, U.S.A. Eng. Pat. 19,538, December 5, 1889. 8d.

This invention relates to improvements in pulverising machines, especially those provided interiorly with rotary fans and beaters, and refers more particularly to the balancing and adjusting of the shafts carrying the beaters and fans, the object being to provide means for overcoming the difficulties caused by the unequally wearing away or straining of certain parts in the working of the apparatus. This is done by effecting an immediate balance of the shaft at any desired moment, whenever the various parts of the beaters, fans, or internal mechanism become worn, by the use of sliding weights and moveable friction bearings. Full details are given in the drawing attached to the specification. There are seven claims.—E. S.

*Improvements in Asbestos Filters.* F. Breyer, Vienna, Austria. Eng. Pat. 5377, April 8, 1890. 8d.

The filtering medium employed is very finely divided asbestos, which has been sterilised by exposure to heated air. The disintegration of the asbestos, before it is made into an emulsion for application to the filtering bodies, is effected by moistening it with water, freezing the mass, and then reducing the frozen material.—E. G. C.

## II.—FUEL, GAS, AND LIGHT.

### PATENTS.

*Improved Process for Burning Petroleum and other Hydrocarbons, and Apparatus therefor.* W. P. Thompson, Liverpool. From J. Wilson, New York, and A. Mason, Brooklyn, U.S.A. Eng. Pat. 10,661, July 2, 1889. 11d.

The object of this invention is to so present the oil to the heating apparatus that perfect combustion ensues, and to obtain more heat from a given quantity of the material than by the usual means of supplying heat to atomise the petroleum, so that continuous uninterrupted combustion takes place; also to obtain a safe, continual, and economical heat for mechanical and other purposes.

Petroleum, or other oil or fatty substance, steam, and air, in proper proportion, are introduced, and the oil is thoroughly atomised and mixed with steam by impinging steam or air upon it, or oil upon steam or air, on its way to the flame. The air makes the combustion perfect, and may, when used without steam, be injected to produce the draught and spray the oil. The steam, when used, supplies the drawing or forcing power, and, in addition, the steam decomposes in the flame, and gives an additional element of heat in the evolving hydrogen and oxygen. This decomposition can be assisted and stimulated, as also the vapourising of the oil, by the use of superheated steam and hot air.

There are eight claims and one sheet of drawings.

—F. S. K.

*Improved Apparatus for Generating Gas or Vapour from Combustible Liquids.* J. Dheyne, Count V. de Nydprück, and P. de la Hault, Brussels, Belgium. Eng. Pat. 11,459, July 17, 1889. 8d.

The apparatus consists of a tank, charged with naphtha, petroleum, or the like, which flows off through a pipe ending in a coil, and the end of which is bent back in such a manner as to throw into the axis of the coil the gas escaping from the end. The coil is in contact with a metal surface or tube, and placed above a vessel or tray within which is burnt, for the purpose of starting the apparatus, any highly combustible liquid. The gas created by the heat of this combustion is ignited at the end of the coil, and the flame heats the latter up to a temperature which can be regulated by means of a cock, thus varying the quantity of gas escaping from the coil. In a second coil or worm, heated by the same flame, the vapour required for working a motive-power engine is generated. Modifications of the apparatus suitable for generating gas for heating purposes, or for supplying lamps, such as are employed in soldering and enamelling, are also described.—F. S. K.

*The Manufacture of an Improved Artificial Fuel.* J. Morris, London. Eng. Pat. 11,855, July 25, 1889. 4d.

The improved smokeless and inodorous fuel is prepared by mixing silicate of sodium, nitrate of potassium, water, powdered charcoal, powdered carbonate of lime, and powdered binoxide of manganese.

The whole is intimately mixed and then compressed into briquettes or bricks, and thoroughly dried at a low temperature.

The rate of combustion can be increased or diminished by increasing or diminishing the percentage of silicate of sodium. It is alleged that "this improved fuel being smokeless and inodorous, and giving off no noxious fumes, can be burned without a flue, and is, therefore, especially applicable for warming carriages, for rooms and other places which have no flues, and is very useful for heating greenhouses."

—F. S. K.

*Improvements in Incandescent Gas Burners.* C. Clamond, Paris, France. Eng. Pat. 12,091, July 30, 1889. 8d.

THE invention consists in an improved arrangement for heating a mixture of air and gas before burning them under a hood of magnesia filaments. A metal chamber divided by a horizontal wire gauze partition into two compartments carries on its upper surface a number of vertical copper or brass tubes open at both ends, and is fixed on a short Bunsen burner provided with suitable air-holes and connected with the gas supply by means of a cap covered with a small perforated plate. A perforated circular gallery for holding a chimney is fixed under the base of the chamber by means of a screwed piece of steatite or some other non-conducting material, the chamber and tubes are also surrounded by a metal casing rising a little above the level of the tubes. A conical cage of magnesia filaments is attached to a metal base fitting on the top of the casing and is held in position by wires of some refractory metal. The gas is ignited at the top of the tubes and raises the magnesia to incandescence; the flame is prevented from flashing back down the Bunsen burner, especially on ignition, by the wire gauze partition in the metal chamber, by a small conical hood screwed mouth downwards on the burner masking the air-holes, and by a saucer screwed on over the hood mouth upwards to intercept the flames from above.—S. B. A. A.

*Improvements in Retorts or Furnaces for the Distillation of Coal, or other analogous Purposes.* J. A. Yeadon and W. Adgie, Leeds. Eng. Pat. 19,183, November 29, 1889. 8d.

ACCORDING to the inventors, the apparatus described below dispenses with the labour of gas stokers, utilises the smallest size of coal, secures regular and continuous feeding and rapid and complete distillation of the fuel, and prevents waste of gas whilst charging or discharging the retorts.

The apparatus consists of a cylindrical or conical fire-clay or metallic retort, the body of which lies inside the furnace and rests on metal sockets, one at each end of the furnace. The sockets are turned up on the circumference where they are supported and revolve upon bearing rollers which have suitable shafts and revolve in metal frames outside the brickwork of the furnace; the whole of the circumference of the retort within the furnace is thus directly heated by the flames. The interior of the retort is made with a number of continuous blades running longitudinally through it, tapering from the front or feeding end towards the other extremity and preferably made solid with the shell; these serve to raise, break up, and gradually force the coal outwards, they also strengthen the retort. At the front end the retort is fitted with a mouthpiece or end-plate connected with a hopper, from the bottom of which coal is fed through a small cylinder projecting some distance into the retort and fitted with an internal shaft with adjustable blades; a diaphragm prevents the coal from working back. At the back end of the retort there is a vertical uptake for the gases and a vertical downtake for the coke; the latter communicates with the furnace grate and with the exterior and contains two sliding doors or valves so that the coke may either be charged on the furnace grate or drawn off into barrows, &c., without any escape of gas. Coal may be supplied to the hoppers and withdrawn from the furnace by means of elevators and endless conveyors. The disposition of some of the driving machinery, details of packing, and various modifications are described and illustrated in the specification.—S. B. A. A.

*Improvements in the Purification of Illuminating or Heating Gases.* W. C. Andrews, New York, U.S.A. Eng. Pat. 6800, May 2, 1890. 8d.

THE invention consists in passing the gas through a solid purifying material in a state of continual or intermittent agitation, or upwards through a shower of the finely divided material, or through successive horizontal layers

of the material, each layer periodically falling through the gas into the position of the layer next below it, the lowest layer being discharged as spent and fresh material supplied in the place of the uppermost layer. An apparatus for practically utilising this invention is described (see following abstract).—S. B. A. A.

*Improvements in Apparatus for the Purification of Illuminating Gas.* W. C. Andrews, New York, U.S.A. Eng. Pat. 6801, May 2, 1890. 8d.

THIS apparatus is adapted to the method of the preceding patent, Eng. Pat. 6800 (preceding abstract). It consists of a closed box or casing containing a series of horizontal rows of shafts parallel with the sides of the box, each shaft moving in bearings on the front and back walls of the box, and having on its upper surface a series of loops or eyes into which horizontal cross-pieces are fixed at right angles to the shaft, the cross-pieces on each shaft alternating with those on adjacent shafts. The spaces between the cross-pieces on the shafts next the sides of the box are partially occupied by a series of slats or short rods fixed to each side; each row of shafts, cross-pieces and slats thus forms a grating or broken platform for the reception of the purifying material. Cranks are attached to one end of each shaft which projects from the box, and a long lever is attached to the ends of each row of cranks. It is evident that any motion of this lever will cause the cross-pieces on the shafts to oscillate and agitate or shake off any matter deposited on them. Two hoppers closed by valves are fixed under the bottom of the box for the reception of the spent material from the lowest platform. Gas is supplied from below and drawn off from the top of the apparatus. The box may be as much as 24 ft. in depth, the horizontal shafts in each row about 1 ft. 6 in. apart, the cross-pieces slightly less than 3 ft. in length, with from 1 in. to 1.5 in. space between adjoining strips. The platforms being covered with purifying material, gas is allowed to enter; one or more of the rows of shafts may then be rocked either continually or when the issuing gas is recognised to be wanting in purity, or they may be left at rest for the most part, and only used to clear the lowest layer to the hopper, and then to remove each succeeding layer in on to the platform below it, fresh material being supplied to the uppermost platform.—S. B. A. A.

*Improvements in Apparatus for the Production and Application of Gases for Metallurgical and other Purposes.* J. J. Shedlock, New Barnet, and J. J. Meldrum, Manchester. Eng. Pat. 6864, May 3, 1890. 6d.

See under X., page 811.

*Improvements in Combustible Coal Bricks.* J. J. Hiertz, St. Louis, U.S.A., and A. S. Garnett, Hot Springs, Ark., U.S.A. Eng. Pat. 6877, May 3, 1890. 6d.

SLACK, dust, or waste of lignite or brown coal is thoroughly dried, and to every ton of combustible is added a compost consisting of 1 bushel of quicklime, slaked and thoroughly incorporated with 7 galls. tar, 25 lb. salt, and 1 gall. of turpentine. The mixture is thoroughly worked together, heated to a temperature sufficient to cause the intimate fusion of all the ingredients, and submitted in hot moulds to a pressure of three tons per square inch.

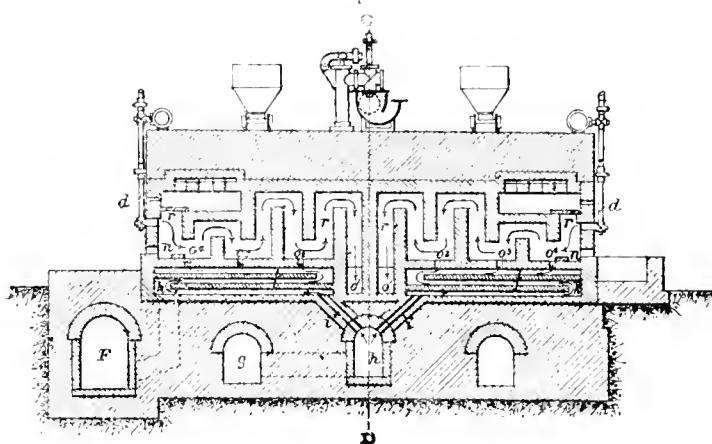
Bituminous coal may be similarly treated, a smaller proportion of tar and turpentine being used, with a higher temperature during the moulding. It is claimed that these bricks are very economical, and that having a glazed surface they are cleanly and impervious to moisture, moreover they burn with little smoke or ash.—S. B. A. A.

*Improvements in Coke-Ovens.* W. Fritsch, Zabrze, Germany.  
Eng. Pat. 13,390, August 24, 1889. 8d.

THE object of this invention is to facilitate the uniform heating and regulation of the temperature of the coke chambers in horizontal ovens. This is effected as follows:—The air required for combustion is supplied by a blower into side channels *g* in the basement of the oven, passes into a central channel *h*, and then through narrow inclined passages *i* into air-heating spaces *k* provided with horizontal baffle-walls *l*, and situated below the chamber walls between the channels *v* which carry away the products of combustion and serve to heat the bottoms of the coke chambers *p*. On leaving these spaces the air is caused to flow in a direction contrary to that of the gases, and is then admitted

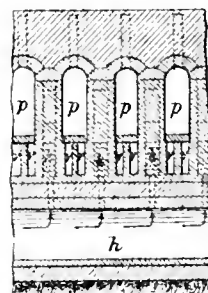
to the combustion chambers *r* through a number of openings progressively increasing in size  $o^1 o^2 o^3 o^4$ , the largest of which ( $o^4$ ) is situated nearest the outer end of the chambers where the gases are also introduced. It is controlled by a damper *n*, by means of which the intense combustion which takes place around the outer end of the coke chamber may be transferred to the interior. When the by-products are collected, the gases after condensation are introduced at both ends of the combustion chambers (*d*); otherwise the gases are directly conducted to the chambers at each end of the oven and thence past adjustable dampers into the combustion chambers. The latter are provided with vertical baffle-walls, and the products of combustion are drawn off through channels *v* into the main flue *F*.—S. B. A. A.

Fig. 1.



IMPROVEMENTS IN COKE OVENS.

Fig. 2.



SECTION THROUGH C D.

### III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

*The Testing of Light Petroleum.* R. Kissling. Chem. Zeit. 14, 508—509.

See under XXIII., page 824.

#### ERRATUM.

The article on the "Occurrence and Extraction of Ozokerite in Galicia," this Journal, 1890, 597, which is an abstract from the Allg. österr. Chemiker-und Tech. Zeit., should have been also credited to the Proc. Inst. Civ. Eng. 1890, Vol. C., 54.

### IV.—COLOURING MATTERS AND DYES.

*Researches on Diazo-compounds.* L. Gattermann. Ber. 23, 1218—1228.

See under XXIII., page 823.

*On the Application of Sandmeyer's Reaction to Diazo-sulphonic Acids and on the Decomposition of these Compounds in presence of Copper or Copper Oxide.* G. Tobins. Ber. 23, 1628—1634.

See under XXIII., page 824.

*Para-nitro-orthotoluene Sulphonic Acid.* J. Hausser. Bull. Soc. Chim. 1890, 797—799.

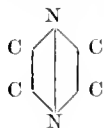
This acid was described by Beilstein and Kuhlberg (Ann. 155, 8), and some of its salts were prepared by Jensen (Ann. 172, 230). It may be prepared by gradually dissolving 200 grms. of paranitrotoluene in 260 grms. of sulphuric acid (44 per cent.  $SO_3$ ), the mass being kept cool at first but heated towards the finish to a temperature not exceeding  $150^\circ$ . The product is poured into water (1,500 grms.), heated, the sulphuric acid removed with powdered calcium carbonate and the filtrate concentrated and allowed to crystallise. The yield is about 64 per cent. The ammonium salt crystallises in long white anhydrous prisms and the sodium salt in prisms containing 2 mols. of  $H_2O$  which however it loses below  $130^\circ$ . Neither of these salts is affected by either strong or dilute sulphuric acid even at  $100^\circ$ . The sulphonic acid possesses very powerful acid properties, displacing sulphuric acid from soluble sulphates, even in the cold; a copper and a zinc sulphate were prepared in this manner. Nitrotoluene metasilphonic acid appears to possess similar properties to its isomeride.

—S. B. A. A.

*The Connection between Chemical Constitution and Colour.* F. Kehrman. Chem. Zeit. 14, 508, 527, 541—542.

THE introduction into the nucleus of an aromatic hydrocarbon of two carbonyl-groups in either the ortho- or para-position is accompanied by the appearance of a more or less intense yellow colour in the compound produced, the *o*-quinones being invariably of a deeper tint than the corresponding *p*-quinones, as is seen in the cases of  $\beta$ - and  $\alpha$ -naphthoquinone, phenanthraquinone and anthraquinone.

Again, the ordinary azo-compounds containing the group  $R-N=N-R$  stand to the azines of the type—



in the same relation as do *o*-quinones to *p*-quinones, the colours of these compounds confirming this view, for the azo-hydrocarbons are yellow or orange whilst the azines are pale yellow or colourless. The influence on the colour-property of quinone compounds due to the presence of positive or negative radicles in the molecule has been studied with respect to *p*-quinones solely, as very little is known regarding such derivatives of *o*-quinones. The following laws, consequently, refer to *p*-quinones.

I. The presence of alkyl-groups in the quinone molecule exercises no essential influence on the colour of the compound; compare, for instance, the colours of benzoquinone, toluquinone, thymoquinone,  $\alpha$ -naphthoquinone, anthraquinone.

II. Halogen-groups affect the colour in direct proportion to their atomic weights. Thus, the effect of iodine is well-marked, red-coloured compounds being formed, all the iodoquinones being of a deep red tint; bromine has a less powerful effect, orange-coloured bodies being produced as in the case of the two bromothymoquinones; chlorine, again, has very little colouring influence, mono-, di-, tri-, and tetra-chloroquinone being of the same hue as quinone itself. Although the influence of fluorine is unknown, it may be conjectured that its derivatives will be coloured similarly to, though perhaps paler than, the corresponding chlorine compounds.

III. Alkoxyl-groups have a reddening influence; methoxy-, ethoxy-, and dimethoxy-quinones have an orange colour; *p*-dichlorodimethoxyquinone and the corresponding ethoxy-compound a deep red.

IV. Less decided is the reddening effect of acetamido-groups, *m*-diacetamidoquinone being orange yellow and *p*-diacetamidoquinone orange.

V. Quinones containing nitro-, carbonyl-, sulphy-, cyano-, and other strongly negative radicles do not apparently exist, or at least are very unstable, except in the presence of hydroxyl- and amido-groups; thus, hydroquinone-carboxylic acid on oxidation decomposes into quinone and carbon dioxide and nitrohydroquinone cannot be oxidised to nitroquinone. Hydroxyquinones, on the other hand, can be nitrated or sulphonated, the effect of such operations being to change the colour of the compound from red to yellow, as is seen, for instance, in the cases of *p*-dihydroxyquinone and nitranilic and eithiochromic acids; the slightly negative acetyl-group shows a similar effect, *p*-diacetyl-tetrahydroxyquinone, for example, being yellower than *p*-dihydroxyquinone.

VI. The effect of the hydroxyl-group varies according as it is present in the quinone-molecule alone or in conjunction with other groups. The effect also changes regularly when the compound is converted into a salt, an ether, or an ester.

(a.) In general the presence of the hydroxyl-group intensifies the colour to red or violet, the particular tint depending upon the position of the substituting groups, and above all, on the number of such groups. A single OH-group effects only a slight, frequently inappreciable, deepening of the yellow hue, hydroxynaphthoquinone and hydroxythymoquinone, for example, are of the same colours respectively as  $\alpha$ -naphthoquinone and thymoquinone; *o*-hydroxyanthraquinone is orange-red, *m*-hydroxyanthraquinone yellow. Two hydroxyl-groups, however, produce a considerable deepening of colour, *p*-dihydroxyquinone being coloured deep orange-red,  $\beta$ - $\beta$ -dihydroxy- $\alpha$ -naphthoquinone brown-red, naphthazarin dark blood-red, alizarin orange. Still further increase in the number of hydroxyl-groups intensifies the tint to violet-black; thus, crystallised tetrahydroxyquinone is pitch-black, in powder violet.

(b.) The simultaneous presence of other groups in hydroxyquinones causes the effects on the colours of the resultant compounds stated below.

(1.) Alkyl-groups are without sensible influence on the colour of hydroxyquinones; *p*-dihydroxyquinone, *p*-dihydroxytoluquinone, and *p*-dihydroxythymoquinone are of almost the same tint. (2.) Halogen-groups exert a reddening action, proportionally stronger with increase of atomic weight; thus, *p*-dihydroxyquinone is orange-yellow, its monochloro-derivative orange, chloranilic acid scarlet, and iodoanilic acid deep red. (3.) Nitro- and sulphy-groups alter the tint from red to yellow; all the nitrohydroxyquinones, for instance, are yellow; nitranilic acid is golden-yellow, nitrohydroxy- $\alpha$ -naphthoquinone pale yellow, eithiochromic acid, as its name signifies, of a fine yellow colour. (4.) The acetamido-group has a slightly greater reddening action than the halogen-group; acetamidohydroxynaphthoquinone, for example, is of a redder yellow shade than chlorohydroxynaphthoquinone. (5.) The amido-group turns violet the hydroxyquinone which contains it; hydroxynaphthoquinone is golden-yellow, amidohydroxynaphthoquinone dark purple. (6.) The phenylazo-group masks the colouring influence of the quinone group; the compounds formed having the colour of hydroxyazobodies.

(c.) Respecting the effect of substitution of the hydroxyl-hydrogen, it is observed that whilst (1), etherification causes no particular change of tint, and (2) esterification renders paler the colour of hydroxyquinones, (3) the substitution of such hydrogen-atoms by metals causes a change of colour from yellow to deep red.

The power possessed by certain hydroxyquinones of dyeing on mordants depends on their property of forming coloured salts; thus, orange-coloured alizarin forms salts varying in colour from crimson to blue; the alkaline salts of purple amidonaphthalic acid are of an indigo-blue colour; scarlet chloranilic acid yields violet-red salts. Such hydroxyquinones as are chemically unacted upon by alkalis, dissolve in solutions of the same with colourations varying from blood-red to green.

VII.—Free amido-groups in quinone-compounds always produce a deepening in the colour of the same from yellow to red, brown, purple, or blue; thus *p*-diamidoquinone is purple, bromamidonaphthoquinone orange-red, the amido-anthraquinones blood-red to dark blue. Replacement of the hydrogen in the amido-group by alkyl- or phenyl-groups has no effect on the colour. Acetylation of the amido-group, and combination of the same with acids, change the tint from red to yellow, purple-coloured amidohydroxy- $\alpha$ -naphthoquinone yields with hydrochloric and sulphuric acids pale yellow-coloured salts of the tint of hydroxy- $\alpha$ -naphthoquinone.

The introduction of amido- or hydroxyl-groups into nitro-compounds results in a deepening of the colour of the same; the nitranilines, for example, are orange, whilst nitrobenzene is almost colourless; *p*-nitrotetrahydroxytoluene, again, forms a purple powder.

In general, if the atomic complex combined with the quinone-group be, on the whole, positive, the colour of the compound is red or violet, whereas, if it be negative, the colour of the compound is yellow.—E. B.

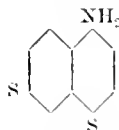
*Studies on the Constitution of the Tri-Derivatives of Naphthalene. No. 4. The Constitution of  $\alpha$ -Naphthylaminedisulphonic Acid* Dahl No. 11. *Naphthalene-1:2'-Disulphonic Acid*. H. E. Armstrong and W. P. Wynne. Proc. Chem. Soc. 1890 (86), 125—126.

WHEN naphthionic acid is sulphonated with 35 per cent. anhydrosulphuric acid at a temperature not exceeding 30° according to Dahl and Co.'s German patent No. 41,957, 1886, two disulphonic acids are produced, which are respectively known as  $\alpha$ -naphthylaminedisulphonic acid No. II. and No. III., the latter constituting the chief product. The constitution of acid No. III. has been given in a previous communication (this Journal, 1890, 283). With the object of comparing the disulphonic acids obtained by the sulphonation of  $\alpha$ -naphthylamine with those known to be produced by the sulphonation of  $\alpha$ -naphthol, the

authors have undertaken the investigation of acids Nos. I. and II. of Dahl and Co.'s patent, and are now in a position to communicate the results obtained by the examination of acid No. II.

The product was in the form of potassium salt, and on investigation was found to contain a noteworthy quantity, perhaps 20 per cent., of an  $\alpha$ -naphthylaminetrisulphonic acid, the formation of which, under the conditions above named, is specially interesting as throwing further light on the process involved in the production of isomeric disulphonic acids. When reduced by the hydrazine method, this  $\alpha$ -naphthylaminetrisulphonic acid yielded a *naphthalenetrisulphonic acid* characterised by forming a *chloride*,  $C_{10}H_7(SO_2Cl)_3$ , which crystallised from a mixture of benzene and petroleum spirit in small prisms melting at about  $191^\circ$ . By the Sandmeyer method it was converted into a *chloronaphthalenetrisulphonic acid* characterised by forming a *chloride*,  $C_{10}H_4Cl(SO_2Cl)_3$ , which crystallised from benzene in minute prisms from a mixture of benzene and petroleum spirit in small crystalline aggregates, and from acetic acid in small scales melting at  $215^\circ$ .

$\alpha$ -Naphthylaminedisulphonic acid No. II., on conversion into naphthalenedisulphonic acid by v. Baeyer's hydrazine method, yielded a new acid, the salts of which will be described in a subsequent communication. This acid was found to be the 1:2'-disulphonic acid, and is the *sixth* known naphthalenedisulphonic acid. It was proved that  $\alpha$ -naphthylaminedisulphonic acid Dahl No. II. is a sulphonated naphthionic acid, and has the constitution—

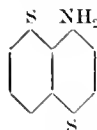


Dahl acid No. II.

The authors reserve the investigation of the properties of the 1:2'-naphthalenedisulphonic acid and its derivatives.

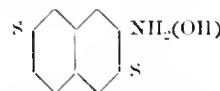
*No. 5. The Constitution of the Schöllkopf  $\alpha$ -Naphthylaminedisulphonic Acid.* H. E. Armstrong and W. P. Wynne. Proc. Chem. Soc. 1890 (86), 126—127.

THE formation of the Schöllkopf  $\alpha$ -naphthylaminedisulphonic acid (German Patent 40,571) by the nitration and subsequent reduction of the authors' naphthalene-1:4'-disulphonic acid (Bernthsen, this Journal, 1890, 491) renders it evident that the generally accepted view of the constitution of this acid is a correct one. Having been favoured by the Actiengesellschaft für Anilinfabrikation with a supply of the material prepared by the method given in the Schöllkopf patent, the authors took occasion to submit this to examination. They found that it was converted by the hydrazine method into naphthalene-1:4'-disulphonic acid, which was characterised by conversion into the chloride crystallising from benzene in prisms melting at  $182^\circ$ , and into 1:4'-dichloronaphthalene melting at  $107^\circ$ . On treatment by the Sandmeyer process it gave a chlorodisulphonic acid, the *chloride* of which crystallised from acetic acid in very small prismatic needles which became opaque on drying, and from a mixture of petroleum spirit and benzene in sparingly-soluble glistening flat plates. It melted at  $135^\circ$ , and on distillation with  $PCl_5$  was converted into  $\delta$ - or 1:4:1'-trichloronaphthalene melting at  $131^\circ$ . These results place it beyond question that the constitution of the Schöllkopf acid is expressed by the formula—



*No. 6. The Constitution of Cassella's  $\beta$ -naphthylamine- $\delta$ -disulphonic Acid.* H. E. Armstrong and W. P. Wynne. Proc. Chem. Soc. 1890 (86), 127—128.

MESSRS. CASSELLA and Co. having been good enough to furnish the authors with a supply of their  $\beta$ -naphthylamine- $\delta$ -disulphonic acid, prepared from the  $\beta$ -naphtholdisulphonic acid of their German Patent No. 44,079, it has been possible to determine its constitution by the hydrazine and Sandmeyer methods already described. On treatment by the hydrazine method, the amido-acid yields, in the first instance, a peculiar rosy, gelatinous, hydrazine, which can only be freed from tin salts with considerable difficulty. The disulphonic acid obtained from this hydrazine yields a chloride crystallising from benzene, in which it is sparingly soluble, in small, flat spear-like needles. This chloride melts at  $225^\circ$ , and on distillation with  $PCl_5$  yields 2:3'-dichloronaphthalene melting at  $135^\circ$ ; it is, therefore, naphthalene-2:3'-disulphonic acid—the  $\beta$ -disulphonic acid of Ebert and Merz. By the Sandmeyer method the amido-acid is converted into a chlorodisulphonic acid, the *chloride* of which crystallises from benzene, in which it is very soluble, in small, characteristic radiate groups of very slender needles, and from a mixture of petroleum spirit and benzene in small, opaque aggregates melting at  $176^\circ$ . On distillation with  $PCl_5$  it yields 2:3:2'-trichloronaphthalene melting at  $90^\circ$ . Combining these results, it follows that Cassella's  $\beta$ -naphthylamine- $\delta$ -disulphonic acid and the  $\beta$ -naphtholdisulphonic acid of the German Patent No. 44,097, from which it is derived, have the formula—



and both are, therefore, tri- $\delta$ -acids isomeric with the corresponding R-acids (this Journal, 1890, 283).

*No. 7. The Disulphonic Acids obtained by Sulphonating the Isomeric Heteronuclear  $\beta$ -Naphthylaminesulphonic Acids.* (First notice.) H. E. Armstrong and W. P. Wynne. Proc. Chem. Soc. 1890 (86), 128—131.

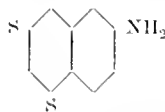
ACCORDING to Gans and Co.'s German Patent, No. 35,019, 1884,  $\beta$ -naphthylamine is converted into the amido-G-disulphonic acid by heating its sulphate with thrice its weight of 20—30 per cent anhydrosulphuric acid at  $110^\circ$ — $140^\circ$  until soluble in water; and the same product is said to result when " $\beta$ -naphthylaminesulphonic acid" is similarly treated. But inasmuch as four monosulphonic acids are obtainable from  $\beta$ -naphthylamine, to determine the law of sulphonation, it was obviously necessary to examine the behaviour of each of these, and to avoid the occurrence of secondary changes as far as possible. Experiments with this object in view were commenced several years ago. Meanwhile, an account of the behaviour of the Brönner acid under conditions similar to those specified by Gans and Co., have been published by Forsling (*Ber.* 1888, 3496).

(1.) 2:1'- $\beta$ -naphthylamine- $\alpha$ -sulphonic Acid (*Badische Acid*).—When the *Badische* acid is stirred into four times its weight of 20 per cent. anhydrosulphuric acid at a temperature not exceeding  $20^\circ$ , and the mixture is allowed to stand, sulphonation proceeds slowly, and is not entirely complete even at the end of three months. The product is found to consist almost entirely of an acid recognised as amido-G-disulphonic acid, inasmuch as it gave by the hydrazine and Sandmeyer methods all the products already described as characteristic of this acid (this Journal, 1890, 283). The naphthalenedisulphochloride prepared from it melted at  $137^\circ$ , and on distillation with  $PCl_5$  gave 1:3-dichloronaphthalene melting at  $61.5^\circ$ . The chloronaphthalenedisulphochloride from it melted at  $169^\circ$ , and on distillation with  $PCl_5$  gave 1:3:2'-trichloronaphthalene melting at  $113^\circ$ .

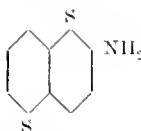
(2.) 2:4'- $\beta$ -naphthylamine- $\alpha$ -sulphonic Acid (*Dahl Acid*).—When stirred into four times its weight of 20 per cent. anhydrosulphuric acid at a temperature not exceeding

20°, the Dahl acid gradually undergoes further sulphonation, the process being usually completed in from 116—120 hours. The product is found to consist of two isomeric acids, which are readily separated by crystallising out the minor product as normal potassium salt, and subsequently purifying the chief product by repeated crystallisation of its acid potassium salt.

**Chief Product.**—The acid potassium salt of the chief product on reduction by the hydrazine method gave naphthalene-1:3-disulphonic acid, and was proved to have the constitution—

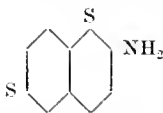


**Minor Product.**—The normal potassium salt of this acid on distillation with  $\text{PCl}_5$  is converted into 1:2:4'-trichloronaphthalene, crystallising from alcohol in long, slender, flat needles melting at 78°—78.5° (Proc. Chem. Soc. 1889, 49). It follows, therefore, that this amido-acid has the constitution—



(3.) **2:3'-β-Naphthylamine-β-sulphonic Acid (Brønner Acid).**—When stirred into four times its weight of 20 per cent. anhydrosulphuric acid at a temperature not exceeding 20°, the Brønner acid is readily further sulphonated, the process being completed in from 16—20 hours. The product is found to consist of two isomeric acids; the minor product, perhaps 20 per cent., has been identified as G-amido-disulphonic acid by the hydrazine and Sandmeyer methods.

**Chief Product.**—The acid potassium salt of this acid crystallises in opaque, white radiate aggregates of short needles, difficultly soluble in water; but the crystalline form and solubility are much influenced by the presence of small quantities of amido-G-acid, and there is every reason to regard it as identical with the acid obtained by Forsling (Ber. 1888, 3496) by heating Brønner acid with 3 to 4 parts of "fuming sulphuric acid" (percentage of added  $\text{SO}_3$  not stated) at 110° until the product was soluble in water. When reduced by the hydrazine process, the amido-acid constituting the chief product gave naphthalene-1:3'-disulphonic acid. On treatment according to the Sandmeyer method, it was converted into a chlorodisulphonic acid, which on distillation with  $\text{PCl}_5$  gave 1:2:3'-trichloronaphthalene. Combining these results, the amido-acid constituting the chief product, and presumably Forsling's acid, has the constitution—



By operating at higher temperatures the yield of this acid is diminished, and that of the amido-G-acid increased.

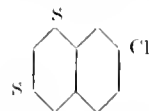
(4.) **2:2'-β-Naphthylamine-β-sulphonic Acid (Bayer and Duisberg's delta-acid).**—When stirred into four times its weight of 20 per cent. anhydrosulphuric acid at a temperature not exceeding 25°, delta-acid yields a complex product, which has not yet been satisfactorily separated into its constituents.

**No. 8. β-Chloronaphthalenedisulphonic Acids.** (First notice.) H. E. Armstrong and W. P. Wynne. Proc. Chem. Soc. 1890 (86), 131—133.

As already indicated in a previous communication, the study of the disulphonic acids obtained by sulphonating chloronaphthalenesulphonic acids of known constitution is

being carried on *pari passu* with that of the acids derived from the various naphthylaminesulphonic acids (this Journal, 1890, 284). A brief account of the results obtained with the four heteronuclear β-chloronaphthalenesulphonic acids under like conditions is now given, details with reference to the composition of the salts, &c. being reserved for a full communication. Sulphonation was effected by adding the theoretical quantity of sulphuric anhydride, employed in the form of 20 per cent. anhydrosulphuric acid, to the dry potassium β-chloronaphthalenesulphonate and heating the warm mixture at 100° for an hour, the potassium sulphate formed being removed by treatment with alcohol and the excess of sulphuric acid by means of barium carbonate.

(1.) **2:1'-β-Chloronaphthalene-α-sulphonic Acid.**—This acid, under the conditions named, gives a uniform chloronaphthalenedisulphonic acid, the *chloride*,  $\text{C}_{10}\text{H}_7\text{Cl}(\text{SO}_2\text{Cl})_2$ , of which crystallises from benzene, in which it is comparatively sparingly soluble in beautiful radiate groups of long, slender, lustrous needles. It melts at 170°, and on distillation with  $\text{PCl}_5$  gives 1:3:2'-trichloronaphthalene melting at 113°. The constitution of the acid is therefore—

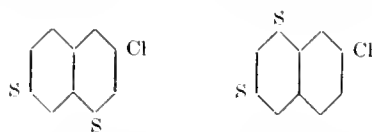


and corresponds with the G-amido-acid obtainable from the 2:1'-β-naphthylamine-α-sulphonic acid.

(2.) **2:4'-β-Chloronaphthalene-α-sulphonic Acid.**—This acid gives what seems to be a uniform product. The potassium salt of the chlorodisulphonic acid is particularly well characterised, since it crystallises in prismatic forms, whereas the corresponding salts of the isomeric acids crystallise in slender needles. The *chloride*,  $\text{C}_{10}\text{H}_7\text{Cl}(\text{SO}_2\text{Cl})_2$ , is apparently dimorphous, and crystallises from benzene for the most part in very lustrous scales, but occasionally in prismatic forms, both of which melt at 156°—156.5°, and on distillation with  $\text{PCl}_5$  give the same trichloronaphthalene melting at 79°. This trichloronaphthalene has not yet been compared by sulphonation, &c. with the 1:2:4'- and the 1:3:3'-trichloronaphthalenes, with one of which it must be identical, owing to the insufficient supply of material; most probably it has the constitution 1:3:3'- and corresponds with that derived from the amido-acid constituting the chief product of the further sulphonation of the 2:4'-β-naphthylamine-α-sulphonic acid.

(3.) **2:3'-β-Chloronaphthalene-β-sulphonic Acid.**—Under the conditions named, this acid gives two chlorodisulphonic acids which are best separated by the fractional crystallisation of the potassium salts. The less soluble potassium salt constituting the minor product yields a *chloride*,  $\text{C}_{10}\text{H}_7\text{Cl}(\text{SO}_2\text{Cl})_2$ , which crystallises from benzene in very slender needles aggregated in prismatic forms, having a silky lustre; it melts at 148°, and on distillation with  $\text{PCl}_5$  gives 1:3:2'-trichloronaphthalene, crystallising from alcohol in characteristic tufts of needles melting at 113°.

The more soluble potassium salt constituting the major product was found to give products identical with those obtained from the chlorodisulphonic acid derived from 2:1'-β-chloronaphthalene-α-sulphonic acid (*vide supra*). The *chloride* crystallises from benzene in radiate groups of needles melting at 169°, and on distillation with  $\text{PCl}_5$  yields 1:3:2'-trichloronaphthalene melting at 113°. The constitution of the two acids is represented by the formulae—

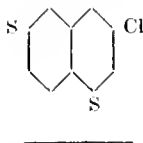


Minor product.  $\text{Cl}(\text{SO}_2\text{Cl})_2$ , m.p. = 147°. Major product.  $\text{Cl}(\text{SO}_2\text{Cl})_2$ , m.p. = 169°.

(4.) **2:2'-β-Chloronaphthalene-β-sulphonic Acid.**—This acid, under the above conditions, yields a uniform chlorodisulphonic acid, the *chloride* of which,  $\text{C}_{10}\text{H}_7\text{Cl}(\text{SO}_2\text{Cl})_2$ ,

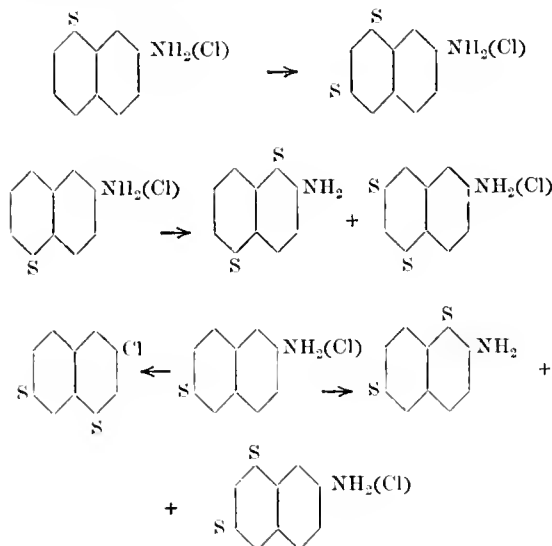


crystallises in beautiful radiate groups of flat, prismatic needles, melting at  $174^{\circ}$ . On distillation with  $\text{PCl}_5$ , the chloride is converted into 1 : 3 : 3'-trichloronaphthalene, crystallising from alcohol in radiate groups of long, slender needles, melting at  $80.5^{\circ}$ . The acid has, therefore, the constitution—



*The Comparative Influence Exercised by the Radicles Cl, OH, and  $\text{NH}_2$  in Naphthalene Derivatives on the Formation of Disulphonic Acids.* II. E. Armstrong and W. P. Wynne. Proc. Chem. Soc. 1890 (86), 133—136.

THE results obtained by the comparative study of the influence exercised by radicles such as Cl, OH, and  $\text{NH}_2$  on the formation of disulphonic acids, of which an account has been given in the foregoing notes and in previous communications by the authors, are such as to throw much light on the laws which govern substitution in the naphthalene series and are moreover highly suggestive. The results obtained in the case of the chloro- and amido-acids are collected in the following diagram :—



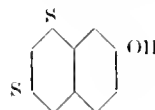
One fact to which attention may be directed, which appears to be most clearly established by the investigation of a very large number of disulphonic acids, is the "invincible objection" of two sulphonic radicles to remain in either contiguous or para- or peri-positions. The expression "remain in" is used advisedly, as the authors believe that, inasmuch as action more often than not takes place in the first instance in accordance with the so-called  $\alpha$ -law to which they have repeatedly directed attention, such positions are sometimes initially assumed by two sulphonic groups. It is a question whether the final acquisition of other positions than  $\alpha$ -positions is a consequence of direct isomeric change, or of the formation of higher sulphonic acids and their subsequent partial hydrolysis.

Different radicles undoubtedly exercise a very different and most important influence in determining such secondary changes. Thus  $\beta$ -naphthol on treatment with sulphonating agents is converted into a sulphate which very readily undergoes isomeric change into the 2 : 3'-sulphonic acid;  $\beta$ -naphthylamine sulphate undergoes a similar change, but much less readily, being converted into the 2 : 3'-sulphonic acid only after long heating at a high temperature: in this latter case the formation of the sulphamic acid probably precedes that of the sulphonic acid, and either the

sulphamic acid is formed with difficulty from the sulphate, or, when formed, does not readily undergo isomeric change.

$\alpha$ -Naphthylamine and  $\alpha$ -naphthol differ in a similar manner.

When excess of sulphuric acid is used, opportunity is given for sulphonation to pursue a different course: the characteristic properties of the phenol and amine are, in a sense, obliterated, owing to the formation of sulphates, and these sulphates behave much as do corresponding derivatives containing a neutral radicle-chlorine, for example. Then, if  $\beta$ -naphthyl sulphate be sulphonated by  $\text{SO}_3\text{HCl}$  without application of heat, a disulphonic acid is at once obtained, which, there is reason to believe, has the constitution—



(Ber. 15, 204); the formation of this compound affords the most significant indication of the course of change; evidently the homo- $\alpha$ -hydrogen atom contiguous to the  $\text{O}.\text{SO}_3\text{H}$ -group of the sulphate is first displaced; the resulting sulpho-sulphate, however, spontaneously undergoes isomeric change.  $\beta$ -naphthylamine sulphate, apparently, behaves somewhat differently, yielding the 2 : 1'- and 2 : 4'-monosulphonic acids; in this case the  $\text{NH}_2.\text{H}_2\text{SO}_4$  group may be supposed to remain unchanged; but it is not improbable that the homo- $\alpha$ -hydrogen atom contiguous to this group is primarily displaced by  $\text{SO}_3\text{H}$ , which then passes spontaneously into the contiguous peri- $\alpha$ -position, giving rise to the 2 : 1'-acid; the formation from the 2 : 3'- and 2 : 4'-monosulphonic acids of disulphonic acids containing the  $\text{SO}_3\text{H}$ -group in the homo- $\alpha$ -position contiguous to the  $\text{NH}_2$  affords the strongest support to this view. The formation of the 2 : 4'-monosulphonic acid from  $\beta$ -naphthylamine sulphate is perhaps the outcome of an independent action.

In the case of  $\beta$ -chloronaphthalene, it is conceivable that the 2 : 1-sulphonic acid is the immediate product, and that this spontaneously changes into the 2 : 1'-acid which is actually obtained as chief product; the 2 : 3'-acid which is obtained as subsidiary product may be formed by spontaneous isomeric change from 2 : 4'-acid initially produced on sulphonation rather than by the isomeric change of the 2 : 1'-acid at the moment of formation; this view being supported by the fact that  $\beta$ -iodonaphthalene affords a small proportion of 2 : 4'-acid (Proc. Chem. Soc. 1889, 120).

In short, it is not impossible that the  $\beta$ -mono-derivatives all behave similarly on sulphonation, acting in a minor degree as naphthalene itself would, but chiefly as mono-derivatives; and that the differences in the structure of the ultimate products are due to the different manner in which secondary change takes place under the diverse influences of various  $\beta$ -radicles. The same argument would apply to  $\alpha$ -compounds.

The peculiar differences manifest in the case of monosulphonic compounds are also to be noted in the case of disulphonic (see diagram)—the product of further sulphonation is, as a rule, simpler in the case of the chloro-acid than in the case of the amido-acid; and change proceeds further in the case of the hydroxy- than in that of the  $\text{NH}_2$ -compound, as is evidenced by the formation of R- together with G-disulphohydroxy acid under conditions which do not give rise to R-amidodisulphonic acid.

In the case of the 2 : 1'-amidodisulphonic acid, the homo- $\alpha$ -position contiguous to the  $\text{NH}_2$  is, perhaps, screened from attack; and either a 1 : 4-disulphonic acid is first formed, or, under the dehydrating influence of the sulphuric acid present in excess, a sulphamic-sulphonic acid is gradually produced, which then undergoes isomeric change: it appears not improbable, from the exceptionally slow manner in which sulphonation takes place, that the latter is the correct explanation.

The remaining three isomeric  $\beta$ -amidodisulphonic acids, in all probability, are at an early stage—for it is possible that a sulphamic acid is the first product of sulphonation—if not initially converted into 2 : 1-homosulpho-derivatives; but it is a question whether these then undergo a simpler isomeric

change, or whether the final products are formed from them by their conversion into higher sulphonie acids which then suffer hydrolysis. The behaviour of 1:2-chloro- $\beta$ -naphthylamine on sulphonation (Proc. Chem. Soc. 1889, 36, 48) appears rather to support this latter view, as the passage of the sulphy-group through the positions  $1 \rightarrow 2 \rightarrow 3$  takes place with a much greater facility than is usual in cases of non-spontaneous isomeric change. It is true that the conditions under which sulphonation is effected are such that no water is present; but, bearing in mind the tendency of  $H_2SO_4$  to combine with  $SO_3$ , it is by no means improbable that  $H_2SO_4$  itself may effect the hydrolysis.

Lastly, attention may be called to the occurrence of "homo-sulphonation" in the case of the two chloro- $\beta$ -sulphonie acids; the products in question are, perhaps, direct sulphonation-products, but it is also conceivable that the 2:1-chlorosulphonie acid is first formed, and that this undergoes isomeric change to the 2:4-acid, in consequence of the guarding influence exercised by the hetero- $\beta$ -sulphonie radicle.

The results obtained in the case of the Dahl No. 11. acid are of special interest, as one of the sulphy-groups is in the  $\beta$ -position alternative to that occupied in the acid which yields naphthol-yellow S; it is, hence, possible that, in the first instance, naphthionie acid yields a peri-disulphonie acid, and not the Schöllkopf acid.

The solution of these various problems will necessitate much further study; but obviously their settlement is of importance in relation to the theory of the formation of substitution-derivatives generally.

#### *Note on the Action of Potash on Naphthalene-1:3-Di-sulphonic Acid.* H. E. Armstrong and W. P. Wynne.

WHEN the authors' naphthalene-meta-disulphonic acid (this Journal, 1890, 283) is fused with 3—4 times its weight of caustic potash at  $280^\circ$ — $300^\circ$  for several hours, it gives a product which consists chiefly of a trihydroxynaphthalene,  $C_{10}H_5(OH)_3$ . This crystallises from water in minute scales, from light petroleum (b. p. =  $30^\circ$ ) in small, white aggregates of no definite form, and melts at  $120^\circ$ — $121^\circ$ . It sublimes in lustrous, thin scales; is extremely soluble in ether, chloroform, carbon bisulphide, acetone, and benzene; and gives no characteristic colouration with ferric chloride. The study of the action of potash on naphthalene-meta-disulphonic acid is being continued.

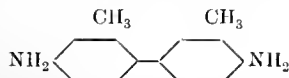
#### PATENTS.

*Improvements in and Relating to Colouring Matters.* H. H. Leigh, London. From R. G. Williams, Albany, U.S.A. Eng. Pat. 4565, March 15, 1889. (Reprint.) 6d.

THIS specification has already been abstracted (see this Journal, 1889, 611). It has been reprinted in consequence of an amendment made by order of the Comptroller prior to the sealing of the patent to the effect that the patentee is "aware that it has been previously proposed to use phenols or their sulphy acids in the production of mixed azo colours."—T. A. L.

*The Production of a New Colouring Matter suitable for Dyeing and Printing Cotton without the Aid of a Mordant.* J. Y. Johnson, London. From the "Badische Anilin und Soda Fabrik," Ludwigshafen, Germany. Eng. Pat. 13,558, August 27, 1889. 6d.

HITHERTO it was generally believed that meta substitution products of benzidine were incapable of yielding direct cotton dyestuffs. The present patent describes the preparation of colouring matters of this class which dye unmordanted cotton. The base employed is that known as *o-m*-tolidine, having the formula—



(Schultz, Ber. 17, 470; this Journal, 1884, 245). The most important dyestuff obtained from it is formed by combining its tetrazo compound with naphthionie acid. To prepare the dyestuff, 10 parts of *o-m*-tolidine sulphate are suspended in 300 litres of water and 18.5 kilos. of hydrochloric acid containing 25 per cent. of HCl and diazotised with 4.5 kilos. of sodium nitrite containing 96 per cent. of  $NaNO_2$ , the solution during the operation being kept at  $0^\circ$ . The diazo solution is then poured into 450 litres of water in which 22.5 kilos. of sodium naphthionate and 20 kilos. of sodium acetate are dissolved. After stirring for about an hour, 9 kilos. of calcined soda are added and the whole stirred for three or four days. The colouring matter is then salted out and can be purified by dissolving in water and reprecipitating with salt.—T. A. L.

#### *Production of Azo-Colouring Matters from Diary-naphthalenemono-sulphonates.* O. Murray, London.

From "The Farbwerke vormals Meister, Lucius and Brüning," Höchst-on-the-Maine, Germany. Eng. Pat. 13,710, August 30, 1889. 4d.

A PROCESS for obtaining substantive colouring matters from the dihydroxynaphthalenemono-sulphonates, the preparation of which was described in Eng. Pat. 9642 of 1889 (this Journal, 1890, 609) by combining them with the following tetrazo compounds:—Tetrazodiphenyl, tetrazoditoly, tetrazodimethoxydiphenyl, tetrazodietoxydiphenyl, tetrazostilbene, tetrazostilbene-disulphonic acid. The reaction takes place in two stages, an intermediate product containing one molecule of the tetrazo compound with one molecule of the dihydroxynaphthalene-sulphonate being first obtained. This can be then further combined with another molecule of the dihydroxynaphthalene-sulphonate or with any other combinable body such as a phenol, amine, or sulphonie acid thereof.—T. A. L.

#### V.—TEXTILES: COTTON, WOOL, SILK, Etc.

##### PATENT.

*Improvements in the Treatment of Woollen, Worsted, Buck-worsted Unions, and other Woven Fabrics.* I. Smith, Batley. Eng. Pat. 14,751, September 19, 1889. 6d.

THE object of this invention "is the superseding of crabbing and steam blowing of the above-mentioned goods, and consists in a method of imparting solidity, softness, and a permanent finish to them, and securing a preparation of evenness necessary for dyeing." The fabric is passed through an apparatus known as a "weegee," consisting of a trough or cistern with two rollers near the bottom, whilst two other rollers are so arranged above the trough that they can be made to rotate alternately in contrary directions. The trough is filled with boiling water or other boiling liquid, and the fabric is placed therein, passing over one of the upper rollers, beneath the lower ones and up to the other top roller. A backward and forward movement is given to the fabric for a suitable time, according to the nature of the fabric. The required solidity, finish, and softness are hereby better attained than by the old process.

—H. S.



## VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

*Azo-Dyestuffs which Dye on the Chrome Mordant.*  
R. Lepetit. Chem. Zeit. 14, 507—508.

SINCE the introduction into commerce of Nietzki's alizarin-yellow (nitraniline-azo-salicylic acid), which is fixed on wool by the aid of the chrome mordant, numerous experiments have been made, not only to discover similar new adjective dyestuffs, but to ascertain whether any of the known azo-dyes have this property of combining with chrome. As a consequence, it has been found that rocellin, amongst older dyes, has the power of forming a weak combination, but cloth red in its various shades, and some still newer dyes, apparently of the azo-class, manufactured by the Farbenfabr. vormals F. Bayer and Co., although capable of dyeing unmordanted wool, yield colours considerably faster to light and milling when that fibre has been previously mordanted with chrome. The shades produced, however, are the same, or nearly so, as when the mordant is absent.

Instead of mordanting with chrome and then dyeing in the way usual with these colouring matters, the author prefers to dye the wool first with the assistance of sodium sulphate and oxalic acid and then, without rinsing, to boil half an hour with 2.5—3 per cent. of potassium bichromate. The shades produced in this way are darker than when the wool is first mordanted and then dyed.—E. B.

*Bleaching Wool and Silk by Sodium Hydrosulphite.*  
G. Dommergue. Monit. Scient. 1890, 4, 684—685.

SULPHUROUS acid is commonly used for bleaching silk and wool. Its use is open to several objections, and the author recommends that sodium hydrosulphite be substituted for it, as he finds that this fulfils the purpose perfectly. It is prepared in the usual way by reducing sodium sulphite by zinc, and as the solution quickly oxidises it should be prepared only when required for immediate use. The well washed material is immersed in the solution for six hours; it must then be carefully pressed and rinsed in distilled water out of contact with the air, otherwise heating takes place and the material is damaged. If the pieces be marked (due to the double sulphite of zinc and sodium) they should be passed through a bath of dilute hydrochloric acid, and then well washed. With a tank holding 1,000 litres, 130 kilos. of wool can be treated each time, and the cost would be 1 fr. 36 c. for each 10 kilos. of wool.—A. L. S.

### PATENTS.

*Improvements in the Process of Bleaching Fibrous Material by the Aid of Electricity.* C. Kellner, Vienna, Austria. Eng. Pat. 5054, April 1, 1890. 6d.

THE fibrous material is impregnated with a solution which, when electrolysed, has the power of bleaching it. The electrodes are preferably in the form of a series of revolving rollers, which draw the fibrous material along, while at the same time they act as conductors for the current. Sometimes the fibrous material is enclosed between two layers of felt, one impregnated with the anion and the other with the cathion, the whole being drawn between the rollers. Unused chlorine is absorbed by such materials as lime, magoesia, &c., placed in the electrolyte. The top rollers are alternately positive and negative, the lower ones being, of course, of opposite polarity to those just above.—E. T.

*Improvements in the Process of and Apparatus for Bleaching Vegetable Fibres.* C. Kellner, Vienna, Austria. Eng. Pat. 5285, April 5, 1890. 8d.

THIS is a process for bleaching by the aid of electrolysis, and is based on the "discovery" that the colouring matters

to be destroyed are easily converted into compounds soluble in water by alternate treatment with chlorine (or compounds containing active chlorine) and alkalis. This action is ascribed to the fact that chlorine, before it converts the colouring matter into compounds soluble in water, forms an intermediate product which, though insoluble in water, is easily soluble in alkalis. As a consequence, by the alternate action of chlorine and alkali, the process is shortened and less chlorine is required. In carrying out the invention a chloride of an alkali is decomposed by electrolysis and the anion and cathion are caused alternately to pass through the fibrous material requiring to be bleached. Afterwards both ions are again united and conveyed back into the electrolysis apparatus. Three forms of apparatus are described for carrying out this process, for the construction of which it is necessary that the drawings accompanying the specification should be consulted.—W. E. K.

*Improvements in or Applicable to Rollers and Blocks used in Printing Wall Papers and the like.* W. H. Clegg and G. Kirby, London. Eng. Pat. 10,039, June 19, 1889. 6d.

THE purpose of this invention is to lessen the cost of producing the rollers and blocks used in printing wall papers, &c. The usual practice is to build up the pattern of metallic pins and strips and then fill in the flat surfaces with felt. By this invention a pliable medium is applied to the whole surface of the roller, of a thickness equal to the usual projection of the metallic pins, and after transferring the pattern to the roller that part of the medium is cut away where no pattern is required. Lines, dots and outlines are put in by strips and pins as usual. After the pattern is cut the medium may be hardened by suitable means. The following substances are given as suitable media: compounds of powdered cork and oxidised linseed oil, &c. (as used in the manufacture of linoleum and "Lincrusta Walton") and the substance known as "fibrous sheet." Where only a small part of the roller is used for printing the medium is affixed in sections as required.—W. E. K.

*Improvements in the Dyeing, Scouring, or like Treatment of Yarn or other Fibrous Materials, and in Apparatus therefor.* E. Maertens, Providence, Rhode Island, U.S.A. Eng. Pat. 10,360, June 25, 1889. 1s. 5d.

"YARN wound into cops or balls, or on spools or bobbins, yarns in the skein or chain, combed tops, cardings, and even raw material, and certain cloths" are treated in an apparatus, which is fully described and sketched in the original specification, "the form, shape, and size of the receptacle for holding the materials to be treated being adapted to the form, shape, and bulk of such materials." Perforated cop tubes are used or unperforated tubes covered, previously to winding the yarn thereon, by loosely braided or woven tubes; in the latter case the ordinary paper cop tubes of the spinner may be employed.—E. B.

*Improvements in Indigo Printing.* J. V. Hulme, Manchester, and J. A. Walton, London. Eng. Pat. 10,635, July 1, 1889. 4d.

INDIGO, finely ground, is thoroughly reduced by means of caustic soda and zinc powder and the mixture thickened with gum. (The use of bisulphite of soda in addition is described in the provisional, but omitted from the final specification.) The mixture is printed on unprepared cloth, run over rollers in air to partially oxidise the indigo, then through a water spray, and finally through water boxes. It is afterwards soured, soaped, washed, and dried.

—W. E. K.

*Improvements in the Manufacture of Tannin Compounds, more especially intended for Printing Purposes.* B. Willeox, London. From "The Farbenfabriken vorm. F. Bayer and Co.," Elberfeld, Germany. Eng. Pat. 10,993, July 8, 1889. 4d.

By heating tannic acid with glycerin or grape sugar to 140° to 160° C. combinations are formed which are readily soluble in water and dilute acetic acid, and which possess the property of being decomposed into their constituent parts during the process of steaming to which textile prints are subjected. These products are introduced under the names of "tannin-glycoside" and "tannin-glyceride," as substitutes for the tannic acid used for the fixation of basic colouring matters in calico printing; it being claimed that the tannin is brought into action in a nascent condition during the steaming process.—W. E. K.

*Improvements in and connected with Apparatus for Dyeing, Washing, and otherwise similarly Treating Yarn in Hanks.* T. O. Arnfield, New Mills. Eng. Pat. 11,098, July 10, 1889. 8d.

This invention relates to that type of apparatus in which a concentric series of tanks have rotating above them a concentric series of pairs of rollers from which the hanks are suspended. The purpose of the invention is to provide facilities (a) for raising the hanks out of the liquors to facilitate their removal and replacement, and (b) for raising the hanks over the edges of the divisions which separate the several vats. To effect the former purpose the top roller of each pair is hung from a lever which has mounted on one end an anti-friction roller arranged to run on a circular track. At the necessary points on this track are double inclined planes, connected by a level higher platform; the motion of the roller up and down the planes produces the required lift and fall in the rollers carrying the hanks, whilst on stopping the machine when the rollers are on the higher platform the removal and replacement of the hanks is readily effected. To ensure the necessary lift from vat to vat of the bottom winches round which the yarn passes, these are connected by a lever to a stud fixed on the revolving frame. This lever is guided by a path formed on the inner edge of the vat, and by a ramp or lift in the path at the necessary points the lower end of the lever and with it the winches are lifted over the divisions which separate the vats. The invention also includes an automatic guard which prevents the yarns running off the bottom rollers.—W. E. K.

*Improvements in Machinery for the Continuous Bleaching, Soaping, Blueing, Washing, Finishing, Sizing, Dyeing, or otherwise analogously Treating in the Hank, of Cotton, Linen, Woollen, Silk, or other Yarns, Tapes, and similar Manufactures.* G. B. Sharples, Manchester. Eng. Pat. 11,382, July 16, 1889. 6d.

THREE rollers are placed parallel to each other at a suitable height above a tank or series of tanks. Two of the rollers are fixed a little distance apart and revolve in the same direction, the other being sustained and revolved by them. The latter roller has one end cone-shaped to facilitate the passing of the hanks on to it, the hanks, as the roller rotates, being carried by traverse hooks to the other end of the roller and then taken off.—E. B.

*Improvements in and relating to Machinery for Printing partially in Colours.* A. Fayol, Bordeaux. Eng. Pat. 12,896, August 15, 1889. 11d.

THE purpose of this invention is to print newspapers, &c. in part in coloured ink, with the view of enhancing the money value of the advertisements so printed. For the mechanical means by which this is effected, in the case both of the flat and of the rotary press, the specification and accompanying 25 drawings must be consulted.—W. E. K.

*A New Liquid or Solid Product applicable for Removing Grease from, Washing, Lixiviating, and Bleaching all Textile Materials, Cloths, Rags, and similar Materials, for the Fulling of Cloths, and for removing Hair from Skins and similar Operations.* J. L. Moret, Paris, France. Eng. Pat. 13,023, August 17, 1889. 6d.

THE product claimed is a mixture of caustic soda and hydrosulphide of soda (NaHS) in equal proportions. The caustic soda may be replaced by carbonate containing a certain proportion of caustic soda, and the hydrosulphide by monosulphide of sodium prepared by dry means. The material to be treated is subjected to a cold or hot bath containing the dissolved substance. If it is desired to increase the bleaching efficiency of the bath, a small quantity of sulphurous acid is added; or the material may be subsequently passed into a second bath of water acidulated with sulphurous acid.—W. E. K.

*Improved Process of applying Luminous Preparation to the Surface of Paper or other Substances.* J. A. Causton and J. Challis, London. Eng. Pat. 14,508, September 14, 1889. 4d.

To produce luminous designs the pattern is printed in any suitable way with an adhesive composition, and the luminous powder dusted or flocked over it whilst still wet or tacky. To obtain a greater body of luminous powder, a portion may also be mixed with the mucilage used in printing the design.—W. E. K.

## VII.—ACIDS, ALKALIS, AND SALTS.

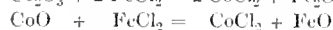
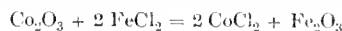
*On some New Double Chromates.* M. Lachaud and C. Lepierre. *Compt. Rend.* **110**, 1035-1038.

By fusing potassium nitrate with lead chromate the authors have obtained three compounds, *i.e.*, a brick-red body insoluble in water and decomposed by dilute acid, having a composition agreeing with the formula  $\text{CrO}_4\text{Pb} \cdot \text{PbO}$ ; a yellow body,  $\text{PbCrO}_4 \cdot \text{K}_2\text{CrO}_4$ , and an orange body  $\text{PbCrO}_4 \cdot \text{K}_2\text{CrO}_4 \cdot 2\text{PbO}$ . In the same way, substituting sodium nitrate for potassium nitrate, they have obtained  $\text{PbCrO}_4 \cdot \text{Na}_2\text{CrO}_4$ , a yellow body soluble in water, and  $\text{PbCrO}_4 \cdot \text{Na}_2\text{CrO}_4 \cdot 2\text{PbO}$ , an orange body. By using lithium nitrate they have obtained  $\text{PbCrO}_4 \cdot \text{Li}_2\text{CrO}_4$ . A better yield of the double chromate is obtained by fusing with the alkaline nitrate and lead chromate some alkaline chromate. —A. L. S.

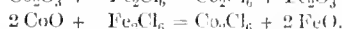
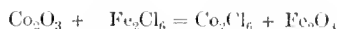
## PATENT.

*Process for the Production of Solutions of the Chlorides of Nickel and Cobalt and other Metals from the Ores or Products containing these Metals.* W. L. Wise, London. From B. Natuseh, Niederlosnitz, Germany. Eng. Pat. 19,334, December 2, 1889. 4d.

THE object of the invention is the production of a pure solution of nickel or cobalt direct from substances containing one or both of these metals. For this purpose the substance, after roasting, is chloridised at a red heat by means of chloride of iron, and the resulting product lixiviated with water. The reactions which take place in the case of cobalt are as follows:—



and—



When a solution obtained by treating a test portion of the roasted mass with water ceases to contain any more iron, the roasting process is finished. The product is

drawn into pans that can be of iron, and treated with water. In order to ensure that the metals present shall be obtained with a minimum amount of loss in one treatment, it is necessary that, as soon as the mixture is at a glowing or red heat throughout, there should be still some undecomposed ferrous chloride present, because the reaction is not completely effected until this temperature is reached.

With a roasted ore containing 10 per cent. of cobalt, 5 per cent. of nickel, and 5 per cent. of copper, an addition of about 20 per cent. of ferrous chloride will be found sufficient to ensure complete decomposition. A charge of about 200 kilos. of such a mixture will require to be roasted for one hour. In addition to cobalt and nickel there will be found, in solution, gold, silver, zinc, and copper, pure iron oxide remaining as residue.—J. B. C.

## VIII.—GLASS, POTTERY, AND EARTHENWARE.

*Contribution to the Knowledge of Felspar Porcelain.* E. Hussak. *Sprechsaal*, 22, 154, 136.

IN continuation of Bünnz's investigations (*Sprechsaal*, 1876, 39—45) the author has microscopically examined some of the materials used in the manufacture of porcelain, and this product in various stages of formation. Kaolinite from Nationale Belle mine, Denver, Colorado, is a nacreous powder consisting of microscopical well-developed hexagonal plates belonging to the monoclinic or to the triclinic system. Levigated China clay is a mass of almost pure kaolinite, the plates have, however, a rounded or irregular outline. Saxony clay contains extremely minute scales, which appear bluish-grey by polarised light and are thickly studded with grains of ferric hydrate. It is very rich in accessory minerals as quartz, zircon, rutile, and occasional scales of mica. No opal was found in any of the above products nor in the kaolins of Cornwall, Linnoges, or Türkismühle.

The author has also repeated some of Fouque and Lévy's experiments on the synthetic formation of minerals. Orthoclase was converted into microcline by melting it to a clear glass, allowing it to solidify, and heating for 24 hours at a temperature close to the melting-point. Hornblende is changed by similar treatment into augite. In the manufacture of porcelain, the orthoclase cannot crystallise out, because the temperature of fusion is too high and the cooling is too rapid. In no case was any decomposition of the orthoclase effected by fusion; the view that fusion is attended with a loss of alkali is therefore very improbable. If glasses be regarded as supersaturated solutions of salts, it is evident that felspar glass must be capable of taking up a certain amount of silica or silicate. This assumption is confirmed by the examination of specimens of porcelain in different stages of baking. In the first stage no glass is yet formed, kaolinite, felspar, and quartz lie side by side; the second stage shows the commencement and progress of vitrification, the gradual disappearance of the felspar, and an increase in the number of pores, but much quartz is still present. At the third stage (samples 4, 5, and 6) much amorphous devitrified glass and a little clear glass is present, the quartz decreases, and the pores are fewer but larger. Sample 6, when completely baked, contained relatively few large quartz fragments, the amorphous mass predominating; the outlines of the fragments of felspar were not so clear as in the other samples. The vitreous mass is permeated by a thick felt-like mass of thin doubly refracting needles resembling sillimanite,  $Al_2SiO_5$ .

The retention of their outlines by the fragments of felspar is noteworthy.—S. B. A. A.

*New Varieties of Porcelain.* Annales Industrielles, 1889, 146.

Messrs. Parvillée have composed a new paste for the porcelain-facing of bricks and wall tiles; they have a better appearance than the ordinary enamelled tiles, and it is claimed they are more durable, since they are impervious to water. The cost is only about one-fourth greater.

Lauth's new Sèvres porcelain is a soft variety resembling Chinese porcelain; it is fired at a low temperature and admits of a variety of underglaze colours being applied. It has the following composition: silica, 64.3 or 71 per cent., alumina, 28.92 or 23 per cent., potash and soda, 7.05 or 6 per cent.

The glaze used has the composition: silica, 66.56 per cent.;  $Al_2O_3 \cdot CaO$ , 14.23 per cent.; potash and soda, 3.59 per cent. The new porcelain is baked at  $1,350^\circ$ , whereas the old variety fritted at  $1,500^\circ$ .

T. Deck has prepared a new paste suitable for moulding (not casting) very large vases, &c. It consists of a real felspar body with a considerable addition of fine sand. The reliefs are fixed on the vessel after shaping. The glaze is sprinkled on.

A new soft-body porcelain has been introduced at Sèvres by Deck (compare this Journal, 1889, 983—984). The new body possesses the same translucency and takes the same glaze and colours as the ancient soft Sèvres porcelain, the former is, however, more easily shaped and may be fired without much difficulty.—S. B. A. A.

*Classification of Earthenware.* Hartig. *Thonindustrie Zeitung*, 12, 120—646.

THE author proposes a system of classifying earthenware goods according as they are glazed, unglazed, or enamelled, and according as the freshly broken surface is white or coloured, and porous or impervious to a drop of water. By this means earthenware goods may be very sharply divided into nine classes. The porosity is ascertained by comparison with a piece of dense porcelain.—S. B. A. A.

*The Action of the Sulphur in Coal on Earthenware.* *Thonindustrie Zeitung*, 14, 59 and 74.

THE author attributes the inferiority of modern to ancient German brick buildings to the use of coal as a fuel in firing the bricks.

The sulphur contained as iron pyrites, &c. in the coal is oxidised and taken up by the bases in the bricks with the formation of sulphate (compare this Journal, 1889, 983). If the bricks contain much calcium or magnesium carbonates this action may go on to a very large extent and the surface may absorb as much as 10 per cent. of sulphuric acid. A portion of the acid separates out again when the tile is heated in a reducing atmosphere, but in most modern kilns an oxidising atmosphere is constantly maintained. These sulphates disintegrate the brick by a mechanical action similar to that of water upon rocks, they deliquesce in damp weather, and on drying they form groups of crystals which press upon the adjoining particles of the brick. When these sulphates are present in any quantity, as frequently happens, they form efflorescences, and are often mistaken for nitrates of calcium, &c.

Clays in which the proportion of lime to ferric oxide is large do not burn reddish or yellowish-brown, but acquire a light red or yellowish-grey colour owing to the formation of a silicate of iron alumina and lime; when, however, the lime is combined with an acid which is not decomposed by silica at the temperature of firing, only a ferric aluminium silicate is formed; such bricks are reddish-brown on the surface, and preserve their normal colour in the interior. The yellow colour may be restored to the exterior by reheating in a reducing atmosphere. No discolouration of this kind can of course occur with wood firing. Glazed facing tiles are subject to the same danger of disruption by the sulphates, as the firing and glazing is conducted in an oxidising atmosphere obtained from coal.—S. B. A. A.

*Modern Majolica.* Henhart. Deutsche Bauzeitung, 1889, 39.

THE author distinguishes three varieties of majolica ware according to the fusibility of the coloured glazes. The first class of glaze is fired at the temperature of a gold lustre; the mixture used in English relief glazing and Linke's glaze are of this character; the want of durability ascribed to it by Krell may be obviated by selecting a glaze adapted to the body, and by sufficient firing.

The second class of glaze is fired at an incipient white heat and is very durable. The third class of glaze is used by various French manufacturers at the temperatures employed for fine earthenware; it is very durable and either underglaze or overglaze colours may be applied: the latter method is adopted by Deck with excellent results.

English productions are especially censured by the author on account of the frequent crazing, a defect easily avoided with the fine clays in use here.—S. B. A. A.

#### PATENTS.

*Improvements in the Manufacture of Terra Cotta or Porcelain Castor Bows, Knobs, and Handles.* F. C. Clare, Steehford. Eng. Pat. 9841, June 15, 1889. 4d.

A NON-VITREOUS glaze, in the form of any varnish, japan, gum, or lacquer, is used instead of the more costly vitreous glaze commonly applied.—E. G. C.

*Improvements Relating to the Enamelling of Articles of Copper.* H. H. Lake, London. From A. Gilbert, Laeken, Belgium. Eng. Pat. 11,979, July 27, 1889. 4d.

COPPER cooking utensils and the like are enamelled internally with a composition consisting of 4 parts of felspar, 3 parts of flint, 5 parts of borax, and 2 parts of cryolite.—E. G. C.

*Improvements in Treating or Ornamenting Glass and Glass-ware.* M. Lugar, London. From C. Lasalle and C. Mautter, Paris, France. Eng. Pat. 13,577, August 28, 1889. 4d.

APERTURES of suitable shapes are cut or otherwise made in the articles to be treated. These are filled in with various coloured glasses or other materials, which may project beyond or be in the same plane as that of this portion of the body. To hold the inserted glass in position, cement may be used or preferably clips of metal placed around the edges of the apertures and of the pieces to be secured therein.

—S. G. R.

### IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

*The Action of Alkalis, Alkaline Earths, Alkaline Silicates, and some Saline Solutions on Mica; the Formation of Sodalite, Amphigene, Orthoclase, Anorthite.* C. and G. Friedel. Compt. Rend. 110, 1170—1178.

THE authors propose making experiments to determine the action of water, containing certain alkaline salts in solution, on different rocks, the experiments being made at an elevated temperature and under such conditions as to realise most nearly the actions which occur in nature; at present the work is only partially completed.

The materials to be acted upon are heated to a temperature of 500° C. in a thick platinum-lined steel tube, two-thirds filled with solution. In this way mica and potash yielded sodium and potassium nepheline,  $2\text{SiO}_2 \cdot \text{Al}_2\text{O}_3 \cdot \left\{ \begin{smallmatrix} \text{Na} \\ \text{K} \end{smallmatrix} \right\}_2\text{O}$ ;

when sodium chloride is also present, a compound of nepheline and sodium chloride is obtained which may be represented as  $3(2\text{SiO}_2 \cdot \text{Al}_2\text{O}_3 \cdot \text{Na}_2\text{O}) \cdot 2\text{NaCl}$ . Silica, potash and mica yielded orthoclase, other proportions gave leucite,  $4\text{SiO}_2 \cdot \text{Al}_2\text{O}_3 \cdot \text{K}_2\text{O}$ . Lime and mica in the presence of calcium chloride gave anorthite.—A. L. S.

*Linoleum Flooring.* Günther. Dingl. Polyt. J. 276, 360—363.

THE author describes the arrangements for flooring with linoleum made at the Duke Ernst seminary at Gotha. Most of the floors were formed of a bed of concrete laid down on iron supports, levelled and smoothed; the linoleum was cemented on with a paste of rye flour and turpentine, a tight fit being secured by pushing the ends into a deep groove in the skirting board. The stairs were of iron covered with concrete and artificial stone; the linoleum was imbedded in the steps to preserve the edges. The author recommends that the concrete floors in similar buildings should only be roughly levelled, and a smooth surface obtained by running on a layer of thin plaster and allowing it to harden. A fireproof flooring is thus formed the cost of which is only a fraction higher than that of a stained wooden flooring. The best linoleum will last for 20 years. It soon loses its characteristic smell if well aired, and should be periodically polished with a mixture of wax and turpentine.—S. B. A. A.

#### PATENTS.

*A Composition for Arresting or Preventing Decay in Wood.* J. T. MacMahon, London. Eng. Pat. 13,203, August 21, 1889. 4d.

See under XVIII., C., page .

*An Improved Composition for use in and in connection with the Manufacture of Portland Cement.* G. Higham, Woulham. Eng. Pat. 6855, May 3, 1890. 4d.

THE patentee mixes  $2\frac{1}{2}$ —3 cwt. of chalk or limestone with 1 cwt. of mud or clay and 8—12 chaldrons of fuel such as turf, breeze, or charcoal, moulds into bricks and burns in a kiln to produce Portland cement.—B. B.

### X.—METALLURGY.

*Jüngst's Experiments on the Application of Ferro-silicon to the Manufacture of Pig Iron.* Dingl. Polyt. J. 276, 346—355.

IN consequence of the experiments of Turner (this Journal, 1886, 289 and 606), Bischof, and Gautier (this Journal, 1889, 791) on the influence of silicon on iron, the Union of German Smelters and the Prussian Minister of Public Works commissioned Jüngst to make a series of experiments to test the possibility of producing a substitute for Scotch pig by the addition of silicon to other classes of iron, and to ascertain the best proportions of ferro-silicon for economical production and the influence of impurities (S, P, Mn, &c.) in presence of silicon.

The materials employed by Jüngst in his experiments were as follows:—(a.) Three kinds of ferro-silicon containing respectively 5·3, 10·4, and 14·3 per cent. of silicon; (b.) three kinds of white iron with 0·85, 0·35, and 0·33 per cent. of silicon, 3·54, 3·41, and 0·52 per cent. of manganese, 3·93, 3·53, and 2·76 per cent. of carbon; 1·07, 1·08, and 0·91 per cent. of phosphorus; (c.) three kinds of scrap iron containing 3·38, 2·71, and 2·05 per cent. of silicon; (d.) two kinds of burnt iron; (e.) seven

varieties of grey iron containing 3.02 to 1.06 per cent. of silicon, 3.52 to 2.77 per cent. of carbon, 2.01 to 0.44 per cent. of manganese, 1.49 to 0.1 per cent. of sulphur; (f.) wrought iron scrap.

Complete analyses were made of each variety, which was then melted down separately, its influence upon various mixtures was then observed.

A comparison of the analyses of pig iron and cast iron before and after remelting shows that silicon decreases irregularly, the average loss being 17 per cent.; combined carbon decreases in proportion as silicon can act upon it; an increase was only noted when burnt iron or grey iron containing no more than 1.06 per cent. of silicon was melted; in castings of a mixture of grey pigs and scrap and burnt iron the percentage of combined carbon remained remarkably constant. The total carbon increases in almost all cases in which silicon and manganese decrease, these elements protecting the carbon from oxidation. Manganese always decreases, the average loss being 29 per cent., phosphorus only undergoes very trifling variations; sulphur generally increases, when this increase is considerable it is due to the sulphur in the fuel. The presence of 1.55–2.25 per cent. of silicon decreases the tendency of the iron to absorb gases, a small percentage of silicon on the contrary increases this tendency, a larger percentage leads to the formation of cavities.

The soundest cast iron was obtained with 2.24 per cent. of silicon, with an increase of silicon the tendency of the castings to crack on cooling is diminished, but the injurious influence of the manganese becomes more apparent. The relation between absorption and contraction renders it probable that silicon diminishes the contraction by causing the combined carbon to separate out as graphite. The small absorption and contraction of iron melted with ferro-silicon renders superfluous the use of large "gits" of metal in casting. The experiments show that castings of this class possess extraordinary strength. This is due partly to the chemical and partly to the structural changes effected by the silicon. The fracture is characteristic, a sound tough iron showing a light grey network of close meshes filled with a dark shiny mass. Jünger considers this network to be a steel iron containing 0.5 per cent. of combined carbon, and the interstitial matter to consist of graphitic and silicon compounds; the former occasion the great toughness of the metal, and the latter conduce to the great resistance to shock and to the slight tendency to absorption. A brittle hard iron exhibits a drawn-out radiating appearance, in soft weak irons the network is very coarsely meshed and discontinuous.

The maximum transverse strength does not depend upon any particular percentage of silicon. Nearly the same results were obtained from pigs containing 1.22 to 3.07 per cent. of silicon, but the best results are only obtained when the combined carbon lies between the limits 0.49 and 0.69 per cent., and the graphite between 1.49 and 2.89 per cent.; the latter element increases the malleability and the deflection. The presence of over 3 per cent. of silicon or of over 1 per cent. of manganese diminishes the transverse strength; but up to 1 per cent. of phosphorus and 0.16 per cent. of sulphur may be present without apparent injury to the quality of the metal.

The tensile and compressive strengths are affected in the same manner as the transverse strength.

The resistance to shock is extraordinarily great; a cube of 1.2 in. side was broken by 11 blows, the work done by each blow amounting to 817 foot pounds; a plate 1 metre square and 20 millim. thick was broken by 24 blows of a ram weighing 55 lb., the last blow falling 5.25 metres; these specimens contained 2.24, 2.57, and 2.09 per cent. of silicon, 2.22, 2.52, and 1.81 per cent. of graphite, and 0.45, 0.89, and 0.55 per cent. of manganese.

Whereas large castings made from mixtures of different brands of grey pig generally contain flaws, &c. and are comparatively weak, large castings (hydraulic cylinders, &c.) made from white iron and ferro-silicon generally proved absolutely flawless and of remarkable strength. The quality of the iron was evident on machining, the turnings occasionally attaining a length of 21 in.

The strength is about 50 per cent. in excess of any cast iron in use, the transverse strength averaging over 23 tons and the tensile strength over 14 tons per square inch. Jünger concludes that Turner, Ledebur, Wood, and Gantier's statements about the influence of silicon on iron are, in general, well founded, and he further draws attention to the following points: Before adding ferro-silicon to a pig iron its composition should be ascertained, and the amount of ferro-silicon added must be proportionate to the amount of combined carbon and graphite present. Ferro-silicon should never be added to grey pigs or to other iron already rich in silicon.

The strength of the iron depends as much upon its structure as upon its chemical composition. This structure is developed in grey pig by repeated remelting, but the soundness of such castings is always doubtful. As large perfectly sound castings of great strength may readily be obtained from white iron and ferro-silicon, it is both technically advantageous and economical to use this metal for purposes in which great strength and soundness are essential. The high price of ferro-silicon would, however, at present prevent its application to common castings.

The variety of ferro-silicon most advantageous for smelting purposes is that containing about 10 per cent. of silicon; the use of a much poorer variety (5 per cent. of silicon) involves the introduction into the metal of a large quantity of manganese which deteriorates the quality of the product. When a richer variety (14 per cent. silicon) is used, very little impurity is introduced, but it becomes necessary to guard against the setting up of a purely local action by thoroughly stirring the fluid metal. Care must be taken that not more than 1 per cent. of manganese or of phosphorus and not more than 0.16 per cent. of sulphur are present; particular care should be taken to avoid an excess of manganese.

Ledebur (Stahl und Eisen, 1890, 4) is of opinion that silicon only acts indirectly upon white iron by converting it into a species of grey iron which is superior to other varieties because of its greater purity.—S. B. A. A.

*The Action of Carbon Monoxide on Nickel.* L. Mond, C. Langer, and F. Quinke. Proc. Chem. Soc. 1890 (86), 112–113.

WHEN carbon monoxide is passed over finely-divided nickel, such as is obtained by reducing nickel oxide by hydrogen at about 400°, at a temperature between 350° and 450°, carbon dioxide is formed, and the nickel is gradually converted into a black, amorphous powder, consisting of carbon and nickel; the composition of this deposit varies widely with temperature and time. A small quantity of nickel can thus change a very large amount of carbon monoxide, the action being complete and rapid at first, and continuing, although at a diminishing rate, for several weeks. A product containing as much as 85 parts carbon to 15 parts nickel was obtained. Acids only partially remove the nickel; the carbon is very readily acted on by steam, carbon dioxide, and hydrogen without a trace of carbon monoxide being formed at a temperature of 350°.

On allowing the substance to cool in a current of carbon monoxide, it was noticed that the flame of a Bunsen burner into which the escaping gas was introduced became luminous, and when the tube through which the gas passed was heated, a deposit of nickel, mixed with a small quantity of carbon, was obtained. The authors were thus led to discover the existence of a volatile nickel compound.

To prepare this compound, a combustion-tube was filled with nickel oxide and this was reduced by hydrogen at about 400°; after cooling the nickel to about 100°, pure dry carbon monoxide was passed over it without further heating, and the issuing gas led through a tube placed in a freezing mixture: the major portion of the nickel compound condensed as a colourless liquid; but since the gas retained about 5 per cent., it was collected, dried and again passed over the metal. When no more liquid condensed, the nickel was again heated to about 400° in a slow current of pure carbon monoxide; it was then cooled to about 100°, and again submitted to the action of the gas.

Nickel carbonoxide thus prepared is a colourless liquid, which boils at  $43^{\circ}$  under 751 mm. pressure; its relative density at  $17^{\circ}$  is 1.3185. It solidifies at  $-25^{\circ}$  to a mass of needle-shaped crystals. Its composition is represented by the formula  $\text{Ni}(\text{CO})_4$ . It dissolves in alcohol, and more readily in benzene and chloroform; dilute acids and alkalis have no action on it, but it is oxidised by concentrated nitric acid. It reduces an ammoniacal solution of cupric chloride, and also causes the separation of silver from an ammoniacal solution of silver chloride. It interacts with chlorine, forming nickel chloride and carbon oxychloride. It is decomposed at  $180^{\circ}$  (in boiling aniline vapour) into nickel and carbon monoxide. The atomic weight of the deposited metal was found in three experiments to be  $58.52-58.64$ , a result closely corresponding with Russell's value,  $58.74$ .

Numerous experiments to obtain similar compounds with other metals, notably with cobalt, iron, copper, and platinum, led to negative results. On experimenting with specially purified cobalt, in the beginning a slight colouration of the Bunsen flame into which the gas was led was noticed, but after a time this was no longer observed. Commercial cobalt afforded a gas which deposited a mirror of pure nickel, it being possible, in fact, to purify cobalt from nickel by carbonic oxide. The nickel mirrors obtained by heating the carbonic oxide compound do not appear to contain any trace of cobalt.

#### PATENTS.

*Improvements in Machinery and Appliances for Separating and Cleaning Auriferous and other Mineral Ores and Substances, and also Precious Stones, from their Gangue and other associated Refuse Matter.* G. M. Edwards, London. Eng. Pat. 8147, May 16, 1889. 8d.

RECTANGULAR channels with perpendicular sides are placed at an inclination of one in six between upper and lower ends. The bottoms of the channels slope at an angle of about one in four for a distance of about two feet and then rise perpendicularly to a height of about 8 in.; this arrangement being repeated to the end of each channel. Above the sloping floor are placed wedge-shaped blocks so shaped that a clear passage of about 1 in. in height is left, along which the ore and water are made to flow. Valves are placed at the end of each slope for the purpose of removing deposited matter.—H. K. T.

*Improvements in the Treatment of Waste Pickle from Galvanising Works.* T. Turner, Birmingham. Eng. Pat. 9225, June 4, 1889. 8d.

A CLOSED fire-brick furnace is used, which is heated from below, and is furnished with working doors, and with a pipe passing through the upper part of the furnace for carrying away the regenerated acid.

The bottom of the furnace is made of large fire-bricks or iron plates. The waste liquor, concentrated if necessary, is introduced from above, by allowing it to drip or trickle through pipes in the roof or sides of the furnace. The liquid is allowed to enter at such a rate, that it distils as fast as it enters, never forming more than a shallow bath of liquid, but leaving a cake of more or less basic chloride of iron, which fills up any cracks in the bottom of the furnace and protects the bottom. The cake is then broken up and heated either in the same or in another furnace to a somewhat higher temperature not exceeding a dull red heat, while air and steam are passed over in suitable proportions. The steam should be used only in such quantities as may be necessary to prevent ferric chloride distilling in the form of brown fumes with the acid (see also this Journal, 1890, 518).

—J. B. C.

*Improvements in Galvanising Iron and in Utilising the Waste Acids therefrom.* A. G. Greenway, Stafford. Eng. Pat. 9680, June 12, 1889. 4d.

THE iron to be galvanised is first treated with either hydrochloric or sulphuric acid, in order to clean the surface. A solution of ferrous chloride or sulphate is formed, which is

at first retained in a tank and then transferred to a second tank in which a solution of ammonium sulphide is added to saturation. This latter is the ordinary residual product as obtained from gas works. Ferrous sulphide is thereby precipitated, and may be separated when required. The iron, which has been cleaned as described, is immersed in a solution of ammonium chloride or sulphate in the second tank, and this may be either heated or used cold. The iron is then immersed in melted zinc and galvanised. The solution of ammonium chloride or sulphate in the second tank and the ferrous sulphide may be separated by filtration, and the solution of ammonium chloride or sulphate evaporated to form commercial salts of ammonium.—J. B. C.

*An Improved Process and Apparatus for the Amalgamation of Gold and Silver contained in their Ores.* H. Cave, London. Eng. Pat. 10,274, June 21, 1889. 8d.

THE finely pulverised ore is heated in contact with mercury vapour. It is then placed in a boat-shaped rocking vessel, together with mercury and mineral acid. When the mercury is sufficiently rich, it is squeezed in the usual way. The preliminary treatment with mercury vapour may be omitted.

—H. K. T.

*Improvements in the Treatment of Blast-Furnace Slag and other Slags, and in the Apparatus therefor.* T. Hydes, Sheffield. Eng. Pat. 10,835, July 5, 1889. 8d.

MOLTEN slag is conveyed by a conductor on to the top of a cone-shaped distributor and meets, at the edge of the latter, with an air or steam blast, or combination of both, which spreads it into a thin sheet. It then falls through zig-zag tubes surrounded by water.—H. K. T.

*Improvements in the Treatment of Blast-Furnace Slag and other Slags, and in the Apparatus used therein.* T. Hydes, Sheffield. Eng. Pat. 10,836, July 5, 1889. 6d.

THE molten slag passes between a pair of rollers, which spread it into a thin sheet. It then passes over an inclined plane, becoming cooled and brittle, and is crushed more or less to powder by a second pair of rollers. The second pair of rollers may be placed vertically below the first pair, in which case the inclined plane may be omitted. The rollers and inclined plane are hollow, and water is passed through them.—H. K. T.

*Improvements in Extracting Precious Metals from their Ores, and in Means and Apparatus employed therein.* S. Trivick, London. Eng. Pat. 11,883, July 26, 1889. 8d.

THIS is a process for leaching roasted ores to be used with the apparatus described in Eng. Pat. 11,882 of concurrent date. The ores are treated with a leaching fluid heated to  $150^{\circ}\text{F.}$  composed of sodium chloride solution of sp. gr. 1.150, to which is added one-eighth per cent. of neutral chloride of copper. The clear liquor is treated with a liquor prepared by boiling 35 lb. of lime and 18 lb. of sulphur with 100 gallons of water until a specific gravity of 1.050 is reached. Sodium chloride is then added until the specific gravity is 1.100. Sufficient of this liquor is added to precipitate the gold and silver only. The clear liquid is then drawn off and the copper in it is precipitated by a further addition of the above liquor, and is used in a fresh roasting operation. The clear liquor is then pumped into a tank to be used again for leaching. The plant consists of the following apparatus:—A roasting furnace; a chamber connected therewith, in which are coils of pipes packed in sand and supplied with water to be used for heating leaching liquor, &c.; a tank over the furnace for evaporating leaching liquor; a pan for preparing precipitating liquor; filter beds composed of basket-work and moss with canvas above and below; a mixing vessel provided with stirrers and precipitating tanks, &c., all connected by suitable pipes and valves.

—H. K. T.



*Improvements in the Manufacture of Ingot Iron and Steel.* T. Twynam, London. Eng. Pat. 12,500, August 7, 1889. 4d.

THE yield of metal for each blow when working the Bessemer process may be increased by adding to the charge iron oxide in the form of scale or ore, mixed with not less than one-fifth its weight of carbon. The mixture may be in the form of powder, but is preferably made up into bricks with tar and subjected to a preliminary coking. The iron ore, if used, should be not too siliceous, and both it and the carbonaceous matter (coal or coke) should be as free as possible from sulphur, but the presence of phosphorus is not objectionable. When a manganiferous ore is used, manganese is reduced to some extent, but is mostly re-oxidised.

The mixture is added at that stage of the blow when the silicon and about one-third of the carbon have been burnt out, or it may be charged into the converter with the lime, and the molten pig iron subsequently run in upon it.

In working charges of five tons of pig iron with 7—10 per cent. of iron scale made into bricks containing about 30 per cent. of carbon, the patentee has found that the yield of ingots has increased by fully 5 per cent., consequent on the reduction of the iron of the scale added, while the length of the after-blow has been lessened.—B. B.

*Improvements in Extracting Metals from their Ores, and Apparatus therefor.* R. Welford, Sunderland. Eng. Pat. 12,525, August 8, 1889. 8d.

THIS is an apparatus in which the filtration of the liquor, after the usual chlorination process, is carried on in the chlorinating vessel itself. It consists of a rotating barrel with hollow trunnions. One trunnion is connected with tanks supplying water and acid, and with a compressed air supply, if necessary. The other trunnion is suitably connected, on the one hand, with a filter-bed placed in the lower side of the barrel, and also with precipitating vessels, &c. Ore and chloride of lime are charged into the vessel through a hopper; the door is then closed, acid and water admitted, and the vessel rotated. After the chlorination is complete the barrel is placed so that the filter is undermost and the valve connected with the filter-bed is opened, the barrel being made to oscillate to prevent the mass "packing." When all the liquor has been drawn off the door is opened and on rotating the barrel the exhausted ore falls out.

—H. K. T.

*Improvements in or connected with the Manufacture of Iron and Steel, and in the Utilisation therefor of Waste Products.* J. C. Bromfield, Hove. Eng. Pat. 12,839, August 14, 1889. 4d.

COAL or coal-dust and the waste from tin plate works, "that is, the cuttings from the plates which are subsequently tinned and the slag from the tips," are ground together in the proportion of 1—2 tons of coal to 1 ton of waste, alumina with lime or its carbonate added if much silica be present in the waste, and the whole mixed with petroleum or other liquid fuel; 5—20 per cent. of hydraulic cement, carbonate of lime, magnesian lime, or lime are then worked in, and the material moulded into bricks or other forms. "Silica and liquid fuel may be added during the process of mixing where it is required to produce oxygen in the retort." The moulded material is coked in a retort similar to that described in Eng. Pat. 5122 of 1881, "whereby practically the whole of the sulphur and phosphorus is eliminated." (If the bricks are only to be used as a fuel in iron and steel manufacture the tin plate waste may be omitted.) The retort is made in two parts, the upper of fireclay, the lower of wrought iron, and the introduction, coking, and withdrawal of the material takes place continuously. The bricks of mixed fuel and waste, after coking, are ready to be smelted for the production of iron and steel.—B. B.

*Improvements in Refining and Separating Precious Metals.* E. Dodé, Paris, France. Eng. Pat. 14,357, September 11, 1889. 8d.

THE material, consisting of a mixture of gold, platinum, silver, copper, and tin is dissolved in aqua regia, half the acids are evaporated and water equal to three times the weight of the acids used is added. The liquid is then placed in a form of separating funnel, and ether, chloroform, or glycerins, &c., are added, using 300 grms. for every 100 grms. of metal. After all the gold is extracted the ethereal solution is drawn off, a solution of salt is added to the residue to precipitate all the silver, and the platinum is extracted from the solution by means of "essence of lavender, turpentine, or thyme" in a manner similar to that used for the gold. Finally the tin is precipitated by means of ammonia and the clear solution evaporated for the sake of the copper. The gold and platinum are obtained from their solutions by distilling the solvents.

—H. K. T.

*Process for the Production of Solutions of the Chlorides of Nickel and Cobalt and other Metals from the Ores or Products containing these Metals.* W. L. Wise, London. From B. Natuseh, Niederlosnitz, Germany. Eng. Pat. 19,334, December 2, 1889. 4d.

See under VII., page 805.

*Improvements in the Wet Method of Extracting Gold from finely divided Auriferous Quartz Ores and other auriferous Material, and in Apparatus therefor.* R. Brown and G. H. Irvine, Brisbane, Queensland. Eng. Pat. 237, January 6, 1890. 8d.

THIS invention consists in the treatment of finely divided auriferous quartz with gaseous chlorine or a solution of chlorine in water, in vacuo, and in the retention of the vacuum during chlorination. The operation is conducted in a fixed vessel provided with a stirrer or in a rotating barrel. The ore and water are first introduced, the vessel closed and exhausted and the chlorine then allowed to enter slowly, but not in sufficient quantity to entirely destroy the vacuum. The liquor is then drawn off and treated in the usual way.—H. K. T.

*An Improved Amalgamator for the Extraction of Gold and Silver from the Ore by the Action of Mercury.* C. Pfeiffer, London. Eng. Pat. 1794, February 3, 1890. 6d.

THIS apparatus consists of two cylinders, one within the other, the outer one fixed, the inner one rotating. The two cylinders communicate through a number of radial ports in the bottom of the inner cylinder. Mercury is first poured into the inner cylinder and is caused by the centrifugal force to fill the annular space between the two cylinders. The ore, mixed with water, is then poured in and under the action of centrifugal force finds its way through the mercury column.—H. K. T.

*A Method for obtaining Uniform Products from the Thomas Converter, Siemens' Open Hearth, or other Basic Furnaces.* L. Pszczolka, Graz, Austria. Eng. Pat. 3782, March 10, 1890. 4d.

"THE object of this invention is to prevent the variations arising in the quality of the metal produced from all kinds of furnaces prepared in the basic manner." "The object is attained by causing the combinations of oxides of iron, which are formed in the metal produced as aforesaid to be scorified by the addition of substances containing silicic acid ( $\text{SiO}_2$ ) instead of reducing them by carbon, manganese or silicon or like additions, by which a soft iron is made, which can never be brought to the same degree of carbonisation as the main part of the metal producing inequality or

variation in the products of basic processes especially in the harder qualities.<sup>2</sup> The metal, fined in the usual manner, is run in a liquid state into a suitable caldron previously heated or into an oxidising smelting furnace. During, before or after pouring in, there is added a considerable quantity of silica in the form of slag, glass, quartz, &c. This combines with the oxides, forming a slag. When the darkening of the slag becomes stationary the operation is complete. The quantities of silica required vary, as also the duration of the process. The metal is then recarbonised in the same vessel before casting or it is run into an ordinary pan for recarbonisation. For producing malleable iron the decarburetted and dephosphorised metal is freed from oxides according to the above described process and then cast without the addition of any recarbonising materials.—J. B. C.

*A Method for Producing Iron or Steel from Ores and other Materials containing Iron.* J. von Ehrenwerth, Austria. Eng. Pat. 4386, March 20 1890. 6d.

IRON ore and a highly carbonised iron are brought together at a high temperature. The ore is reduced at the expense of the carbon of the metal. The latter is then recarbonised by contact with carbon and a further quantity of ore is added. The operation is best conducted on the hearth of a Siemens furnace, the iron-containing materials being added in a fluid state to the metal bath. When the carbon of the latter is exhausted it is run out into a small shaft furnace containing coke or charcoal. It is then returned to the Siemens furnace and a fresh portion of ore is added. When a sufficient quantity of metal has been obtained, it is recarbonised in part or whole and cast in the usual way. Instead of working intermittently, the ore and carbon may be supplied continuously, the process only being interrupted for the removal of slag.—H. K. T.

*Improvements in Processes for obtaining Chromium and Chromium Alloys.* A. K. Eaton and K. A. Vanderbilt, Brooklyn, U.S.A. Eng. Pat. 5003, March 13, 1890. 4d.

A CHROMITE is first formed by heating together to a white heat equivalent proportions of potassium bichromate and the sulphate, preferably that of the metal whose chromite it is desired to obtain. The metals best fitted for this purpose are iron, copper, tin, and zinc. The chromite is then reduced by heating with carbon or a carbonaceous substance such as sugar, an alloy of chromium and the metal selected resulting. The latter may then be removed by a suitable solvent yielding metallic chromium. The metal best adapted for the preparation of pure chromium is zinc, which can be dissolved out from the alloy by means of nitric acid. With other metals the chromium alloys obtained are themselves valuable; thus copper and chromium give a good chromium bronze; iron and chromium yield a hard alloy capable of taking a high polish; while tin and chromium give a product which when further diluted with tin is said to resemble silver.—B. B.

*Improvements in Apparatus for the Production and Application of Gases for Metallurgical and other Purposes.* J. J. Shedlock, New Barnet, and J. J. Meldrum, Manchester. Eng. Pat. 6864, May 3, 1890. 6d.

THIS invention has for its object the economical production of a continuous supply of reducing and heating gases in which the proportions of carbonic oxide and hydrogen may be properly regulated, with special reference to the process described in Eng. Pat. 15,504 of 1884. A mixture of steam and air are forced into a large condensing chamber by means of a steam injector and after condensation of any desired proportion of the steam, the remaining gases are conveyed into a superheater, there raised to a high temperature and introduced under considerable pressure into a gas producer; the producer gas is then forced into a bath of molten metal simultaneously with the finely-divided ore under treatment; the metals in the ore are reduced and

alloy with the metal in the bath. The superheater and boiler are likewise heated by gaseous fuel from a producer supplied with air and steam by an injector or mechanical blower.—S. B. A. A.

*Improvements in and Preparation of Materials for Use in Converting Iron or Low Steel into High-grade Steel by the Cementation Process.* F. G. Bates, Philadelphia, U.S.A. Eng. Pat. 7820, May 20, 1890. 4d.

MILD steel or wrought iron is converted into high carbon steel by the cementation process, using the patentee's composition consisting of 80—100 parts of carbon and 5—10 of cryolite in the case of mild or Bessemer steel, with the addition of 10—20 parts of slaked lime for wrought iron, and 5—10 parts of rosin or carbonate of soda for malleable cast iron. After heating as in the ordinary cementation process the articles treated are preferably cooled rapidly. Metal thus prepared is capable of being hardened and tempered, and is suitable for cutting-tools; plates partially converted may be used as compound armour-plates.

Of the ingredients necessary, the cryolite may be replaced by other fluorine compounds and the slaked lime by other hydrated substances, while the carbon may be in the form of coke or charcoal; it is preferable to use the residue obtained by charring crude petroleum, by allowing it to fall in a slow stream into a heated retort; failing this, crude petroleum may be used directly as an ingredient in the cementation mixture.—B. B.

## XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

*The Separation of Copper from Arsenic by Electrolisis.* L. W. McCay. Chem. Zeit. 14, 509.

See under XXIII., page 822.

### PATENTS.

*Improvements in the Manufacture of Elements for Voltaic Batteries.* D. G. FitzGerald, London. Eng. Pat. 4671, April 15, 1885. Amended May 24, 1890. 8d.

THIS is the amended patent for Lithanode plates. A lead oxide, such as litharge, is mixed with a salt which is decomposed by the oxide with the production of an insoluble salt of lead, so causing the whole to set. Such a salt is the sulphate of ammonia. The mixture is placed in a frame or mould and strongly compressed. A little slagwool, pumice, or such material may be added to increase the porosity. The grey plates are made preferably by pasting each face of a thin sheet of lead. The electrolyte in which the plates are formed is preferably a solution of sulphate of magnesia. The brown plates are charged and discharged several times in the above electrolyte, and then in dilute sulphuric acid for a number of times depending on their porosity. Before the electrolytic treatment, the brown plates are preferably formed superficially by the action of sodic or magnesic hypochlorite. This may be done by placing them in a tank containing hot water and some calcined magnesite; then by passing chlorine through, the magnesic hypochlorite is produced and effects the required superficial action.

In the amended specification the third claim has been erased, viz.:—"The herein described process of peroxidising plates, or masses, of lead which has been caused to 'set' as herein described, by submitting them to the oxidising action of chlorine in the presence of water, substantially as specified." (See also this Journal, 1887, 558.)—E. T.



*Improvements in Electrical Accumulators or Secondary Batteries.* F. Bosshardt, Manchester. From R. F. Verdier, Brussels, Belgium. Eng. Pat. 8973, May 30, 1889. 8d.

THE chief features of this patent are the composition of the active substance and of the electrolyte. The former is a mixture of "oxide of lead, litharge or red lead" with a vegetable siccativ oil, or glycerin and water. The plate is pasted with this mixture, and the paste is compressed and furnished with perforations. When hardened sufficiently it is put into the cell, resting on a horizontal perforated support, with a sheet of some such material as asbestos between. For forming, the electrolyte is a solution of an alkaline sulphate, but for after-use an acid electrolyte is used. The remainder of the patent refers to minor details of arrangement and construction of the electrodes.—E. T.

*Improvements in Electric Batteries.* H. H. Lake, London. From C. A. Hussey and E. H. Brown, New York, U.S.A. Eng. Pat. 10,695, July 2, 1889. 8d.

IN this two-fluid battery a horizontal porous partition is employed and the preferable mode of obtaining this horizontal arrangement is by using a porous cup-shaped vessel, the sides of which have been rendered non-porous either in the manufacture or by the application of some substance such as paraffin. The cup is also provided with lugs by means of which it can be supported by the walls of the containing vessel.

The containing vessel is made with a large spout through which the necessary materials can either be put into or withdrawn from the vessel.—B. T.

*Improvements in Electric Batteries.* H. H. Lake, London. From C. A. Hussey and E. H. Brown, New York, U.S.A. Eng. Pat. 10,696, July 2, 1889. 8d.

THE invention is an improvement in the construction of those cells employing a solution of a copper salt. The zinc is placed at the top of the cell, suspended in a porous cup but without touching it. The negative electrode is at the bottom. Below the porous cell is a perforated disc of some such metal as iron, which can decompose any of the copper salt that may diffuse upwards, before it can reach the zinc. The latter has a groove round its top surface to contain mercury.—E. T.

*Improved Apparatus for Extracting Gold and Silver from their Ores by Electrical Amalgamation.* G. Button and W. E. Wyeth, Cambridge, South Africa. Eng. Pat. 10,822, July 4, 1889. 4d.

THE ore, suspended in water, is made to travel across channels containing mercury. The channels form part of a table covered with copper. Above them are placed metallic brushes suspended so as to have a to-and-fro motion. The table is connected with the negative pole of a dynamo; the brushes with the positive pole. The ore and water are made to flow over flat plates before and after passing over the corrugated table. The plates and table are coated with gold, burnt in.—H. K. T.

*Manufacture of Elements or Plates for Secondary Batteries.* J. H. Johnson, London. From A. L. Riker, New York, U.S.A. Eng. Pat. 11,070, July 9, 1889. 8d.

IN order to retain the active material a grid or plate of lead is cast with holes of such a shape that they have a larger area on one side than on the other. These holes are then packed with the active material and the plate is folded on itself, so that the large ends of the holes are inside and the conical wedge of lead oxide cannot possibly fall out. A sheet of absorbent material, such as asbestos, may be placed in the fold in order to increase the capacity of the plate by exposing more surface to the action of the electrolyte.

—B. T.

*Improvements in and Connected with the Manufacture of Iron and Steel.* T. Parker, Wolverhampton. Eng. Pat. 11,393, July 16, 1889. 4d.

IRON is dissolved by hydrochloric or sulphuric acid, the solution neutralised by an alkali, and the liquid used as the electrolyte of a bath in which the anode is iron in the form of borings, turnings, or scrap, and the cathode, preferably iron, shaped to fit the containing vessel. With a difference of potential of 1.5—2 volts, iron is continuously dissolved from the anode and re-deposited on the cathode. The metal thus obtained is said to be free from the impurities that impair its value for making high class iron and steel.

—B. B.

*Improvements in the Formation of Electrodes for Use in Primary and Secondary Electric Batteries.* L. Epstein, Berlin, Germany. Eng. Pat. 350, January 8, 1890. 8d.

ELECTRODES of plain, grooved, or corrugated lead are boiled for some time in a weak solution of an acid or salt that has the power of dissolving lead; such, for example, as dilute nitric acid, carbonate of soda, &c. They are then dried by exposure to the air. These are then made into positives by forming in the usual way in an electrolyte of sulphuric acid, or a suitable sulphate, —a little acetic or similar acid being added. Negatives are produced from positives made as above, by a second forming process, with the current passing in the reverse direction. Such electrodes are said to have large storage capacity, rate of output, and durability, and to require very little time and no reversals for the process of formation.—E. T.

*Improvements in Electric Batteries.* H. H. Lake, London. From the Crosby Electric Company, New York, U.S.A. Eng. Pat. 3924, March 12, 1890. 6d.

IN a zinc containing-jar, preferably amalgamated, is placed a carbon pencil insulated by hard rubber rings. The space between the carbon and the zinc jar is packed with cotton waste or other spongy material prepared as follows: It is first soaked in strong sal-ammoniac and then squeezed out, the greasy matters being thereby removed; it is then once more saturated and packed between the zinc and the carbon. There should be crystals of sal-ammoniac adhering to it. Connexion is preferably made with the carbon by a cap of tin-lead alloy cast on.—E. T.

*Improvements in Secondary Voltaic Batteries.* H. T. Cheswright, Carcassonne, France. Eng. Pat. 4087, March 15, 1890. 6d.

LEAD tubing of round or other convenient section is made in the usual way except that it is furnished with longitudinal ribs as shown. Lengths of this tubing are placed horizontally one above the other, in a suitable supporting frame.



ELECTRODE FOR SECONDARY BATTERIES.

A plate is thus formed with a number of grooves on each face. Into these grooves the active material is pasted. The supporting frame has a projecting lug or bracket with a short transverse piece of tube at its end. When all the positives or negatives are placed in position, these short pieces of tube come into line so that a bolt can be put through the whole series, and by tightening the nut on this bolt the whole can be firmly bound together. The pieces of

tube are of such a length that when so tightened up the various plates will be the correct distance apart. Such electrodes are said to have great strength and yet to be very light.—E. T.

*Improvements in the Process of Bleaching Fibrous Material by the Aid of Electricity.* C. Kellner, Vienna, Austria. Eng. Pat. 5054, April 1, 1890. 6d.

See under VI., page 804.

*An Improved Insulating Compound.* H. H. Lake, London. From A. A. Kundson, Brooklyn, U.S.A. Eng. Pat. 5730, April 15, 1890. 4d.

This compound consists of about equal parts of shellac or its equivalent, and carbolic acid.—E. G. C.

*Improvements in Secondary Batteries.* G. Barker, Birmingham. From H. Woodward, Toronto, Canada. Eng. Pat. 6176, April 23, 1890. 8d.

Each electrode consists of a central lead conductor, in a perforated tube of vulcanised rubber or other suitable material, the space between being packed with filings, shavings, &c. of lead or lead alloy. These tubes are placed vertically in rows, passing at top and bottom through holes in horizontal plates of insulating material. The central conductors of similar electrodes are attached to a common cross-piece at the top of the cell. Formation is hastened by saturating the lead filings in nitric acid and straining, before filling into the tubes. It is also hastened by the addition of a small quantity of sulphate of soda or magnesia.—E. T.

*An Improved Galvanic Element with Constant Current, applicable especially for producing Incandescent Light.* J. von der Poppenburg, Berlin, Germany. Eng. Pat. 6893, May 3, 1890. 4d.

A porous material, such as chaff, wood, &c., is packed between the zinc and carbon of the cell, together with a liquid containing "vegetable ethers in aqueous solution." It is said that oxide of zinc is formed and, being retained near the surface of the zinc plate, reunites with it in the shape of "projecting points or bodies of zinc." The "ethers" also are said to have the effect of "regenerating the element when at rest."—E. T.

*Improvements in and relating to Galvanic Batteries.* A. Million and P. Bony, Lyons, France. Eng. Pat. 7444, May 13, 1890. 6d.

In a circular containing-cell are placed two co-axial porous tubes, joined together at the bottom so as to form an annular porous cell. In this is placed a cylinder of zinc, while carbon rods are placed inside the innermost cylinder, and more carbon rods outside the outer one. Each set of carbon rods has a lead cap cast on the upper ends, which are first protected by a coating of some inoxidisable material such as gold or platinum.—E. T.

*Improvements in Electric Accumulators or Secondary Batteries, and in the Manufacture and Production of Plates or Elements therefor.* J. Y. Johnson, London. From C. Hering, Paris, France. Eng. Pat. 8578, June 3, 1890. 8d.

The solid peroxide plates used in these cells are made as follows:—The oxide of lead, together with some salt of lead that contracts when converted into peroxide, such as the carbonate, is mixed into a firm paste with a solution of a soluble salt of lead such as the acetate. This is compressed into slabs, and perforated. A little peroxide is added to the other materials to increase the conductivity. The contraction of the carbonate in its conversion to peroxide in the process of formation causes the plate to become porous. The plate may be hardened by immersion in sulphuric acid,

or by the deposition upon it of peroxide of lead by electrolysis in a solution of nitrate of lead. Very porous and tenacious grey plates are produced by compression of the arborescent crystals of lead obtained by the electrolysis of a soluble lead salt such as the nitrate. The plates may be used vertically or horizontally. In the latter case, the order of arrangement is as follows:—Conducting plate of lead, active material, sheet of insulating material; conducting plate, active material usually of opposite polarity, insulating material: this order repeated as far as desired. The whole pile is then bound together between compression plates by bolts and nuts. The current enters and leaves only at the edges of the plates, and in the perforations, which all agree throughout the pile.—E. T.

## XII.—FATS, OILS, AND SOAP MANUFACTURE.

### PATENTS.

*An Improvement in the Manufacture of Soap Powder.* J. Taylor, Leith. Eng. Pat. 8567, May 23, 1889. 4d.

The patentee claims the employment of Irish moss, or a jelly made therefrom, in the manufacture of "soap powder, dry soap, or extract of soap."—O. H.

*Improvements in or appertaining to Presses for Extracting Oil or other Liquids from Seeds or the like.* W. P. Thompson, Liverpool. From A. Schumacher and Tschiffeli, Marseilles, France. Eng. Pat. 8740, May 27, 1889. 8d.

The inventors' system makes it possible to increase at will the pressure on the substance in the extraction cages without increasing the manometrical pressure of the water or other liquid injected into the cylinder. It further dispenses with the "envelopes" of hair, aloë fibre, or esparto. This apparatus enables the operator to get a very much higher proportion of oil at the first pressing, and is claimed to effect a notable economy in the amount of labour and of motive power necessary in oil mills. For details the specification must be consulted.—C. F. C.

*Improvements in Sheep Dips or Washes for Animals.* R. Angus and G. Craig, Cumnock. Eng. Pat. 13,674, August 30, 1889. 4d.

PHENOLOID or like substances are combined with caustic alkali and sulphur. By the term phenoloid the inventors designate phenol or any of its homologues; and they prefer to use "eresol, xylenol, and the like, or the varying mixtures thereof derivable from the tars produced at gas works, blast furnaces, coke ovens, and gas producers."—E. G. C.

*Improvements in Apparatus for Extracting Fatty and other Matters from Substances by means of Volatile Solvents.* H. A. A. Dombrain, Leeds, and O. Trumper, Stratford. Eng. Pat. 18,345, November 16, 1889. 8d.

The inventors' improved apparatus consists of a series of "inclined cells provided with rotating worms and communications such that the material is caused to pass up and down in a zigzag course through the successive cells, while the liquid solvent flows through them in an opposite direction." The extraction cells are combined with a still and condensers, as shown in the annexed figure.—C. F. C.

*An Improved Washing and Cleaning Composition.* J. Bowden, W. C. Penn, and E. L. Downing, Cardiff. Eng. Pat. 1741, February 1, 1890. 4d.

THE inventors claim a mixture of turpentine, peppermint, soap, petroleum, Roumanian bark, soda, water, and scent, as affording a washing composition of quite remarkable properties.—C. F. C.

*Improvements in the Manufacture of Cleansing and Polishing Materials.* J. A. Fisher and J. C. S. Melay, London. Eng. Pat. 4436, March 21, 1890. 4d.

THE patentees prepare materials for such purposes by mixing asbestos powder, soap, ground rock-crystal, and whiting, the specific claim being for the former.—C. F. C.

*Improvements in and Apparatus for the Treatment of Waste Rubber Goods, to recover the Rubber therefrom, and the Production of a New Product by such treatment.* J. Y. Johnson, London. From N. C. Mitchell, Philadelphia, U.S.A. Eng. Pat. 20,289, December 17, 1889. 2s.

THERE are 32 claims and seven sheets of drawings in this specification, which describes in detail methods for separating from the waste stock to be treated (old boots, shoes, &c.), all foreign matters, such as buckles, nails, stiffening fabrics, cotton, wool, and the like, reducing the available stock to fragments of a size convenient for further treatment, devulcanising, by preliminary admixture with calcium sulphide and petroleum, and subsequent treatment with steam under pressure, drying the product, and the production of a devulcanised rubber powder (a new commodity).—E. G. C.

*An Improved Manufacture of Anti-Corrosive Compound.* D. Fulton, Manchester. From D. F. Schmid, Baltimore, U.S.A. Eng. Pat. 5387, April 9, 1890. 4d.

THIS compound is made by mixing molten lead with cotton-seed oil, re-melting, and again mixing, until the oil has absorbed the desired quantity of lead (about 10 lb. of lead to the gallon of oil). The compound may be applied with a sponge or brush, like ordinary paint.—E. G. C.

*An Improved Insulating Compound.* H. H. Lake, London. From A. A. Knudson, Brooklyn, U.S.A. Eng. Pat. 5730, April 15, 1890.

See under XI., page 813.

### XIII.—PAINTS, PIGMENTS, VARNISHES, AND RESINS.

#### PATENTS.

*An Improved Manufacture of Liquid Waterproof Glue.* W. G. Richardson, Bradford. Eng. Pat. 10,900, July 6, 1889. 4d.

THE components are:—Shellac, 12 lb.; naphtha (or spirit), 1 gall.; tar,  $\frac{1}{4}$  lb.; boracic acid, 1 fl. oz.; vegetable oil, 1 fl. oz.; and dye, *q.s.*—E. G. C.

*Improvements in Machinery or Apparatus for Spreading Waterproof or other Plastic Materials or Cements upon Fabrics.* H. Birley and T. Williams, Manchester. Eng. Pat. 12,060, July 30, 1889. 6d.

ACCORDING to this invention, each gauge or spreader is divided into two or more lengths, each of which is suitable for the narrow fabric upon which the plastic material is to be spread; and each line of spreaders swings or oscillates upon a spindle or arbor, passing through the whole line of spreaders, or otherwise so arranged that each length can be independently raised from the fabric in the usual way. A wide machine is thus rendered available for spreading the plastic material simultaneously on two or more narrow widths of fabric.—E. G. C.

*A New Compound to be Used in Paint or Pigment.* R. Condy, London. Eng. Pat. 12,292, August 2, 1889. 6d.

HYDRATED oxide of lead is precipitated, in conjunction with sulphate of lead, from solutions of lead salts; or it is ground and intimately mixed with sulphate of lead.—E. G. C.

*Improvements in Gutta-Percha Compounds, and the Modes and Means of Preparing or Treating the same.* R. Dick, Glasgow. Eng. Pat. 12,756, August 13, 1889. 6d.

THE inventor claims "the manufacture and use of compounds composed of raw gutta-percha, balata, or gutta-percha and balata, with raw india-rubber, either with or without a proportion of crumb or reduced vulcanised india-rubber."—E. G. C.

*Improvements in Waterproof Fabrics for Surgical or Curative Purposes.* E. G. Hughes, Manchester. Eng. Pat. 13,588, August 28, 1889. 4d.

THIS muslin or cambric is treated on both sides with a solution of pure india-rubber, the fabric being then subjected to steam vulcanisation. The product is stated to be much superior to oiled silk, &c.—E. G. C.

### XIV.—TANNING, LEATHER, GLUE, AND SIZE.

#### PATENTS.

*Improvements in and Apparatus or Means for the Tanning of Hides, Skins, or other Leather Goods.* B. Nicholson, South Norwood, and T. Palmer, London. Eng. Pat. 9776, June 13, 1889. 8d.

THIS invention consists in improvements in rotating drums according to which means are provided within the drum for the suspension of the hides to be tanned. The hides are suspended on rods which pass through the length of the drum and through the heads thereof, the points of contact between the rods and the heads of the drum being insulated. Currents of electricity are passed through these rods and thus through the hides, and the electrical connexions are so arranged that the current passes through the rods only when they are not immersed in the tan liquor. It is preferred that the hides should represent the positive pole and the tan liquor the negative. A drawing is given, and for further particulars the specification must be consulted.—B. H.

*A New Liquid or Solid Product applicable for Removing Grease from Washing, Lixiviating and Bleaching all Textile Materials, for the Felling of Cloths, and for Removing Hair from Skins, and similar Operations.* J. L. Moret, Paris, France. Eng. Pat. 13,023, Aug. 17, 1889. 6d.

See under VI., page 805.

*An Improved Shoemaker's Cement.* J. Saunders, London. Eng. Pat. 1727, February 1, 1890. 4d.

This cement is to be used for attaching leather or dermatine soles to boots and shoes, for splices, and for belts of every description. The cement is prepared by mixing together the following five ingredients in the proportions given: Methylated ether, 1 lb.; carbon bisulphide, 8 lb.; raw gutta-percha, 1 oz.; elastic glue, 1 oz.; india-rubber tissue, 1 lb. The mixture is left for 12 hours, and after being agitated for one hour is fit for use. The mode of application is to roughen the surfaces of the articles to be united, to apply one or two coats of the cement, allow to dry, warm gently, press both articles firmly together, and tap with a hammer.—B. H.

*Improvements in Machinery for Dressing Skins or Hides.* C. A. Brown, Exeter. Eng. Pat. 6115, April 22, 1890. 6d.

This invention relates to an improved mode of mounting the tools used in striking, scouring, and fleshing skins or hides, by means of which the tools shall bear evenly throughout their traverse on the skins or leather. In operating upon the skin it is usual to have a double set of tool holders and to work the tools in opposite directions from the centre to the opposite edges of the skin, and a description, with drawing, is given of the means employed for raising, depressing, and traversing the tool holders in opposite directions over the work, and for maintaining their parallelism with the work while their respective sets of tools are in action.—B. H.

## XVI.—SUGAR, STARCH, GUM, Etc.

*Further Contributions to a Knowledge of Melitriose and Melibiose.* C. Scheibler and H. Mittelmeier. Ber. 23, 1438—1443.

In a previous contribution (Ber. 22, 1678; this Journal, 1889, 718) the authors have described some of the properties of melibiose, a disaccharide which they obtained by the limited inversion of melitriose (raffinose). They have not yet succeeded in crystallising it. A combustion of the sugar gave numbers pointing to the formula  $C_{12}H_{22}O_{11}$ . Two determinations of the specific rotatory power gave  $[\alpha]_D = 126.7^\circ$  and  $[\alpha]_D = 127.9^\circ$ .

Melibiosephenylhydrazine was prepared by dissolving as much phenylhydrazine as possible in a thick syrup of the sugar. After standing some days, the syrup was dissolved in alcohol, and the compound precipitated by ether and crystallised from hot alcohol. Its formula is  $C_{13}H_{23}O_{10}N$ . It is insoluble in ether, benzene, chloroform, easily soluble in water, and difficultly in alcohol. It melts at about  $145^\circ$ . Its aqueous solution, heated with phenylhydrazine acetate, yields phenylmelibiosazon.

Oetaacetylmelibiose,  $C_{12}H_{14}O_{11}(C_2H_3O)_8$  is prepared by heating the dry sugar with acetic anhydride and sodium acetate. It crystallises from hot alcohol in large well-formed needles, radiating from a common centre. It melts at  $170^\circ$ — $171^\circ$ ; this is rather noteworthy, as the oetaacetyl compound of lactose, which melibiose closely resembles, melts at  $86^\circ$ . It is almost insoluble in cold water, rather more soluble in hot water, from which it crystallises on cooling. It is easily soluble in chloroform, also in hot alcohol, in glacial acetic acid, and benzene, less easily in ether and difficultly in carbon disulphide and petroleum spirit. Dissolved in a mixture of alcohol and chloroform its specific rotatory power was  $[\alpha]_D = 94.2$ . It reduces Fehling's solution, and is without action on phenylhydrazine, just as with the pentaacetyl compounds of dextrose, galactose, and levulose.

In order to throw further light on the constitution of melitriose, an acetyl derivative was prepared in the usual way; its composition was found to be  $C_{13}H_{20}O_{10}(C_2H_3O)_8$ . This compound does not reduce Fehling's solution, it is easily soluble in cold absolute alcohol and very easily in hot, and separates out on cooling as an amorphous mass. It behaves in the same way with ether. It is also soluble in

phenylhydrazine, aniline, chloroform, benzene, glacial acetic acid, and slightly in carbon disulphide and petroleum ether. From all the solutions it separates in the amorphous state. It melts at  $99^\circ$ — $101^\circ C.$ , and in alcoholic solution its specific rotatory power is  $[\alpha]_D = 92.2^\circ$ .—A. L. S.

*Extraction of Raffinose from Molasses.* L. Lindet. Bull. Soc. Chim. 1890, 682—685.

THE methods proposed by Scheibler (this Journal, 1885, 607), Lippmann (this Journal, 1885, 356), and Pellett (Jour. fab. Sucre, 1885, 22) have the common defect of treating the molasses without previous clarification. Treatment with subacetate of lead (as in Lippmann and Pellett's methods) is useless since the acetic acid left after precipitating the lead would invert the sugars during the evaporation of the liquors. The author proceeds as follows:—The molasses diluted with 5 or 6 parts of water is agitated in the cold with about 5 per cent. by weight of mercuric sulphate, filtered, the sulphuric acid precipitated with baryta water, and the slightly alkaline liquor boiled for some time to remove the last traces of mercury as basic salt, concentrated to a syrupy consistency in a vacuum and taken up with concentrated methyl alcohol. The solution, which is only slightly coloured, contains the sugars, the separation of which depends upon the following differences of solubility:—

100 cc. of the Solvent take up Grms. of	Methyl Alcohol.	Ethyl Alcohol.			
	Anhydrous.	$95^\circ$	$90^\circ$	$85^\circ$	$80^\circ$
Saccharose.....	0.4	0.30	1.00	2.23	6.20
Raffinose .....	9.5	0.06	0.08	0.10	0.21

The solution is heated in a flask connected by a worm with a second flask filled with quicklime and attached to a condensing apparatus leading back into the first flask. Both flasks are heated on the water-bath, and whilst both alcoholic and aqueous vapours pass into the quicklime only anhydrous alcohol is returned, the solution is thus gradually dehydrated and the saccharose deposited. As complete dehydration is impossible on account of the energy with which the sugars retain water, the mother-liquor is treated with ethyl alcohol, and the syrupy precipitate of the mixed sugars crystallised from alcohol of  $80^\circ$ — $85^\circ$ . The whole of the saccharose may in this way easily be left in solution.

The manner in which the presence of either of these sugars increases the solubility of the other, and the modification of the crystalline form of saccharose effected by a small quantity of raffinose renders the existence of some compound of the two sugars very probable.—S. B. A. A.

*On the Products of the Saccharification of Starchy Materials by Acids.* G. Flourens. Compt. Rend. 110, 1204—1206.

THE author has examined the products formed at different stages of the saccharification of starch by dilute acids, and finds that their cupric reducing power and optical activity agree with the supposition that only dextrose and dextrin and no maltose are formed.—A. L. S.

## PATENT.

*Improvements in Multiple-Effect Evaporating Apparatus for Evaporating or Concentrating Sugar Juice and other Liquids.* W. Maxwell, Djoeja, Java. Eng. Pat. 12,809, August 14, 1889. 8d.

See under 1., page 794.

## XVII.—BREWING, WINES, SPIRITS, Etc.

*On the Bouquet of Wines and Spirits.* A. Rommier.  
Compt. Rend. 110, 1039—1041.

THE author has previously shown (Compt. Rend. 99, 879; this Journal, 1885, 462) that if ellipsoidal yeast be added to pressed grapes at a temperature below  $21^{\circ}$ — $22^{\circ}$  C., it plays the chief part in the subsequent fermentation to the exclusion of the natural yeast, but if the temperature be above  $21^{\circ}$ — $22^{\circ}$  the ellipsoidal and natural yeast develop side by side, the artificially introduced yeast still modifying the character of the wine.

Martinand (Bull. de l'Assoc. des Chimistes de sucrerie et de distillerie, 1889, 184) has sterilised grapes by immersion in 5 per cent. bisulphite of soda and after washing pressed them in the usual way and fermented the juice with yeast from (1) spontaneously fermenting cherries, (2) wine of Beaujolais, (3) Burgundy, (4) Champagne, (5) Bordeaux. The wine from each fermentation had a different flavour. The author has made similar experiments to determine if these differences of flavour extend also to the spirits distilled from these wines. A solution of cane sugar with suitable yeast food was fermented with yeast from different vineyards. The alcohols obtained by distillation had all a different character.—A. L. S.

*Invertase: a Contribution to the History of an Enzyme or Unorganised Ferment.* C. O'Sullivan and F. W. Thompson.  
Proc. Chem. Soc. 1890 (86), 107—112.

THE substance present in yeast, &c., known as *invertase*, which possesses the power, under suitable conditions, of inducing the hydrolysis of cane-sugar, has been studied by the authors with the object of determining the precise manner in which it acts, and its constitution.

The method adopted in determining the functions of invertase is essentially that developed by Vernon Harcourt in his paper "On the observation of the course of chemical change." A solution of cane-sugar was mixed with a measured amount of invertase, and the action was allowed to take place at a known temperature during a definite time; action was then stopped by the addition of alkali, and its extent determined.

The authors arrived at the following conclusions:—

1. The rate of hydrolysis of cane-sugar by means of invertase may always be expressed by a definite time-curve; this curve is practically that given by Harcourt as expressing a chemical change "of which no condition varies excepting the diminution of the changing substance." There are, however, some slight and apparently constant deviations from the theoretical curve.

2. Whatever the conditions may be under which hydrolysis is taking place, as long as these conditions remain unchanged, this curve is adhered to.

3. When the degree of acidity is that most favourable for the action of invertase, the rapidity of the action is in proportion to the amount of invertase present.

4. The most favourable concentration of the sugar solution at a temperature of  $54^{\circ}$  C. is about 20 per cent. Below that there is a rapid decline in the speed of hydrolysis. Greater concentrations are only slightly less favourable until about 40 grms. per 100 cc. is reached. In saturated solutions hydrolysis only proceeds with extreme slowness.

5. The speed of hydrolysis increases rapidly with the temperature until  $55^{\circ}$ — $60^{\circ}$  C. is reached. At  $65^{\circ}$  C. the invertase is slowly destroyed, and at  $75^{\circ}$  C. it is immediately destroyed. At the lower temperatures the speed of the action increases with rise of temperature in accordance with Harcourt's law: the rate being about doubled for  $10^{\circ}$  rise; but above  $30^{\circ}$  C. the increase is not nearly so rapid.

6. Elevated temperatures have no permanent effect on the activity of the invertase so long as they are not sufficiently high to destroy it.

7. The caustic alkalis, even in very small proportions, are instantly and irretrievably destructive of invertase.

8. Minute quantities of sulphuric acid are exceedingly favourable to the action, but a slight increase of acidity

beyond the most favourable point is very detrimental. The most favourable amount of acid increases to some extent with the proportion of invertase, and decreases with rise of temperature, but the authors have not been able to discover on what it depends.

9. In studying the action of invertase it is of the utmost importance that the most favourable amount of acid should be employed; otherwise correct results cannot be obtained. At a temperature of  $60^{\circ}$  C. the action is almost stopped unless exactly the right amount of acid is used, whilst if this factor is properly adjusted hydrolysis proceeds at (probably) the maximum speed.

10. The influence of alcohol varies in direct proportion with the amount present. Five per cent. of alcohol decreases the speed of the action by about one-half.

11. The dextrose formed by the action of invertase is initially in the birotatory state.

12. The optical activity of a solution undergoing hydrolysis is no guide to the amount of hydrolysis that has taken place.

13. If a caustic alkali be added to a solution undergoing hydrolysis, and the optical activity be allowed sufficient time to become constant, it is a true indicator of the amount of inversion that had taken place at the moment of adding the alkali.

14. A sample of invertase which had induced hydrolysis of 100,000 times its own weight of cane-sugar was still active.

15. Invertase itself is not injured or destroyed during its action on cane-sugar.

16. There is no limit to the amount of sugar which can be hydrolysed with the aid of invertase.

17. The hydrolysis of cane-sugar by means of invertase is a simple chemical change differing in no important way from those which inorganic substances undergo.

18. The products of hydrolysis have no influence on the rate of the action.

19. A solution of invertase will withstand a temperature  $25^{\circ}$  C. higher in the presence of cane-sugar than in its absence.

20. The authors are of opinion that when invertase inverts cane-sugar, combination takes place between the two substances, and that the invertase remains in combination with the invert-sugar. This combination breaks up in the presence of molecules of cane-sugar.

21. A means of estimating the activity of a material containing invertase has been devised, which consists in recording the result by means of the time factor  $\pm 0 = x$  min. In this equation  $\pm 0 =$  a certain definite amount of work and  $x =$  the time necessary to perform it. The expression means that the given inverting material takes  $x$  minutes to invert a standard amount of cane-sugar under standard conditions. The number  $x$  varies in inverse proportion to the actual amount of invertase contained in the material or materials under examination.

22. If sound brewers' yeast be pressed and then kept at the ordinary temperature for a month or two, it does not undergo putrefaction, but changes into a heavy yellow liquid; the product possesses no power of fermentation, but an apparent increase takes place in the invertive power.

23. From such liquefied yeast it is easy to filter off a bright solution of high hydrolytic power. It is shown that all the invertase of the yeast is in this solution, which is termed yeast liquor.

24. Yeast liquor has a relative density of about 1080. It will remain for a long time unaltered, excepting that the colour darkens. If exposed to the air it may slowly become covered with mould.

25. If spirit be added to yeast liquor until the mixture contains 47 per cent. of alcohol, the whole of the invertase separates with only a slight loss of power. This precipitated invertase may be washed with spirit of the same strength and then the residue either dehydrated with strong alcohol and dried *in vacuo*, or else it may be extracted by means of 10 to 20 per cent. alcohol and then filtered. The filtrate contains the invertase. On one occasion the extent of the loss involved by this process was determined, and it was found that all the invertase of the yeast liquor was present in the filtrate except 12.3 per cent.

26. The authors have not succeeded in further purifying invertase preparations carefully made in this manner. The slightest attempt at purification destroys the invertase.

27. They have prepared invertase almost free from ash.

28. The inverting power of pressed English yeast varies from  $0 = 1000''$  to  $\pm 0 = \pm 3000''$ , or about one-third of these amounts if calculated on the dry solid matter of the yeast.

29. The inverting power of the most active invertase preparation made was  $\pm 0 = 25 \cdot 1$  on the dry solid matter. It is believed that pure invertase would approximately have  $\pm 0 = 22 \cdot 5$  min.

30. The dry solid matter of yeast contains from 2 to 6 per cent. of invertase. 5·8 per cent. of invertase was separated from one sample of yeast.

31. During the preparation of invertase from yeast liquor an albuminoid is obtained, which is not redissolved by water. This is termed yeast albuminoid.

32. Invertase, when it approaches a pure state, is a very unstable substance. The products of its decomposition have been carefully examined and are found to constitute a new series of substances belonging to the *invertan* series.

33. The invertan series is a homologous series of substances which on analysis yield numbers which may be expressed in terms of an albuminoid and a carbohydrate. Seven members of the series are described.

34. The authors consider that invertase itself is a member of the invertan series, and they call it  $\beta$ -invertan.

35. If the products of the decomposition of  $\beta$ -invertan are examined, it is usually found that they consist of  $\alpha$ - and  $\delta$ -invertan. The former contains more, and the latter less, nitrogen than invertase.

36.  $\alpha$ -Invertan is insoluble in water, and in all its other properties seems to resemble yeast albuminoid. It contains 8·35 per cent. N. It is a very stable substance.

37.  $\beta$ -Invertan or invertase is soluble in water, and is the only member of the series which has the power of inverting cane-sugar. It contains about 3·69 per cent. N., and its optical activity is  $[\alpha]_D = + 80^\circ (?)$ .

38.  $\gamma$ - and  $\delta$ -invertan are the products of the simplification of invertase. One or other of these two substances seems invariably to be formed. They both contain less nitrogen than invertase, and are always accompanied by  $\alpha$ -invertan. Both are readily soluble in water. They contain respectively 3·15 and 2·43 per cent. N., and their optical activity is  $[\alpha]_D = + 45^\circ$  and  $+ 54^\circ$ .

39.  $\epsilon$ -Invertan is formed from the slow breaking down of  $\delta$ -invertan. At the same time an insoluble substance, resembling  $\alpha$ -invertan, is formed. This probably consists of another member of the series coming between  $\alpha$ - and  $\beta$ -invertan.  $\epsilon$ -Invertan is soluble in water, and has  $[\alpha]_D = + 65^\circ$ . It contains 2·07 per cent. N.

40.  $\zeta$ -Invertan results from the splitting up of  $\epsilon$ -invertan in the same way that the latter is formed from  $\delta$ -invertan. Its optical activity is  $[\alpha]_D = + 75^\circ$ , and it yields 1·61 per cent. N.

41.  $\eta$ -Invertan is formed by the action of boiling sulphuric acid on  $\zeta$ -invertan. It contains less nitrogen than the latter substance, but the substance has not yet been sufficiently investigated to give reliable figures.

42. The further products of the action of sulphuric acid on  $\zeta$ -invertan are two soluble substances, one containing a considerable amount of nitrogen, and the other with little or no nitrogen, a high cupric reducing power and a low (dextrorotatory) optical activity.

43. The properties of all the members of the series, except  $\alpha$ -invertan, are very similar. They are all soluble in water, forming bright solutions, which do not cloud on boiling. They are all readily thrown out of solution by alcohol, provided a little acid is present; the precipitates so formed are transparent colourless syrups, miscible with water in all proportions. The solutions are all dextrorotatory.

44. All the members of the invertan series, except  $\eta$ -invertan, yield a pink colouration on boiling with Millon's reagent.

45. All the members of the invertan series, except  $\alpha$ -invertan, when submitted to the action of an alkaline copper solution, readily yield a very characteristic copper compound, from which the invertan (except  $\beta$ -invertan)

may be separated unaltered. Invertan can probably form several such copper compounds, all having a similar appearance, but affording different percentages of copper oxide.

46. In the presence of a very large excess of alkali, a copper compound is also formed from  $\alpha$ -invertan, but on examination it is found that the  $\alpha$ -invertan has been split up into an albuminoid and  $\zeta$ -invertan, the copper compound being that of  $\zeta$ -invertan.

47. It is believed that the members of the invertan series are combinations of yeast albuminoid with  $\eta$ -invertan, and that yeast albuminoid itself is probably a combination of yeast albuminoid with a carbohydrate.

48. According to this theory the composition of the carbohydrate present in the invertan series, calculated from the average of all the analyses, would be C = 43·22 per cent. and H = 6·28 per cent. These numbers agree very closely with those required by a hypothetical carbohydrate coming midway between the *in* and the *on* groups.

49. It is thought that  $\eta$ -invertan contains 18 parts by weight of this carbohydrate to 1 of albuminoid, and that  $\alpha$ -invertan contains 3 parts of the carbohydrate to 4 of albuminoid.

50. The other members of the series are formed by the union of these two substances according to the general formula  $\eta + \alpha_n$ , where  $\eta$  represents  $\eta$ -invertan and  $\alpha$  represents  $\alpha$ -invertan. In this way we look upon invertase ( $\beta$ -invertan) as  $\eta\alpha_3$ . This splits up into  $\alpha$ - and  $\gamma$ -invertan according to the formula  $\eta\alpha_3 = \eta\alpha_1 + \alpha$ .  $\eta\alpha_1$  represents  $\gamma$ -invertan, and this may be further transformed by elimination of  $\alpha$  into  $\eta\alpha_2$ , or  $\delta$ -invertan.

60. The numbers calculated for the constitution of the above theoretical homologous series agree with considerable closeness with those obtained from the analysis of members of the invertan series.

61. The number obtained from a single determination by Raoult's freezing process for the molecular weight of  $\zeta$ -invertan is considerably less than the possible molecular weight of  $\zeta$ -invertan, according to the authors' theory of its constitution.

## XVIII.—CHEMISTRY OF FOODS, SANITARY CHEMISTRY, AND DISINFECTANTS.

### (A.)—CHEMISTRY OF FOOD.

#### PATENTS.

*Improvements in the Method of and Apparatus for Preparing Milk so as to Enable the same to be Preserved Indefinitely.* J. F. H. Gronwald and E. H. C. Oehlmann, Berlin, Germany. Eng. Pat. 6280, April 12, 1889. 11d.

THE milk if acid is neutralised with potassium bicarbonate. It is then placed in a pan with a closely fitting lid and steam jacket. It is then heated to  $100^\circ\text{C}$ . by admitting steam into the jacket and into the space above the milk. The vessels (usually bottles) into which the sterilised milk is to be transferred are first sterilised by means of steam in a chamber specially constructed for the purpose; the milk is then forced over from the pan into a vessel which is situated in the chamber above mentioned, and from this vessel it is run into the bottles. By means of an ingenious arrangement the bottles are closed hermetically with stoppers whilst still in the chamber. Glazed slots in the sides of the chamber enable one to observe what is going on inside. For filling larger vessels, such as casks, a modified chamber is employed but the principle is the same. 100 litres of milk can thus be worked up at a time.

—F. W. T. K.



*Improvements in the Preparation and Combination of Animal Substances with Vegetable Substances for Use in Food.* J. L. Johnston, London. Eng. Pat. 8945, May 29, 1889. 4d.

The animal ingredients, such as powdered meat, albumen, meat peptone, and gelatin or other suitable thickening ingredient are thoroughly mixed with chocolate, cocoa, or coffee extract, and heat is applied so as to reduce the whole to a solid consistency. By means of suitable compressing apparatus lozenges or tablets are prepared. The cocoa and chocolate are preferably previously deprived of their vegetable fat, but flake cocoa or an infusion at once from the nibs may be used. The infusion of coffee is filtered from the residual grounds before being incorporated with the meat preparation above mentioned.

*Soluble Saccharin* is employed to sweeten the preparations if this is necessary. For use the preparation is infused with hot water so as to make a broth.—F. W. T. K.

*Improvements in Food Preparations.* G. A. Brinck and A. W. Rehnström, Stockholm, Sweden. Eng. Pat. 10,352, June 25, 1889. 6d.

This food, which is intended for human beings, horses, dogs, and other animals, is prepared in the form of cakes, bread, biscuits, puddings, and sausages. The ingredients are varied both in quality and quantity according to special requirements. The principal ingredient which is used throughout is dried and crushed milk curds (casein). Other ingredients are, flour or any suitable form of meal, butter, lard, fat, or other animal or vegetable ingredients, and mineral matters (usually in the proportion of 1 to 3 per cent.), bone flour being especially desirable.

—F. W. T. K.

*Improvements relating to the Manufacture of Butter and to Apparatus therefor.* H. H. Lake, London. From F. R. Nörlöw, Copenhagen, Denmark. Eng. Pat. 12,360, August 3, 1889. 8d.

The object of this invention is to produce butter which shall be free from the milk-sugar and albuminous substances contained in the milk. The cream or the fresh milk is centrifugally forced through a perforated or porous material such as wire gauze, woven fabric, clay.—F. G. C.

## (B.)—SANITARY CHEMISTRY.

### PATENTS.

*Improvements in the Clarification or Purification of Sewage and other Foul Waters of Analogous Nature.* C. H. Harvey, London. Eng. Pat. 6994, April 26, 1889. (Second Edition.) 6d.

This invention consists in the use of "acid ferrie salts prepared from waste sulphide of iron, in conjunction with an alkaline earth."—F. G. C.

*An Improved Process and Means to be Employed for Partially Destroying and Mixing Night-Soil and other Refuse, and for Converting the same into Manure suitable for Grass Land and Arable Purposes.* J. W. Lodge, Sowerby Bridge. Eng. Pat. 9087, June 1, 1889. 6d.

By means of a series of reciprocating and revolving riddles and rollers, combustible and metallic portions of the town refuse are separated; the former are burnt, the latter are destroyed in a cupola, the saleable portions, however, being recovered. The residual portion is crushed and ground and finally mixed off with excreta and urine, the mixture being deodorised with sulphuric acid and dried by the addition of

gypsum. Mixed excreta and ashes and other refuse found in old ash-places and privies are subjected to a preliminary treatment of blending and mixing, being at the same time rendered friable, before they are passed through the above apparatus and finally made into manure.

The machinery may also be used for grinding and preparing basic or other slag, cinders, and cement, in fact, anything which needs to be pulverised and separated. This improved machinery may also be used for mixing materials for the manufacture of briquettes made from coal dust in combination with tar and other ingredients.—F. W. T. K.

*Improvements connected with the Filtration of Water and in Cleansing the Filters.* G. W. Hawksley, Sheffield. Eng. Pat. 9512, June 8, 1889. 8d.

INSTEAD of purifying the water by mixing precipitants with it and running the mixture into settling tanks, the precipitants are admitted into the water as it passes through the suction pipe of a pump or pumps which are employed to force the mixture through a series of filters. One advantage claimed for this method is economy of space. For cleansing the filters the inventor does not proceed in the usual way, *i.e.*, by reversing the flow of unfiltered water, but forces the water, filtered by one or two of the filters, through the remaining ones in the reverse direction. Or instead of using water for the purpose he blows steam through the filters in the reverse way or charges them with steam and then suddenly blows it off. This breaks the deposit and causes it to crumble, and it is then carried off through the sludge pipe in which test taps or gauge glasses are fixed for the purpose of watching the cleansing process. An intermediate high pressure tank for the purpose of more thoroughly mixing the ingredients and retaining the grosser deposit and floating material may be inserted between the pump and the filters.

Charcoal, asbestos cloth, wire gauze or other materials supported in one or more layers by perforated filter plates fixed inside the filters are used as filtering media.

—F. W. T. K.

*Improvements in Apparatus for Purifying Water.* W. Anderson, Westminster. Eng. Pat. 10,706, July 2, 1889. 8d.

This is an improvement on Eng. Pats. 5496 of 1883 and 12,256 of 1884 (see this Journal, 1884, 529, and 1885, 133, 363, 546). The water to be purified is admitted into one end of a revolving cylinder on the inside of which are fixed curved radial shelves instead of flat radial shelves, as heretofore used. These shelves are made up of short lengths, about 3 ft., and serve to hold up the iron which is the purifying agent, and by placing them in echelon instead of in lines parallel to the axis throughout, a more uniform stress is brought to bear on the driving machinery, and the iron is more thoroughly brought into contact with the water. The curved plates hold more iron than the flat ones. The water before and after passage through the cylinder is aerated by means of jets of air passed in through perforated pipes. A perforated pipe also passes along the axis of the cylinder, and the air forced through this aerates the water during its passage through the same. This aeration promotes the chemical action which takes place. The excess of air is allowed to escape by means of an automatic valve in the cylinder case. The entrance to the outlet pipe is covered with a hood, to prevent iron being carried away. It is sometimes necessary to keep the iron in more compact masses. Instead of using longitudinal shelves, the cylinder is then divided into transverse cells with perforated sides. These are filled with iron, and the water is made to flow in a sinuous manner between these cells, so as to have as long a contact as possible. The cells are filled through apertures in the cylinder sides, which are closed by means of doors.

—F. W. T. K.



*A System of Purification and Filtration of Water for City and Town Supplies.* J. A. Crocker, Brooklyn, U.S.A. Eng. Pat. 5445, April 10, 1890. 8d.

IN combination with a water supply, this inventor uses a battery of filters, one or more interposed precipitating basins, a precipitating solution tank situated nearest the inlet of the basin supply, and one or more pumping, lifting, and reservoir-distributing stations.—E. G. C.

### (C)—DISINFECTANTS.

#### PATENT.

*A Composition for Arresting or Preventing Decay in Wood.* J. T. McMahon, London. Eng. Pat. 13,203, August 21, 1889. 4d.

THE components of this preparation are as follows: Mercuric chloride,  $1\frac{3}{4}$  oz.; ammonium chloride, 3 oz.; soft soap, 5 lb.; methylated spirit, 2 galls.; distilled water, 1 gall.; Venice turpentine, 10 oz.—E. G. C.

## XIX.—PAPER, PASTEBOARD, Etc.

#### PATENTS.

*Improvements in the Manufacture of Paper.* J. Craig, Cheadle. Eng. Pat. 9145, June 3, 1889. 8d.

THIS invention relates to the production of duplex or triplex paper having suitable designs impressed upon it. For this purpose a web of paper is passed between a pair of metal rollers furnished with punches, whereby the design is punched out. This paper is then united with a web of paper in process of manufacture, as in the ordinary method of making duplex paper. Three sheets can also be so united to form a triplex paper.—E. J. B.

*Improvements in Machinery or Apparatus used in the Manufacture of Paper and Cardboard.* A. Durand, Paris, France. Eng. Pat. 11,779, July 24, 1889. 11d.

THIS invention relates to improvements in the driving of paper and cardboard machines, whereby greater uniformity and increased speed are obtained. For this purpose the power is transmitted by a system of cones instead of spur gearing. The inventor also claims the utilisation of exhaust steam for heating the drying cylinders by means of a novel apparatus. Complete drawings accompany the specification.—E. J. B.

*Improvements in Apparatus for Cutting up Wood into Discs for use in making Wood Pulp.* H. Wigger, Unna, Germany. Eng. Pat. 14,563, September 16, 1889. 6d.

INSTEAD of cutting up the wood into discs with saws, as at present practised in the Mitscherlich process of manufacturing wood pulp, the inventor causes the logs of wood, previously barked, to be acted upon by a revolving knife. The wood is presented to the knife obliquely, both as regards the vertical and horizontal planes. Complete drawings accompany the specification.—E. J. B.

*Improvements in the Process of and Means for Purifying Paper Pulp.* C. Kellner, Vienna, Austria. Eng. Pat. 4960, March 29, 1890. 8d.

ACCORDING to this invention paper pulp largely diluted with water (1–15 oz. per gall.) is caused to flow into a rapidly revolving cylinder placed inside a fixed cylinder. As the inner cylinder revolves the heavy impurities are thrown towards the sides, while the purified pulp flows away in a continuous stream from the centre. As the

impure pulp enters at the bottom of the cylinder in a continuous stream, the impurities gradually rise along the sides and overflow into the outer cylinder, from which they can be from time to time drawn off.—E. J. B.

*An Improved Process of Softening Brittle Paper Pulp.* C. Kellner, Vienna, Austria. Eng. Pat. 5053, April 1, 1890. 8d.

THE inventor causes the pulp to pass into the space between two cones, one of which is rapidly revolving. The cones are covered with helical flutings. Pulp-treating in such an apparatus is more uniform than that produced by the ordinary beating engines. In another form of apparatus the pulp is caused to flow between two horizontal millstones.—E. J. B.

*Improvements in Apparatus for Use in the Manufacture of Wood-Wool.* Ole Evenstad and Olaus Senstad, Rasten Storelvedalen, Norway. Eng. Pat. 8422, May 30, 1890. 6d.

AN apparatus is described for cutting wood shavings into longitudinal strips. It is specially designed for use in combination with the machine patented by the same inventors (Eng. Pat. 4296 of 1888).—E. J. B.

## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

*The Oxidation of Turpentine in Sunlight.* H. E. Armstrong. Proc. Chem. Soc. 1890 (85), 99–101.

It was pointed out by Sobrero in 1851 (Compt. Rend. **33**, 66) that when turpentine is exposed to light in presence of moisture and oxygen a crystalline substance is formed which has the composition represented by the formula  $C_{10}H_{16}O_2$ ; and that this substance is decomposed when boiled with dilute sulphuric acid, an oil being formed which has a powerful odour recalling both that of camphor and that of turpentine. The author's attention became directed to this substance about 12 years ago in the course of his studies of the terpenes and camphor, and in most years since, during the summer, he has carried on experiments on the oxidation of  $C_{10}H_{16}$  hydrocarbons in sunlight, and has been able to confirm Sobrero's statements in every particular. As the crystalline product in question has not yet been named, it is proposed to term it—at all events, provisionally and until its constitution is determined—sobrerol.

Sobrerol is readily soluble in alcohol, and crystallises from this solvent usually in large, flexible, monosymmetric prisms having a peculiar hour-glass structure inside and showing hemihedrism. It is slightly soluble in water, benzene, chloroform and petroleum spirit; the aqueous solution has a bitter taste. It melts at about  $150^\circ$ . The formula is  $C_{10}H_{16}O_2 = C_{10}H_{16} + H_2O_2$ , and there can be very little doubt that it is a glycol. Sobrerol is optically active in a high degree, the apparent specific rotatory power of the products from French turpentine in a 5 per cent. solution in alcohol (b.p.) being slightly above  $150^\circ$ . Sobrerol from American turpentine (from Savannah) was found to have about the same rotatory power, but in the opposite direction. The optical similarity of the two products is noteworthy, inasmuch as the American has less than half the rotatory power of French turpentine. It would seem that only the terpenes proper, and not the citrenes, &c. (Chem. Soc. Trans. 1879, 734; this Journal, 1882, 478) form sobrerol.

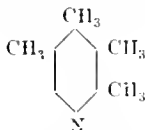
When boiled with dilute sulphuric acid, sobrerol is readily converted into the oil referred to by Sobrero; the product is undoubtedly identical with the isomeride of camphor which Wallach and Otto have obtained by treating turpentine

with nitrous acid (Annalen, 253, 249), and which they have provisionally named *pinol*: as the compound is not an oil, i.e., an alcohol, it may be suggested that it might appropriately be termed *sobrerone*. The product from sobrerol begins to boil at  $150^{\circ}$ – $160^{\circ}$ , but passes over almost entirely at about  $183^{\circ}$ , leaving a small amount of a viscid oil; it readily combines with bromine, forming a dibromide (bromine found 50.78 per cent.) which crystallises very beautifully in forms of the rhombic system. The dibromide was found to melt at  $93.5^{\circ}$ .

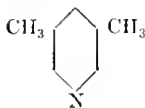
Sobrerol is probably always the initial product of oxidation of turpentine. It may be expected that soberone will be found among the oxygenated constituents of some essential oils, and it is proposed to search for it.

*A New Lutidine.* E. Dürkopf and H. Göttisch. Ber. 23, 1113–1114.

This base was obtained from dimethylpyridine carboxylic acid, itself obtained by the oxidation of the parvoline, boiling at  $216^{\circ}$ – $217^{\circ}$ , and proved by the authors to have the constitution represented by the formula—



(Ber. 23, 1110–1112). It was formed by eliminating carbonic anhydride, boils at  $169$ – $170^{\circ}$ , and has the composition  $C_7H_9N$ , agreeing with that of lutidine. It is a colourless highly refractive liquid, having a feeble and not unpleasant odour. It dissolves sparingly in cold, more readily in hot water. It has a specific gravity of 0.9614 at  $0^{\circ}$  (water at  $4^{\circ}$ =1). The *mercuriochloride* crystallises in fine long sparingly soluble needles, melting at  $170^{\circ}$ . The same base is formed in small quantities by the action of propaldehyde-ammonia on propaldehyde, it being found possible to isolate from the fraction boiling at  $170^{\circ}$ – $180^{\circ}$  a base agreeing in all its properties with the above lutidine. In order to determine the constitution of the latter, the base was subjected to oxidation, a product being obtained the analysis of which showed it to be a pyridinedicarboxylic acid. It melts at  $314^{\circ}$ – $315^{\circ}$ . From its high melting point and external character the authors infer that the acid is dimaleic acid, so that by regarding the above lutidine as a  $\beta\beta$ -dimethylpyridine its constitution is represented by the formula—



—D. B.

*On the Composition of Digitonin.* H. Kiliani. Ber. 23, 1555–1560.

DIGITALIN of commerce contains besides digitoxin (used in pharmacy and not yielding any sugar by heating with acids) at least three glucosides: digitonin, digitalin, and digitalein, of which the first is present in greatest quantity and appears to be identical with saponin. It has been observed by various observers that digitalin when heated with acids yields bodies capable of reducing cupric oxide, and Schmiedeberg has observed that there is also formed a crystalline body insoluble in water which he named *digitogenin*. He described some of the properties of this body, but did not determine the nature of the sugars formed. The author has made further experiments to determine the nature of these bodies.

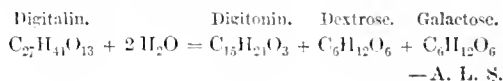
Some commercial digitalin (pur. pulv.) was obtained; it was almost completely soluble in water. Emulsion was without action on it. It was treated with its own weight of hydrochloric acid (1.19) diluted with 10 times its weight of

water, at the boiling point for six hours. As the reaction progressed an insoluble substance separated out, which at the conclusion was filtered and washed with water.

The filtrate was treated with silver oxide to remove hydrochloric acid and evaporated to a syrup; on exposure over sulphuric acid in a desiccator, it solidified to a crystalline mass. This was treated with boiling methyl alcohol, and a sugar was left, which was found to be galactose, the methyl alcohol solution was found to contain dextrose and a little more galactose. About 16 per cent. of each sugar was obtained. The insoluble body was about 50 per cent. of the digitalin taken. It was purified by dissolving in chloroform, distilling this off and treating the residue with a little boiling alcohol, which dissolved out most of the dirt and recrystallising the residue from 30–40 times its weight of boiling alcohol. To determine if this was a pure body, it was fractionated into four portions, and these were found to be identical. A determination of the carbon and hydrogen pointed to the formula  $(C_{15}H_{21}O_3)_x$ ; the composition of crystallised derivatives of digitogenin to the formula,  $C_{15}H_{21}O_3$ .

Digitogenin is soluble in 35 parts of boiling and in more than 100 parts of cold 93 per cent. alcohol; in 20 parts of boiling and 30 parts of cold chloroform; in 30 parts of cold glacial acetic acid, and in rather less of hot, and it is insoluble in water and aqueous potash. With alcoholic potash it forms a crystalline potash compound. Phenylhydrazine does not act on it, nor does a hot concentrated solution of baryta form any well marked compound, whilst mineral acids and oxidising agents act energetically.

The reaction is expressed by the following equation—



*Manufacture of Tannins free from Colouring Matter.* A. Villon. Bull. Soc. Chim. 1890, 784–786.

To prepare tannin free from colouring matters from chestnut wood, quebracho, divi-divi, sumac, and other astringents, an extract is first obtained in the usual way, using six vats and a current of carbonic acid, at a temperature of  $80^{\circ}$ – $90^{\circ}$ , this is cooled in ordinary settling vats, the liquor drawn off, 0.5 per cent. of zinc sulphate added, the temperature is then maintained for half an hour at  $+2^{\circ}$  by means of a refrigerating vat similar to those used in breweries; the precipitated impurities are separated in a filter-press. The filtrate is transferred to a closed vat, provided with a mechanical agitator, a solution of zinc sulphate is then added in the proportion of 2.5 kilos. of the salt per kilo. of tannin present; and a current of ammonia gas (obtained by decomposing 2.5 kilos. of ammonium sulphate per kilo. of tannin) is passed in and the whole heated to boiling; the excess of gas is collected in another vat. Tannate of zinc is thus precipitated, and ammonium sulphate left in solution; the former is separated in a filter-press, washed successively with warm and cold ammoniacal water and with cold water, suspended in five times its volume of water, and decomposed with dilute sulphuric acid; a solution of barium sulphide is then added till no further precipitate is formed. The precipitate, which consists of zinc sulphide and barium sulphate, is removed by filtration, an almost colourless liquid is thus obtained, containing 20–30 per cent. of tannin ( $10^{\circ}$ – $15^{\circ}$  B.) and quite free from extractive matters. The ammonia is recovered from the filtrate from the zinc tannate; the mixed precipitate of zinc sulphide and barium sulphate is treated with dilute sulphuric acid, zinc sulphate passing into solution, the barium sulphate is then reconverted into the sulphide by calcination with coal.

The above process is quite as economical as the ordinary methods of manufacturing extracts.—S. B. A. A.

## PATENTS.

*Improvements in the Separation of the Isomeric Substances produced in the Manufacture of Dithiosalicilic Acid.*  
A. M. Clark, London. From Dr. F. von Heyden's Nachf., Radebeul, Germany. Eng. Pat. 13,027, August 17, 1889. 4d.

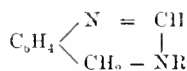
The dithiosalicilic acid produced according to Eng. Pat. 14,443 of 1888 (this Journal, 1890, 213), is a mixture of two isomerides which can be easily separated in the form of their salts. The less soluble dithiosalicylate No. 1. may be precipitated with common salt from the aqueous solution of an alkali salt of the crude acid. Another way is to treat the dry sodium salt of the crude acid with boiling alcohol, which dissolves only the sodium dithiosalicylate, No. II.

—E. G. C.

*Improvements in the Production of Quinazoline Derivatives.*

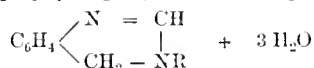
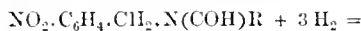
H. H. Lake, London. From Kalle and Co., Biebrich-on-the-Rhine, Germany. Eng. Pat. 13,343, August 23, 1889. 6d.

A PROCESS for the preparation of new pharmaceutical products belonging to the dihydroquinazolines and having the general formula,—

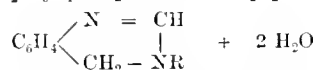
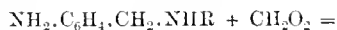


The two methods given for the preparation of these bodies are: (1.) Reducing formyl-*o*-nitro-benzylaniline, formyl-*o*-nitrobenzyl-*p*-toluidine, formyl-*o*-nitrobenzylanisidine, formyl-*o*-nitrobenzyl-*p*-phenetidine and similar products. (2.) Treating *o*-amidobenzylaniline, *o*-amidobenzyl-*p*-toluidine, *o*-amidobenzyl-*p*-anisidine, *o*-amidobenzyl-*p*-phenetidine and similar products with formic acid. The reactions which take place are expressed by the following equations—

I.

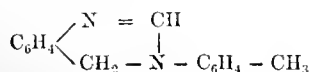


II.



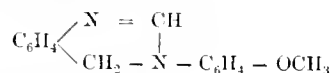
In working according to the first method 10 kilos. of *o*-nitrobenzylformanilide, melting point 77° (by heating 10 parts of *o*-nitrobenzylchloride with 8 parts of sodium formanilide in benzene) (see also Lellman and Stichel, Ber. 19, 1604) are reduced in 30 kilos. of alcohol with 14 kilos. of tin and 28 kilos. of hydrochloric acid. A salt having the formula,  $\text{C}_{14}\text{H}_{12}\text{N}_2\text{HClSnCl}_2$ , melting point 130°–134°, crystallises out, and when freed from tin yields the hydrochloride of phenylhydroquinazoline. This latter compound is also obtained by reducing *o*-nitrobenzylaniline with ammonium sulphide and boiling the *o*-amidobenzylaniline thus obtained with an excess of formic acid. The solution is then diluted with water, filtered, precipitated with soda, and the base obtained dissolved in dilute hydrochloric acid. On evaporation crystals of phenylhydroquinazoline hydrochloride separate out. It has the formula  $\text{C}_{11}\text{H}_{12}\text{N}_2\text{HCl} \cdot 2 \text{H}_2\text{O}$  and melts at 80°. When anhydrous it melts at 220°. The salt is easily soluble in water and alcohol, but can be precipitated from its aqueous solution by means of salt. The base melts at 95° and is almost insoluble in water, but very easily soluble in alcohol and ether. The properties of the following substances are also given:—

*p*-Tolyldihydroquinazoline—



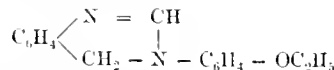
Its hydrochloride crystallises with two molecules of water and melts at 85°. The anhydrous salt melts at 221°. The base crystallises in leaflets melting at 120°.

*p*-Anisylidihydroquinazoline—



white needles, melting point 85°.

*p*-Phenetyldihydroquinazoline—



white leaflets, melting point 109°.

The phenylhydroquinazoline is known as orexine (see this Journal, 1890, 321).—T. A. L.

*A Process for the Production of Thio-oxydiphenylamine.*

M. Lange, Amersfoort, Holland. Eng. Pat. 15,706, October 7, 1889. 4d.

A SALT of *m*-oxydiphenylamine is heated in an aqueous solution with or without the addition of an alkali or alkaline carbonate and in an open vessel or in a digester. 250 kilos. of caustic soda are dissolved in water and 185 kilos. of "*m*-oxydiphenylamine salt" are added. The solution is heated and 200 kilos. of sulphur are gradually introduced and the mixture boiled until all the sulphur is dissolved. On adding sodium bicarbonate to the hot liquor the thio-product is precipitated as a light yellow powder. It is easily soluble in alkalis, less so in alkaline carbonates, and is precipitated from its solutions by acids and acid salts as a light yellow tasteless powder. It is also soluble in alcohol and acetic acid, but insoluble in water and benzene. The melting point is about 155°. The product is to be used as a substitute for iodoform as a dressing for wounds. It has no smell and is innocuous.—T. A. L.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

## PATENTS.

*Improvements in Indelible and Waterproof Prints, Pictures, Designs, and similar Objects, and in the Method of Producing the same.* J. R. France, New York, U.S.A. Eng. Pat. 20,964, December 31, 1889. 6d.

THE inventor places a print, photograph, or design on paper, linen, or other suitable material between thin transparent sheets of celluloid, uniting them by means of any suitable solvent for celluloid, or by a transparent size or gum.—E. J. B.

*Improvements in the Manufacture of Nitro-cellulose and Apparatus for that Purpose.* J. R. France, Plainfield, New Jersey, U.S.A. Eng. Pat. 5364, April 8, 1890. 6d.

CELLULOSE intended for conversion into nitro-cellulose is first reduced to a fine state of division by suitable machinery, which is fully described in the specification.—E. J. B.

## XXII.—EXPLOSIVES, MATCHES, Etc.

## PATENTS.

*Improvements in Fuses and Detonators.* P. Ward and E. M. Gregory, London. Eng. Pat. 11,500, July 18, 1889. 8d.

This invention is an improvement on Eng. Pat. 3981 of 1884, whereby the electric fuses and detonators are rendered absolutely impervious to the action of moisture, air, &c.

The detonating composition is introduced into a glass tube, in the closed end of which two wires have been previously sealed. A small plug of any suitable non-inflammable substance, such as asbestos, slag-wool, &c., is inserted on top of the explosive composition and the open end of the glass tube is fused together in the blow-pipe, or closed by a sufficient quantity of Chatterton's composition, sealing wax, or other suitable substance. Detailed descriptions and drawings are given of the methods and tools employed for preparing the glass tubes so that an absolutely uniform arrangement of the wires is ensured, both as to the surface exposed and their separation from each other.

—W. M.

*Improvements in the Preparation and Application of Explosive Compounds.* A. V. Newton, London. From A. Nobel, Paris, France. Eng. Pat. 12,307, August 2, 1889. 6d.

This specification describes a manner of forming propelling charges for firearms out of smokeless explosives of the celluloidal kind, the charges "consisting of rolled up sheets, or plates, or tubes, or hollow caps, corrugated or pierced with holes, for the purpose of increasing their surface of explosive combustion."—W. M.

*Improvements in the Manufacture of Lucifer Matches.* J. Lutz and L. Oltozy, Vienna, Austria. Eng. Pat. 12,763, August 13, 1889. 4d.

In this invention the matches are made by dipping the ends of the splints in bundles into a solution composed of 30 parts by weight of water, 20 parts of sodium chlorate, 4 parts of ammonium sulphate, and 2 parts of gum arabic, sugar, or other carbohydrate, and afterwards allowing them to dry. The matches thus made differ from those in ordinary use in having no enlarged heads. They ignite when rubbed on a surface covered with amorphous phosphorus and antimony trisulphide.—W. M.

*Improvements in Damp-Proof Cartridge Cases.* J. C. Butterfield and T. C. Batchelor, London. Eng. Pat. 14,025, September 5, 1889. 8d.

"This invention relates to the manufacture of cartridge cases, preferably of metal (this Journal, 1890, 765), which shall be impervious to damp or water, and consists in the employment of an inner pocket in which the fuse is placed when the cartridge is to be exploded."

In one form the pocket is cylindrical and concentric with the axis of the cartridge. In another it is simply a longitudinal groove in the outside of the cartridge case, the detonator being placed either inside the pocket or in the groove, and held in position by binding. In either case the detonator when exploded destroys the wall of the pocket or groove and explodes the cartridge. In this way a cartridge case is provided which does not require to be cut or opened in any way previous to firing.—W. M.

## XXIII.—ANALYTICAL CHEMISTRY.

*Determination of the Specific Gravity of Salts Soluble in Water.* J. W. Retgers. Zeit. f. Phys. Chem. 3, 289.

The published determination of the specific gravity of salts varies very much. The discrepancies are chiefly due to the difficulty of obtaining crystals free from cavities, these imperfect crystals being always lighter than the homogeneous ones. The author recommends that a liquid be prepared of such a density that only the heaviest, and consequently the homogeneous crystals float in it, whilst the lighter ones, that is those containing cavities, float on the surface. A mixture of methylene iodide and benzene is a convenient liquid. A few determinations made by this method are given.—A. L. S.

*A New Reaction of Hydrogen Peroxide.* G. Denigès. Bull. Soc. Chim. 1890, 797.

ONE or two cc. of a 10 per cent. aqueous solution of ammonium or sodium molybdate, containing an equal volume of concentrated sulphuric acid acquires on addition of a few drops of hydrogen peroxide a yellow colouration approaching in intensity that of solutions of the alkaline chromates and bichromates. The intensity of the colour is rapidly diminished on addition of water, and more gradually on diluting with sulphuric acid; it is slowly destroyed by nitric acid in the cold and speedily on warming. The reaction is not affected by boiling, and is probably due to the formation of a permolybdic acid; it is sufficiently sensitive to detect 0.1 mgrm. of hydrogen peroxide, whereas the chromic reaction does not indicate less than 0.5 mgrm.—S. B. A. A.

*Easy Method of Determining Nitrogen in Sodium Nitrate.* O. Foerster. Chem. Zeit. 14, 509—510.

Two to 3 grms. of the sample to be tested are weighed into a fair-sized porcelain crucible of known weight and dried at 150° or at such a temperature that the salt just commences to fuse. After cooling and weighing, the mass is dissolved in 25 cc. of hydrochloric acid of 19 per cent. strength and dried on the water-bath, the addition of acid and subsequent drying being thrice repeated to ensure the conversion of all nitrate into chloride. The crucible is next heated for a little time to 150°, then to a dull-red heat over the bare flame, and afterwards cooled and weighed, the result being calculated on the basis of 61.89 parts of NO<sub>3</sub> being replaced by 35.37 parts of Cl.

Satisfactory results are obtained with this method in the testing of Chili saltpetre, except when this substance has been sophisticated with magnesium chloride, in which case the method is unavailable.—E. B.

*The Separation of Copper from Arsenic by Electrolysis.* L. W. McCay. Chem. Zeit. 14, 509.

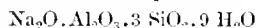
If the current from 4—6 Meidinger's cells be passed through a solution of an alkaline arsenate rendered alkaline with ammonia, no separation of arsenic or reduction of the arsenic acid takes place. If, however, such a current be passed through an ammoniacal solution of a copper salt, complete separation of the copper in time takes place. The experiment was tried with a mixture of known quantities of copper salt and potassium arsenate, to which ammonia in slight excess had been added, and it was found that 6 hours' passage of the current sufficed to remove the blue colour of the solution. The current, however, was continued for 18 hours longer, to ensure the complete reduction of the copper. The copper was then weighed and found to be quantitatively separated, the original amount also of arsenic acid being found in solution.

—E. B.

*The Analysis of Sodium Aluminate.* K. J. Bayer. Chem. Zeit. **14**, 736--737.

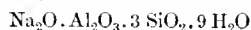
LUNGE has recently published (Zeits. f. angew. Chem. 1890, 227; this Journal, 1890, 766 and 767) a method of analysis of sodium aluminate similar in detail to the method previously described by the author (Zeits. Anal. Chem. **24**, 542; 1886, **25**, 180; this Journal, 1886, 41 and 550). Lunge, however, takes into consideration the silicic acid which may happen to be present, making, when this is the case, a deduction from the volume of standard acid required to change the tint of the methyl-orange indicator, *i.e.*, corresponding to the alumina present. This, the author states, is incorrect, since silicic acid has for acids no neutralising power whatever. For, if a solution of sodium silicate be titrated with standard acid, and either methyl orange or phenolphthalein, practically the same result is obtained; thus, in an actual experiment, 11.60 cc. of acid were required with methyl-orange as indicator against 11.45 cc. with phenolphthalein, the slight difference being due to the presence of alumina in the sample.

Lunge further states that the volumetric method in question is inapplicable to the determination of alumina in solutions containing much silica. To this objection the author rejoins that the method is intended for the analysis of sodium aluminate, and that sodium aluminate is no more likely to contain more than traces of silica than sodium silicate is of alumina. As a matter of fact, when solutions of sodium aluminate and silicate are mixed together, a voluminous precipitate is obtained, forming slowly in dilute solutions, but, in stronger ones, usually causing the whole mass to set to a jelly. When washed and air-dried this precipitate has the constant composition—



It forms an exceedingly fine, loose, probably crystalline powder, easily soluble in acids and sufficiently so in water to communicate an alkaline reaction thereto. Dissolved in a known amount of normal acid and titrated back with standard soda and methyl-orange, the volume of acid found to be neutralised corresponds to the alumina and soda contained in the compound. When, however, phenolphthalein is used as indicator a lower figure is obtained than would correspond to the soda alone which is present, the original compound, or a similar one, thus appearing to be partially regenerated. It was found, for example, that 0.2140 grm. of the crystalline compound neutralised 3.45 cc. of normal acid (1,000 cc. = 29.68 grms. of  $\text{Na}_2\text{O}$  or 16.37 grms. of  $\text{Al}_2\text{O}_3$ ) with methyl-orange as indicator, and with phenolphthalein 0.4458 grm. of substance neutralised 1.02 cc. of acid instead of 1.58 cc. actually corresponding to the soda in the compound. Now the amount of silica present in the solution of sodium aluminate tested by Lunge appears to be as high as can possibly be contained in the same, and yet from the above considerations it only causes in the determination an error of 0.014 cc. of standard acid, and may therefore be neglected. It is, moreover, worthy of note that the very slight error in the method, when silica is present in the solution to be tested, extends to the soda as well as the alumina, the percentage of the former being lowered in proportion as that of the latter is raised.

The only defect in the method, in the author's opinion, is the difficulty of obtaining a sharply-defined end-reaction, but this, after practice, can be obtained to within a couple of drops of acid. Of more practical consequence is the fact that each part of silica which remains behind, as almost all of it does, in the form of—



on lixiviating the roasted raw material, represents a loss of 0.37 part of  $\text{Al}_2\text{O}_3$  and 0.34 of  $\text{Na}_2\text{O}$ .—E. B.

*On the Estimation and Separation of Zinc in the Presence of Iron and Manganese.* J. Ribau. Compt. Rend. **110**, 1196--1199.

THE author bases his method on the following facts, which he has previously proved (Compt. Rend. **107**, 341; this Journal, 1889, 771):—(1.) Zinc in the presence of alkaline

thiosulphates or ammonia gives with hydrogen sulphide a dense granular precipitate of sulphide insoluble in cold thiosulphuric acid, which is here liberated. (2.) Iron is not precipitated in such a solution; the zinc precipitate carries down only a trace of iron.

The solution containing zinc, iron, and manganese is diluted until it contains not more than 1 grm. of zinc per litre. Sodium carbonate is added until a precipitate is just formed, and then is dissolved by a drop of dilute hydrochloric acid. Sulphuretted hydrogen is passed into the cold solution until the greater part of the zinc is precipitated together with some sulphur caused by reduction of the ferric chloride, a large excess of sodium thiosulphate is added, and the current of sulphuretted hydrogen continued until all the zinc is precipitated.

When it is desired to precipitate the iron with ammonia, after removing the zinc, it is preferable to substitute ammonium thiosulphate for the sodium salt, as the iron is liable to carry down some alkali with it, and the initial neutralisation should be made with ammonia or its carbonate.

The precipitate of zinc sulphide is allowed to stand five or six hours. It is then filtered off, washed with sulphuretted hydrogen water, dried in a current of hydrogen, and weighed as zinc sulphide. Several analytical numbers are given, showing that the amount of iron or manganese carried down with the zinc is so small as to be negligible.—A. L. S.

*On the Composition of Clays and Kaolins.* G. Vogt. Compt. Rend. **110**, 1199--1202.

THE author has made experiments which show that it is not possible by levigation to separate pure aluminium silicate from finely-divided quartz, felspar, or mica.

If clay, mica, quartz, or felspar be treated separately by sulphuric acid, the two first will be dissolved and the other will not. Thus a clay containing quartz, felspar, and mica may be separated into two parts by sulphuric acid. If either or both of these portions contain alkali, the origin of the alkali is determined.—A. L. S.

*Oxidising and Decolourising Properties of Charcoal.* P. Cazeneuve. Bull. Soc. Chim. 1890, 786--788.

See this Journal, 1890, 657.

*Test for Nitrobenzene.* J. Morpurgo. Pharm. Post, 1890, **23**, 258.

INTO a small porcelain basin are brought two drops of melted phenol, three drops of distilled water, and a small piece of potassium hydrate; the mixture is heated to the boil, and a few drops of a mixture of nitrobenzene and water added, when a crimson ring is produced round the edge of the mixture, the intensity of the colouration produced being proportional to the amount of nitrobenzene added; on the addition of a saturated solution of bleaching powder the colour of the ring changes to emerald green.

Use is made of the above reaction in the detection of nitrobenzene, which is effected by mixing with lime the solution of the substance to be tested, evaporating, if necessary, to dryness before extracting with ether, expelling the ether by heat, mixing the residue with a little distilled water, and then applying the test.—E. B.

*Researches on Diazo-compounds.* L. Gattermann. Ber. **23**, 1218--1228.

COPPER in a finely-divided state acts upon diazo-compounds very energetically even at 0° C., the product being chlorobenzene when diazo-benzene chloride is used, instead of diphenyl as was expected. As a consequence of this observation, it was discovered that the amido-group of aniline and of its homologues can be replaced by bromine and cyanogen, as well as by the nitro- and sulphocyanogen-groups. This reaction, which presents many points of

resemblance to the Sandmeyer's reaction with cuprous salts is in many respects superior to it. For example, it is carried out in the cold, and thus the heating of large volumes of solution is avoided; moreover the yields in consequence of the use of a low temperature are superior.

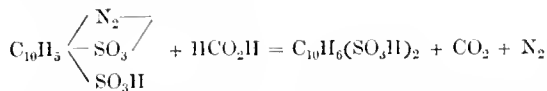
On acting on diazo-benzene sulphate in presence of alcohol, with copper, diphenyl was obtained on distillation with steam, as principal product. Zinc dust or iron powder yield the same result.

**Preparation of the Copper Powder.**—The copper powder was prepared by gradually dusting zinc powder through a very fine sieve into a solution of copper sulphate, which has to be continually stirred. It is, however, advisable not to continue until the blue solution of copper sulphate is decolourised, but to cease when it yet shows a slight blue tinge. Thus the presence of zinc in the copper is avoided. After decantation and repeated washing, the copper is treated with very dilute hydrochloric acid to remove traces of zinc, and finally washed until the wash-water shows a neutral reaction. It has to be kept moist in tightly closed vessels, being very susceptible to oxidation.

The following transformations were effected, and the percentage yields determined:—Chlorobenzene from Aniline; *o*-Chlorotoluene from *o*-toluidine; *p*-Chlorotoluene from *p*-toluidine;  $\beta$ -Chloronaphthalene from  $\beta$ -naphthylamine; *p*-Chloronitrobenzene from *p*-nitraniline; *m*-Chlorobenzaldehyde from *m*-Nitrobenzaldehyde; Bromobenzene from aniline; Iodobenzene from aniline; *p*-Iodotoluene from *p*-toluidine; Phenyleyanate from aniline; *o*-Tolyleyanate from *o*-toluidine; Diphenyl from aniline.—A. L.

**On the Application of Sandmeyer's Reaction to Diazo-sulphonic Acids, and on the Decomposition of these Compounds in presence of Copper or Copper Oxide.** G. Tobias. Ber. 23, 1628—1634.

THE author has observed that it is not necessary to use as large quantities of copper protochloride as Sandmeyer did for the decomposition of diazo-compounds. He further found that a piece of copper foil covered with a layer of protoxide would act in the same way as copper protochloride, and he also used the agent which has formed the subject of a communication by Gattermann (see preceding abstract), viz., copper powder. Evolution of nitrogen sets in in presence of dilute nitric, sulphuric, acetic, or formic acid and also of methyl and ethyl alcohol. On treating diazonaphthalene sulphonic acid with copper in presence of formic acid, naphthalene disulphonic acid was obtained according to the equation—



Diazonaphthalene sulphonic acid prepared from naphthionic acid was acted upon with copper in presence of alcohol and yielded naphthalene- $\alpha$ -sulphonic acid. Diazonaphthalene disulphonic acid yielded when treated under the same conditions, naphthalene disulphonic acid.—A. L.

**Estimation of Acetone in Methyl Alcohol and in Crude Wood Spirit.** L. Vignon. Bull. Soc. Chim. 1890, 595, —598.

See this Journal, 1890, 659 and 660.

**Estimation of Acetone in Crude Wood Spirit.** Vincent and Delachaux. Bull. Soc. Chim. 1890, 681—82.

THIS is a reply to Messinger's claim for priority for the process described by these authors (this Journal, 1889, 139).—S. B. A. A.

**Estimation of Hydrochloric Acid in Hydroxylamine Hydrochloride.** J. A. Muller. Bull. Soc. Chim. 1890, 605—606.

LÉGER (Jour. de Pharm. [5], 11, 425) found that the acids contained in the salts of most natural alkaloids may be determined volumetrically if phenolphthalein be used as the indicator, since the alkaloids do not redden that substance. The acid in hydroxylamine hydrochloride may likewise be titrated with sodium hydroxide free from carbonate, exactly like an aqueous solution of hydrochloric acid. This determination affords a means of rapidly ascertaining the quantity of soda required in the preparation of aldoximes (by V. Meyer's method) and of acetoximes.

The author finds that pyridine and the picolines and lutidines behave toward phenolphthalein like neutral compounds or sensibly so, and he proposes to utilise Léger's method for the estimation of acids in combination with these substances.—S. B. A. A.

**The Testing of Light Petroleum.** R. Kissling. Chem. Zeit. 14, 508—509.

THE author, having occasion to examine annually a large number of samples of light petroleum, has adopted the method of testing described below. The fractionating flask used is of the form and size recommended by Engler (Chem. Zeit. 10, 1238; this Journal, 1886, 654). 100 cc. of the petroleum sample are measured into the flask, which is connected with a Liebig's condenser 60 cm. long. The flask is supported by a thick piece of wire gauze, such as is used in the filter-presses of sugar refineries. The distillate is received in a cylinder graduated to 0.5 cc. It is advisable, at the commencement of distillation, to make the end of the adapter dip pretty deeply into the cylinder, in order to avoid loss by evaporation of the low-boiling fractions. The rate of distillation is such that 2—2.5 cc. are driven over per minute. When the heat is first applied, the mercury in the thermometer rises rapidly, then more slowly, and next becomes stationary for a while; the last position marks the "lower limit" of the boiling point of the sample. The distillation is now carried on, readings of the volume distilled being taken at intervals of 10°, beginning with the lowest multiple of 10 above the "lower limit." Finally, when the flask has become very nearly empty, the full flame of the Bunsen burner is directed to the bottom of it, and the temperature of the thermometer noted, this temperature being the "higher limit." It is not necessary to drive over the last few drops in the flask, but, should it be desirable to do so, the thermometer should be lowered to the level of the bottom of the neck of the flask. In the following table there are shown some results obtained by this method.

Kind of Petroleum.	Boiling Points Guaranteed by Vendor.	Volume (in cc.) Distilled.												“ Lower Limit.”	“ Higher Limit.”
		30°.	40°.	50°.	60°.	70°.	80°.	90°.	100°.	110°.	120°.	130°.			
Gasoline (“ Petroleum ether”).	30—100	0	37	66	81	95	97	97.5	98	..	..	..	31	121 (98.0 %)	
“ Light Benzine ” .....	60—100	..	..	..	0	53	89	96.5	97.5	98	98.5	..	62	123 (99.0 %)	
“ Middle Benzine ” .....	80—120	..	..	..	..	..	0	59	98	98.5	99.0	..	80	119 (99.0 %)	
“ Heavy Benzine ” .....	100—130	..	..	..	..	..	..	..	0	67	94	99	101	138 (99.5 %)	

*Gunning's Modification of Kjeldahl's Method of Estimating Nitrogen.* A. Atterberg. Chem. Zeit. 14, 509.

As certain nitrogenous compounds, such as alkaloids and azo-compounds, are with difficulty completely oxidised by boiling sulphuric acid, the author tried the modified method proposed by Gunning (this Journal, 1889, 823) on two such bodies, namely, quinine and indigo, comparing the results with those obtained by the ordinary Kjeldahl method and the soda-lime method; the results are tabulated below:—

	Nitrogen found in the Sample of	
	Quinine	Indigo.
	Per Cent.	Per Cent.
Original method of Kjeldahl ....	5.80	8.90
	5.99	8.92
Gunning's modified method.....	8.40	9.16
	8.40	9.50
Soda-lime method .....	8.29	9.38
	8.11	9.44

Gunning's method has the advantage of dispensing with the potassium permanganate, mercury, phosphoric acid, and sodium sulphide used in other modifications of Kjeldahl's method, and, besides, has the advantage of being quicker. The author strongly recommends it.—E. B.

*The Estimation of Sugar by a Solution of Copper Carbonate in Potassium Carbonate Solution.* H. Ost. Ber. 23, 1035—1039.

SOLDANI, in 1876, recommended a solution of copper carbonate in potassium carbonate solution in place of Fehling's solution for the estimation of sugar, but the solution did not work well, and does not appear to have had any considerable use. The author has modified the process and states that the solution he recommends gives reliable results with invert sugar, does not reduce cane sugar in the slightest, and possesses the advantage over Fehling's solution, that it is perfectly stable.

The copper solution is prepared by adding a solution of 23.5 grms. of pure crystalline copper sulphate to a solution containing 250 grms. of potassium carbonate and 100 grms. of potassium bicarbonate, and making the solution up to 1 litre.

In a volumetric estimation 50 cc. of the copper solution were decolourised by 25 cc. of a pure 0.4 per cent. solution of invert sugar after 9—10 minutes' boiling. Experiments were made to determine the effect of the presence of various quantities of cane sugar, and it was found that a solution as above, containing 100 parts of cane sugar, (1) to 100 parts or more of invert sugar, required 25 cc. of copper solution; (2) to 20 parts of invert sugar, 24.8 cc.; (3) to 5 parts of invert sugar, 24.5 cc.; (4) to two parts of invert sugar, 24.25 cc.; and (5) to one part of invert sugar, 23.75 cc. of copper solution. In these experiments if Fehling's solution had been used the results would have been much worse.

In a gravimetric determination it was found that when 50 cc. of the copper solution were boiled six minutes with 50 mgrms. invert sugar dissolved in 25 cc. water; 1 mgrm. of invert sugar reduced 3.40 mgrms. of copper, and that if less or more invert sugar were used then each mgrm. of invert sugar reduced less copper, if cane sugar were also present rather more copper was reduced for the same weight of invert sugar. For substances such as raw beet sugar containing a large quantity of cane sugar and only a little invert sugar, a solution containing less copper is used.

Tables are given showing the influence of various amounts of cane sugar in altering the amount of copper reduced by a unit of invert sugar.—A. L. S.

*Rosin Oils: A Method for Distinguishing Oils of the First and Second Distillation; their Alteration by the Action of Air.* A. Cheuvreux. Monit. Scient. 1890, 4, 685—689.

Rosin oils are composed of hydrocarbons belonging to the terpene group and the products of their oxidation, acids, aldehydes, acetones, &c. These last may be considered as impurities, although there are few commercial oils from which they are absent. The author has estimated the quantity of these acids present in different samples by a gravimetric and a volumetric method. By the soap the oil is saponified by caustic soda, the solution of first poured off, the resin liberated from this by hydrochloric acid, dried, and weighed. Its density is 1.02—1.04 and it is a similar body to colophony. By the volumetric method the oil is dissolved in alcohol and titrated with standard soda, using rosolic acid or phthalein as an indicator. Their molecular weight may be taken as 302; on this assumption 85—80 per cent. of resin acids were found in a resin extracted from the oil by the saponification method. The best oils contain the smallest quantity of acid, the rectified oils containing less than 4 per cent. and the commoner varieties from 4 to 10 per cent. of resin acids.

It is well-known that rosin oils thicken considerably on exposure to the air; the author finds that this is unaccompanied by any increase in the amount of resin acids.

—A. L. S.

*The Estimation of Cotton-Seed Oil in Lard.* L. de Koninek. Ned. Tijdschr. v. Pharm. 1890, 22.

A. VAN ASBÖTH found the iodine numbers of oleic acid from lard oil and from cotton-seed oil to be respectively 92 and 134. The author has obtained similar numbers (93 and 135) and proposes to determine the percentage of cotton-seed oil in lard from a consideration of the iodine number of the oleic acid extracted from the mixture. If, for example, this iodine number be 120, the ratio of the amounts of the two acids will be—

$$(134 - 92) : (120 - 92) = 42 : 28,$$

therefore 60 per cent. of the acid is derived from the seed oil. If the total acids form 65 per cent. of the sample, the

acid from the seed oil will form 39 per cent.  $\frac{(65 \times 60)}{100}$

of the sample, this corresponds to 55.5 per cent. of cotton-seed oil, since the latter only contains 70 per cent. of oleic acid. (See this Journal, 1889, 30, 63, 123, 480, 641, 658, and 741.)

The process is conducted as follows: About 3 grms. are saponified with freshly prepared alcoholic potash, the air being excluded as far as possible, a little phenolphthalein is added, the solution neutralised with acetic acid, and stirred into a boiling solution of 3 grms. of lead acetate in 200 cc. of water. The precipitate is washed with boiling water, extracted with ether, the extract after shaking with dilute hydrochloric acid and repeated washing with water, is made up to 200 cc., 50 cc. are then taken out, the ether almost entirely evaporated, 50 cc. of pure alcohol added and the solution titrated with decinormal sodium carbonate, using phenolphthalein as indicator. 1 cc. of soda solution = 0.0282 gm. of oleic acid.

A quantity of the ethereal extract containing exactly 0.5 gm. of oleic acid is withdrawn, the ether removed by a stream of carbon dioxide, the residue dissolved in 50 cc. of Hübl's reagent (25 grms. iodine and 30 grms. of mercuric chloride in 1,000 cc. of absolute alcohol), allowed to stand in the dark for 12 hours, 15 cc. of chloroform added and the mixture titrated with decinormal sodium thiosulphate. A blank experiment should be made with Hübl's reagent and chloroform. This method is inapplicable when the quantity of cotton-seed oil is less than 15 per cent. (See also this Journal, 1890, 418.)—S. B. A. A.



## New Books.

**PRINCIPLES OF GENERAL ORGANIC CHEMISTRY.** By PROF. E. HJELT. Translated from the Author's German edition of the original work by J. Bishop Tingle, Ph.D., F.C.S., Assistant in the Laboratory of the Heriot-Watt College, Edinburgh. London: Longmans, Green, and Co. New York: 15, East 16th Street. 1890.

SMALL 8vo. volume, bound in cloth, price 6s. 6d. It contains Translator's and Author's Prefaces, Table of Contents, 213 pages of subject-matter, and an alphabetical index. The text is divided into three parts, Part I. dealing with "The Composition, Constitution, and Classification of Organic Compounds"; Part II. with "The Physical Properties of Organic Compounds"; and Part III. with "The General Behaviour of Organic Compounds."

It is stated by the translator that "No similar work exists in English." In Part III., the reactions described are in all cases arranged according to the results; dehydration processes, for example, being all classed together. The work may be briefly defined as a condensed account of the present condition of the theory of Organic Chemistry, more especially designed "as a supplement to, rather than a substitute for ordinary text-books."

**THE ORGANIC ANALYSIS OF POTABLE WATERS.** By J. A. BLAIR, M.B., D.Sc., &c. London: J. and A. Churchill, 11, New Burlington Street. 1890.

THIS little work (8vo., bound in cloth) has for its object the brief practical description of the leading processes for the analysis of potable waters, together with a collection of results obtained by the author in the use of them. The various analytical processes are considered in the following order:—

I. The Albuminoid Ammonia and Oxygen Processes, and then some considerations in reference to the analyses of waters by both methods.

II. The Oxygen Process at 100° C.

III. The Estimation of the Organic Nitrogen by the Sulphuric Acid Process.

IV. The estimation of the Organic Carbon by the Sulphuric Acid and Permanganate Process.

The little work is thus divided into four parts, and in an Appendix sub-divided into groups A, B, C, D and E, is described the preparation of the various solutions required in carrying out the analytical methods described. Thus A.—Solutions for Albuminoid Ammonia. Process B.—Solutions for Oxygen Process at ordinary temperatures. C.—Solutions for Oxygen process at 100° C. D.—Estimation of Nitrates by Arsenious Acid. E.—Solutions for the Sulphuric Acid and Permanganate Process for Organic Carbon.

The text covers 118 pages and the price of the book is 3s. 6d.

**CHEMISCH-TECHNISCHES REPERTORIUM. ÜBERSICHTLICH GEORDNETE MITTHEILUNGEN DER NEUESTEN ERFINDUNGEN, FORTSCHRITTE UND VERBESSERUNGEN AUF DEM GEBIETE DES TECHNISCHEN UND INDUSTRIELLEN CHEMIE MIT HINWEIS AUF MASCHINEN, APPARATE UND LITERATUR.** Herausgegeben von DR. EMIL JACOBSEN. 1889. *Erstes Halbjahr. Zweite Hälfte.* Berlin: R. Gaertner's Verlagsbuchhandlung. Hermann Heyfelder, S.W. Schönebergerstrasse 26. London: H. Grevel and Co., 33, King Street, Covent Garden.

OCTAVO volume in paper cover; the second issue for the first half year of 1889. It contains 171 pages of subject matter, copiously illustrated with wood engravings. In the text the following subjects are treated of:—Foodstuffs and Confections; Paper; Photography; By-products of various kinds; Residual Matters; Manures; Disinfection and Sanitation; Soap; Explosives, &c. Preparation and Purification of Chemicals; Chemical Analysis; Apparatus; Machinery; Electro-technology; Technology of Heat; Appendix; Adulterations of Trade Products, &c. New Books; Alphabetical Index.

## Trade Report.

### TARIFF CHANGES AND CUSTOMS REGULATIONS.

#### RUSSIA.

##### *Regulations respecting Tare Allowance.*

Mr. Audley Gosling, Her Majesty's Chargé d'Affaires at St. Petersburg, in a despatch to the Foreign Office, dated the 23rd July, transmits translation of some new Custom house regulations respecting the allowance for tare on certain wrappers and packings in calculating the duties on imported goods. This translation reads as follows:—

All imported goods specified in the established table of allowance for tare and brought to Russia in wrappers, packing, &c., are generally cleared according to the weight that shall appear after deducting from their gross weight the percentage allowance of tare fixed by the table.

The net weight of these goods is, however, subject to verification (1) when the Customs officers shall have reason to doubt the applicability to certain goods of the proportion of tare allowed under the Table; (2) when the excessive weight of the tare shall be evident before the presentation by an importer to the Customs of the invoice of his goods; but the Customs may refuse to verify the tare if the board of a Custom house shall determine that its verification in any given case would be attended with special difficulties, danger, or injury to the health of the examining Customs officer.

No allowance for tare is made for internal wrappers, &c. which are not destined for preserving goods against injury when in transit, and which are made over to a purchaser together with the goods. Exception to this rule is afforded by the packing cases, &c. indicated under the Customs tariff as being liable to duty according to the materials of which they are made.

Paper wrappers in which woven and other stuffs are enveloped, boards and pasteboard sheets and rounds on which are wound tissues, ribbons, lace, and other goods, are not taken into account at the computation of duty, for which purpose the weight is deducted from the gross weight of the goods. In order to ascertain the weight of such packages it is necessary to weigh only a few of them if the packing be of a uniform character.

In addition to the allowance of tare made on chemicals, drugs, and cosmetics imported in glass, earthenware, and metal vessels placed in cases filled with sawdust, a further tare allowance of 10 per cent. is made on such cases with sawdust.

On all goods imported in vessels, wrappers, earboard boxes, &c. enclosed in an additional outside case, and the duty on which is fixed under the tariff on the gross weight, the duty shall be determined on the gross weight of the goods with their interior packing alone, *i.e.*, exclusive of the additional outside case or packing.—*Board of Trade Journal.*

#### SPAIN.

##### *Exemption from Import Duty of Sulphate of Copper.*

According to a despatch, dated the 2nd July, which has been received at the Foreign Office from Mr. W. A. C. Barrington, Her Majesty's Chargé d'Affaires at Madrid, the Madrid *Official Gazette* publishes a law of the Spanish Government providing that sulphate of copper may be imported into Spain free of duty, irrespective of the use to which it may be destined.—*Ibid.*

#### SERVIA.

##### *New Explosives Law.*

The *Moniteur Belge* says that the Servian law which authorises the Government to manufacture *en régie* powder, dynamite, and all other explosive materials, came into force on the 17th (29th) May last.

The Department of Finance has just published a circular recalling the fact that these products will have to pay on entry into Servia, besides the Customs duties in force, the following monopoly taxes:—

a. Dynamite and any other explosive material intended for trade will pay 1 franc per kilogramme.

Dynamite intended for such industrial undertakings as have legally acquired a privilege for the importation, machines and instruments necessary for them, will only pay 60 centimes per kilogramme.

b. Cartridges for all kinds of guns will pay 1 franc per hundred.

c. Revolver cartridges will pay 50 centimes per hundred.

d. All kinds of fuses and fireworks will pay 50 centimes per kilogramme of their gross weight.

Caps and empty cartridges will pay no monopoly tax.—*Ibid.*

#### NEW SOUTH WALES.

##### Customs Tariff.

The following is a statement of the rates of duty levied under the Customs tariff of New South Wales on certain articles imported into that colony:—

Articles.	Rates of Duty.		
		£	s. d.
Candles.....	Lb.	0	0 1
Cement.....	Barrel	0	2 0
Essences, flavouring, containing not more than 25 per cent. proof spirit.....	Gallon	0	4 0
Essences, flavouring, containing more than 25 per cent. proof spirit.....	"	0	14 0
Glucose, liquid and syrup.....	Cwt.	0	3 4
Glucose, solid.....	"	0	5 0
Naphtha and gasoline.....	Gallon	0	0 6
Oils, except black cocoa-nut, sperm, and palm, in bottle:—			
Reputed quarts.....	Dozen	0	1 6
Reputed pints.....	"	6	0 9
Reputed half-pints and smaller sizes.....	"	0	0 6
Opium and any preparation thereof not imported for use as a known medicine.....	Lb.	1	0 6
Paints and colours ground in oil.....	Cwt.	0	3 0
Varnish and lithographic varnishes.....	Gallon	0	2 0
Blasting powder and dynamite.....	Lb.	0	0 1
Salt.....	Ton	1	0 0
Methylated spirits.....	Gallon	0	4 0
Perfumed spirits.....	Liq. Gall.	0	15 0
Sugar, refined.....	Cwt.	0	6 8

—*Ibid.*

#### EXTRACTS FROM DIPLOMATIC AND CONSULAR REPORTS.

##### SPANISH COLONIES.

##### Pumice-Stone from the Canaries.

An important mine of pumice-stone exists on the Teneriffe Peak, of which the working was only started in 1888. The stone is found in that part of the peak called the "Canadas," at about 2,000 ft. above sea-level, which has an area of some 6,000 hectares, out of the middle of which rises the highest part of the peak. The Russian Consul at St. Croix bought this property of the Spanish Government in con-

sideration of an annual payment for the pumice-stone working. The Russian Consul has associated himself with a Belgian, and they, under the firm styled "Aguilar and Valcke," commenced operations in 1888, but it was only last year exportation was really started. At the Paris Exhibition this stone obtained a silver medal, and, in view of the requirements of England, France, and America, it may develop into a trade of great importance before many years. So far the Lipari Islands have practically furnished the world's supply of this product, exporting about 100,000 tons per annum. The Teneriffe stone is of excellent quality, and its extraction being a much more simple matter than in the Lipari Islands, it follows that the price is much less. More capital will, however, be wanted to extend the working operations.—*Chemist and Druggist.*

##### INDUSTRIAL ENTERPRISE IN BISCAY.

Her Majesty's Consul at Bilbao states that it is very difficult to point out what articles of commerce would be most in demand for a profitable market in this district; but as capital is abundant, and the present tendency of local capitalists is to implant industrial centres in the country, it may be safely stated that judicious projects of any kind would meet with ready support and aid, if undertaken by responsible and intelligent persons.

The construction of broad and narrow gauge railways continues for the purpose of putting Bilbao in direct communication with all places from which it is expected traffic would result. Among those worth mentioning is an important narrow gauge line now in course of construction between Bilbao and the provinces of Asturias, for the purpose of transporting coal to Bilbao from the mining districts of Asturias and Palencia. The length of this railway would exceed 200 kiloms. (110 miles), and its cost is estimated at 640,000*l.* It is apprehended it would convey from 200,000 tons to 400,000 tons of coal for the annual requirements of Bilbao.

Other industries are also in course of development in the province, such as paper, chemical, and arms manufactures.

The works for the new exterior port, designed and under construction by the eminent and indefatigable engineer-in-chief, Señor Churrua, are proceeding satisfactorily; and it is confidently stated that within eight years the breakwater will be sufficiently advanced to enable vessels to take advantage of its shelter, although its completion will probably not be effected for ten years.—(*No. 733, Foreign Office Annual Series.*)

##### THE SUPPLY OF COPPER FROM CHILI.

A report just issued by the Foreign Office from Vice-Consul Beazley, Caldera, contains several useful statements respecting the copper-mining industry of Chili. That gentleman states that the disastrous termination of the copper syndicate naturally affected the district very considerably, but before the full effects of the breakdown were experienced the price of copper again rose, and the present outlook is very encouraging. The Vice-Consul states that as long as the price of copper keeps above 4*l.* mining and the production of bar pay very well. That statement is one which may be commended to the careful consideration of those who contend that the present prices will not tend to cause an augmented production. The Vice-Consul goes on to state that the province has for many years laboured under the disadvantage of a monopoly in mining matters. One firm has managed to "stifle all competition;" but latterly a successful rival has appeared upon the scene, and, having established a powerful agency at Copiapo, is paying a fair price for metals, thereby producing a favourable reaction in business. Another disadvantage is the poverty of the mine owners, whose want of capital prevents the proper opening up of the rich mineral deposits of the country. There is, however, now some prospect of European capital being more largely invested, in addition to which the proposed construction of the Trans-Andine railway from Tinnogasta to Caldera seems certain to open up the mineral districts very advantageously. The Vice-Consul says that although the exports of bar copper have

fallen off considerably, there is a stock of 1,500 tons on hand ready for shipment at any moment. That was the case, at all events, on March 29th, when the report in question was written. This fact, also, is worthy of being noticed by the "bulls" of copper prices here. Another fact calculated to encourage the production of copper is, that all machinery is now admitted into Chili free of duty. With regard to the disaster which occurred early this year at the Copiapo Mines, the Vice-Consul states that the shaft has been reopened, and the mine will soon be in a better state than ever. The smelting works of Edwards and Co. are also again in full operation, and it is added "the production of 10 months of copper bar is accumulated in the establishment." It is possible that this is the stock already mentioned, but the point is not clearly brought out in the report. Another large concern which had been suspended owing to financial difficulties had resumed work under very favourable auspices, and on a sound basis. The general inference from these various statements is that the production of copper in Chili is being more actively carried on than for some years past, and that we are pretty sure to have larger shipments of bars thence so long as the price is over 47*l.* in the London market.—*Ironmonger.*

#### TUNISIAN PHOSPHATES OF LIME.

The following is an extract from a report by the Belgian Consul at Tunis, dated the 29th June last:—

The Mines Department of the Regency of Tunis has recently discovered the existence of beds of phosphates of lime in the mountainous region to the west of Gafsa, extending over a surface of about 50 kilometres.

The works recently undertaken have conclusively established the existence of phosphates in the regions of Djebel Limra, Seldja, and Stah, to the west of Gafsa, in large deposits, the workable thickness of which varies from 8 to 12 metres, over an extent of 50 kilometres. Analyses have shown that these deposits contain about 60 per cent. of tribasic phosphate, increased by about 3 to 5 per cent. by the washings. The total quantity of phosphates in this region may therefore be estimated at about six million tons, or about five million tons of washed ore, having a strength of at least 62 per cent. of tribasic phosphate of lime.

Other less important deposits are found in the mountains of Djebel Jellabia, Schib, and Refsa, to the south of Gafsa. This region is devoid of water, while to the west, for Limra, Seldja, and Stah, the River Selja supplies the water requisite for the preparation of the ore.

According to information obtained at Tunis, phosphates are worth about 60 francs (2*l.* 8*s.*) per ton. The working of the deposits in question, to the west of Gafsa, would require the construction of a railway connecting them *via* Gafsa to the nearest sea-port, called Skira. If this railway were constructed, the working of the phosphates, after deducting the expenses of extraction, washing, grinding, carriage, &c. would give a net profit of about 10 francs (8*s.*) per ton. The average annual extraction possible would reach 100,000 tons.—*Board of Trade Journal.*

#### FRANCE.

##### *The coming Tariff Revision.*

The summary of the answers given by French chambers and syndicates of commerce to the question issued by order of the Government, asking them to state their views on the approaching expiration of the commercial treaties (the *Chemist and Druggist*, April 19), has recently been issued. It is remarkable, inasmuch as it shows that, however strong the current of protectionism may be in Parliament, it is tempered amongst the manufacturers and merchants by a lively sense of the advantages of moderation.

#### *Chemicals.*

As regards the chemical trade, Lyons, Paris, and Calais are unanimous in advocating free trade in raw materials required for the manufacture of chemicals. All chemicals, they maintain, used in the production of textiles should be imported at low rates, because high duties would be prejudicial to the industries in which such materials are used.

If existing rates are to be changed, they should be reduced rather than increased. If increased, the new charge can only be compensated by export drawbacks, which are not easily applied, and necessarily lead to fraud.

The duties on soda, salts, acids, and methyl-alcohol should be reduced, and free import be allowed in favour of agriculture to pyrites, phosphates, nitrate of potash, sulphate of ammonia, and chloride of potash.

All produce obtained from coal-tar by distillation should remain free from duty, viz., oils and essences, benzene, benzol, naphthalin, &c.; but chemicals obtained from the products of the distillation of coal-tar, such as nitrobenzene, nitro-toluene, aniline, &c. should be charged at the rate of 15 frs. per 100 kilos., or about 5 to 6 per cent. *ad valorem.*

All colouring matters derived from coal-tar should be allowed to enter at a reduction of duty of 50 per cent.

On certain articles of interest to British trade the demands of French makers are protective. Marseilles, Nancy, and Avesnes want an additional duty on caustic soda. Paris would be content with the duty of 8 frs. levied under the present general tariff. But the Syndical Chamber of Paris Dyers and Cleaners advocates a lower charge of 3 frs. and 5 frs. per 100 kilos. in the minimum and maximum tariffs respectively. Dieppe asks for a duty of 1 fr. 50 c. on nitrates of potash and soda; but Granville, Nantes, Orleans, Clermont Ferrand, and others would keep nitrate of soda on the free list, and Paris thinks that a new duty would have to be compensated by a drawback.

Sulphate of copper now pays a duty of 3 frs. per 100 kilos. It is in great demand for dressing vines. The Chamber of Le Mans would raise the tariff, and the Syndical Chamber of Chemical Industries of Paris wants an increase of 65 per cent., but the Syndical Chamber of Dyers and Cleaners claims entire freedom for the article. Calais dyers object to any imposition of duty on aniline.

Paris wishes to put oxalic acid into the list of free imports; Arras, Marseilles, and Nîmes that the duty of 10 frs. on stearic acid should be increased by 10 to 50 per cent. Vienne and Paris propose a duty of 16 frs. per 100 kilos. The dyers of Paris ask for the repeal of the present duty of 10 frs. on chromate of potash, and the suppression of the duty on glycerin. But the merchant brokers of Paris would only admit raw glycerin free, charging 12 frs. to 15 frs. per 100 kilos. on distilled and dynamite glycerin.—*Chemist and Druggist.*

#### GUATEMALA.

##### *The Drug Trade.*

This Central American Republic of Guatemala had a prosperous year in 1889. Its trade increased considerably, and the imports of drugs alone rose from 15,171*l.* in 1888 to 20,155*l.* The drugs mostly come from the United States. The prosperity of the country is ascribed to the advance in the price of coffee, the principal product. The result of the participation of Guatemala in the Paris Exhibition is that the importations from France have more than doubled.—*Ibid.*

#### JAPAN.

##### *Drugs in Yokohama.*

The importation of drugs and chemicals into Yokohama is an increasing trade, large quantities of chemicals being imported for manufacturing purposes, such as paper and match making, glassworks, and other industries.

The Japanese Government are themselves manufacturers of caustic and other sodas, and of sulphuric acid, which is shipped in considerable amounts to the China market, or sold to private Japanese dealers.

Medicines for pharmaceutical purposes are also made on a large scale at Japanese establishments generally reported to be furnished with Government loans. Some importers complain of the continual difficulties, almost prohibitory, raised in regard to the sale of foreign drugs and medicines, these being constantly rejected by the Government analysts,

as not being up to the standard of the Japanese Pharmacopœia; and it appears, from a recent Government notification to Japanese traders, that this condition will in future be strictly enforced. The standard is higher than that of England, and necessitates the preparations submitted to the test being chemically or absolutely pure and free from inherent impurity, such as bismuth without a trace of arsenic. Under the Japanese Pharmacopœia, iodide of potassium is required to be 97 per cent. pure; in the British it is 95 per cent. This staple article, when pure, turns a yellow colour; but it is then apt to be rejected by the trader. The whole question, being of great importance to Japanese consumers and foreign importers, is entitled to earnest attention.

The imports of medicinal drugs and chemicals into Yokohama in 1889 were valued at 106,559*l.*, against 129,914*l.* in 1888.—*Ibid.*

### MISCELLANEOUS TRADE NOTICES.

#### BANANA PAPER.

The *South American Journal* for the 12th July has the following note:—

A new factor is entering the papermakers' market. It has been proved that the banana plant contains a greater quantity of pure fibre than any of the other numerous vegetable products used for the manufacture of paper. During the first 12 months of its vegetation the plant only produces a single bunch of fruit, after which it dies; but from its roots four to 10 young plants spring up. The adaptation of the plant to commercial purposes will, it is anticipated, revolutionise the paper material market, and largely affect the industry. The plant is easy of cultivation, and has long since been recognised by the wandering tribes in Central America as yielding many useful adjuncts to every-day domestic requirements.

#### THE PROGRESS OF WYOMING.

The following is an extract from *Bradstreet's* for the 5th July:—

In mineral wealth Wyoming undoubtedly excels. Coal mining has so far received the most attention. Coal is found in every county, varying from 2 to 40 ft. in thickness. The total production in 1888 was 1,512,936 short tons, worth at the mines 4,538,808 *dols.* One-third of this amount was expended for labour. The number of miners engaged was 1,527, who received at the rate of 90 cents to 1·10 *dols.* a ton for mining. The Union Pacific railroad has done the greater part of this work, monopolising it previous to 1886, but since then it has given outsiders a chance, and they have opened and developed a number of mines.

The soda deposits of the territory are of special interest, for soda has not hitherto been a natural product of the United States. The salt cake of commerce is produced in the eastern States at a few places where salt is cheap and sulphuric acid can be made as a by-product; but the larger amount is imported from England, where it is manufactured from common salt by treating with sulphuric acid and roasting. In Wyoming all that has to be done is to dig it out and dry it; and the territorial geologist says the deposits will amount to 52,000,000 tons. Last year we imported about 150,000 tons of carbonate of soda (including sal soda and soda ash), valued at 2,900,827 *dols.*, and in addition bicarbonate of soda worth 19,912 *dols.*, and caustic soda to the value of 1,300,851 *dols.*

It is further said that Wyoming has iron ores of higher grade and purity than those found in Alabama, abundance of limestone to flux them, and, at easy distances, producing oil wells ready to supply fuel for steel-making or other manufactures. Tin also gives great promise.

#### CHEMICALS IN JAPAN.

Drugs and chemicals are gradually forming a more important feature in the Japanese import trade. Large quantities of chemicals are being imported for manufacturing purposes, such as paper and match making, glass works, and other industries. They come chiefly from Great Britain, but a considerable portion is of German origin. The Japanese Government are themselves manufacturers of caustic and other sodas, and of sulphuric acid. Medicines for pharmaceutical purposes are also made in Japan on a large scale. Some importers complain of the great difficulties raised in regard to the sale of foreign drugs and medicines, these being constantly rejected by the Government analysts as not being up to the standard of the Japanese pharmacopœia, which is higher than the British.—*Industries.*

#### RUSSIAN COAL PRODUCTION.

The *Russie Commerciale* has recently published some particulars respecting the working of the Russian coal-fields. According to this publication the quantity of coal produced in the different countries is distributed as follows:—England, 166,000,000 tons; United States, 118,000,000 tons; Germany, 76,000,000 tons; France, 21,000,000 tons; Austria-Hungary, 22,000,000 tons; Belgium, 18,000,000 tons; Russia, 5,500,000 tons; and other countries, 5,000,000 tons. According to this, Russia only occupies a very secondary place among coal-producing countries, and yet Russia is the richest country in coal-fields. Rich coal-fields are found in the Governments of Moscow, Riazan, Toula, Poland, Caucasus, and particularly in the basin of the Donetz. Coal-fields which have up to the present remained unworked are found in the Island of Sakhaline, Western Siberia, in the neighbourhood of the Oural, in the Kirghish Steppes, and the plains of Turkestan; but it is particularly the basin of the Donetz which is remarkable for its coal wealth. According to the estimates of Professor Mendeleiev, the surface of the ground occupied by the coal-beds in this province extends over nearly 50,000 square kilometres, or 11,580 square miles. Taking the average figure of 50,000,000 poods (a pood is equivalent to 36 lb. avoirdupois) to the square kilometre, it will be found that the general yield of coal will amount to the total of 1,500,000,000,000 poods, or 21,000,000,000 tons. The entire world, says Professor Mendeleiev, might be supplied during the next 50 years with coal from the Donetz basin. The above remarks apply only to the superficial beds; as regards the others, which are at considerable depth, and which have not yet been worked, they are said to be even of greater richness. The working of coal lying at great depths can only up to the present be considered as an experiment, and the same may be said of the beds situated in the Kirghish Steppes, in the Governments of Tomsk, Caucasus, Turkestan, and the Island of Sakhaline, where the working is in a very elementary state. Coal mining has received a very considerable impetus in Poland, and in the basin of the Donetz. In 1888, Poland yielded 142,000,000 of poods, and the basin of the Donetz, 140,000,000 (2,290,000 tons and 2,258,000 tons respectively). Although the working of coal is rapidly on the increase, the quantity extracted is not sufficient for the requirements of the country, which is obliged to have recourse to foreign importation. In spite of its immense coal-fields, Russia imports annually nearly 107,000,000 poods of coal (1,726,000 tons), of which 85,000,000 poods are supplied by England, 19,000,000 by Germany, and more than 2,000,000 poods by Austria-Hungary. Russia, in addition, consumes enormous quantities of wood for her various industries, and this leads in a great measure to the destruction of her forests, with all its disastrous consequences. In spite of its good qualities, Russian coal cannot compete with foreign coal, even in the southern ports, which are found in proximity to the coal-fields of Donetz. Notwithstanding the increase in the duties on importation of foreign coal at Odessa, Sebastopol, and in the other ports of the Black Sea, English coal is no dearer than Donetz coal, and far from diminishing, its importation has greatly increased.

of recent years. "This proves," says the *Russie Commerciale*, "that the demands of the various industries for coal increase more rapidly than the working of this combustible in Russia, and that the condition of the means of transport is far from satisfactory."—*Journ. Soc. Arts.*

#### THE RUSSIAN NAPHTHA INDUSTRY.

According to a report of the Russian Finance Minister, both the production and export of Baku oil products continue to grow. In the five first months of the present year Russia exported 22,720,000 poods of naphtha, or 7,560,000 poods more than in the corresponding period of 1889. It should, however, be remarked that of this so-called naphtha 17,320,000 poods weight was refined kerosene. Russia itself took 53,150,000 poods of naphtha, 5,200,000 poods of this being refined kerosene and 40,850,000 poods residuals. Upon the completion of the Wladikawkas-Baku railway a further important development of the naphtha trade is expected. Valuable naphtha springs have lately been discovered on Sakhalin Island, and a company has been formed to work them. A representative of the concern is at present in Baku studying the processes of boring for and shipping oil.—*Ironmonger.*

#### GAMBIER AT SINGAPORE.

Referring to an extract from the annual report of the Singapore Botanical Gardens which appeared in the June number of this Journal, pp. 670—671, we are requested to state that, though the attempt to propagate the plant by seed has failed, yet the plants themselves have been transported to this country by means of Wardian cases, and may be seen in full vigour at Kew Gardens, where they only await purchasers to be transferred to any colony with a suitable climate.

#### EXPORTS OF CHEMICALS FROM THE TYNE.

The following chemicals were exported from the Tyne during the month just ended and corresponding month of 1889:—

	1890.	1889.
	Tons.	Tons.
Alkali and soda ash .....	1,267	1,111
Bicarbonate of soda .....	..	14
Bleaching powder .....	2,808	1,736
Artificial manure .....	3,931	2,854
Soda crystals .....	982	1,017
Sulphate of soda .....	42	10
Caustic soda .....	910	..
Other chemicals .....	1,115	1,752
Total .....	11,055	8,554

Making an increase of 29·23 per cent. for the month. The heavy increase in the quantity of bleaching powder exported is owing to makers being unrestricted in their output this year, whereas their make during the previous year was curtailed by the Bleach Association. Bicarbonate of soda is not now manufactured on Tyneside, the production having ceased some months ago.—*Chemist and Druggist.*

#### SULPHUR EXPORTS FROM ITALY.

The official returns show that the gross exports of raw and refined sulphur from Italy, up to the end of May last, amounted to 1,700,106 quintals, in contrast with 1,503,394 quintals in the same five months last year, this being an increase of 196,712 quintals. This export is valued at 11,900,742 lire in 1890, and 10,523,758 lire in 1889, thus showing an advance on the gross value of 1,375,984 lire. The official valuation puts sulphur at 7 lire per quintal. The destination of the exports is given, as presented in the

annexed table, for the first five months of the last three years:—

Exported to	1888.	1889.	1890.	Difference 1890 on 1889.
	Quintals.	Quintals.	Quintals.	
Austria-Hungary .....	64,816	61,191	90,181	+ 28,990
Belgium .....	21,468	45,185	33,991	— 11,194
France .....	397,360	333,203	494,876	+ 71,673
Germany .....	72,772	61,925	60,115	— 1,810
Great Britain .....	193,587	169,787	137,174	— 32,163
Greece .....	64,345	102,935	137,679	+ 34,744
Holland .....	7,223	8,824	6,607	— 2,217
Portugal .....	85,130	106,252	173,121	+ 66,869
Russia .....	18,638	87,157	53,666	— 33,491
Spain .....	50,220	49,166	51,510	+ 2,344
Sweden and Norway ..	4,803	19,097	29,284	+ 10,187
Turkey .....	10,290	25,246	57,269	+ 32,023
North America .....	545,068	410,267	497,293	+ 27,026
Central and South America .....	45,995	7,629	1,686	— 5,943
Other countries .....	10,131	15,230	25,651	+ 10,421
Total .....	1,501,746	1,503,394	1,700,106	+ 196,712

The position of the trade is so clearly shown in the foregoing that there is no need for comment.—*Chemical Trade Journal.*

#### THE GERMAN CEMENT INDUSTRY.

The manufacture of Portland cement has made rapid progress during the last 25 years. In 1865 the industry was in its infancy, and was carried on upon a small scale at Bonn, Stettin, Lünenburg, &c., the manufacturers working under licenses from a Mr. Ashlin, an Englishman, who owned the German patent for the so-called wet process. The manufacture of cement has grown so rapidly that at the present time the annual output is estimated at 10,000,000 barrels, worth 70,000,000 M. The chief seat of the industry is North Germany, in the vicinity of Berlin, Stettin, Hamburg, and Hanover. The export trade has increased from 137,000 kilocentners in 1879 to 1,341,300 kilocentners in 1889. The chief foreign market is the United States, to which destination some 405,000 kilocentners were exported in 1889. Roumania, during the same period, took 211,000; Australia, 171,000; Brazil, 126,000; Chili, 895,000; Japan, 88,500 kilocentners.—*Ironmonger.*

#### BOARD OF TRADE RETURNS.

##### QUICKSILVER.

	IMPORTS.		EXPORTS.	
	1890.	1889.	1890.	1889.
	Bottles.	Bottles.	Bottles.	Bottles.
July .....	1,183	2,613	3,048	2,479
Jan.—July .....	54,568	54,756	56,097	35,492
	1890.		1889.	
Average Price:	£ s. d.		£ s. d.	
July .....	10 2 6		9 2 6	

## SUMMARY OF IMPORTS.

	Month ended 31st July	
	1889.	1890.
	£	£
Metals.....	1,893,018	2,129,311
Chemicals and dyestuffs.....	643,376	601,100
Oils.....	793,559	629,464
Raw materials for non-textile industries.....	5,073,477	3,497,637
Total value of all imports ....	35,903,518	33,982,629

## SUMMARY OF EXPORTS.

	Month ended 31st July	
	1889.	1890.
	£	£
Metals (other than machinery) ....	3,303,409	4,117,237
Chemicals and medicines .....	612,575	731,141
Miscellaneous articles.....	2,751,161	3,143,229
Total value of all exports.....	22,124,506	24,321,336

## IMPORTS OF METALS FOR MONTH ENDED 31ST JULY.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Copper:—			£	£
Ore..... Tons	14,276	7,749	87,530	62,712
Regulus .....	11,886	11,311	312,492	347,776
Unwrought ....	1,787	4,686	81,051	270,204
Iron and steel:—				
Iron ore .....	322,531	449,571	235,240	352,694
Iron bolt, bar, &c. ..	19,254	12,525	160,381	124,687
Steel, unwrought..	2,176	634	16,420	7,052
Lead, pig and sheet ..	13,283	13,095	169,624	170,312
Pyrites .....	50,810	54,929	99,832	104,785
Quicksilver .....	195,950	88,736	20,096	14,391
Tin .....	54,795	37,356	246,931	175,855
Zinc .....	5,579	4,867	95,559	109,666
Other articles ... Value £	..	..	367,592	383,778
Total value of metals	..	..	1,833,048	2,129,311

## IMPORTS OF CHEMICALS AND DYE STUFFS FOR MONTH ENDED 31ST JULY.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Alkali..... Cwt.	3,595	3,156	£ 2,946	£ 2,369
Bark (for tanners, &c.) .....	50,266	82,515	43,742	48,279
Brimstone .....	76,946	24,393	15,882	5,286
Chemicals..... Value £	..	..	132,349	126,953
Cochineal .....	639	163	4,593	922
Cutch and gambier Tons	2,265	2,296	64,221	68,201
Dyes:—				
Aniline .....	..	..	23,127	18,656
Alizarine .....	..	..	26,630	27,198
Other .....	..	..	1,134	1,887
Indigo .....	2,871	1,039	43,812	15,471
Madder .....	1,225	1,114	1,009	1,403
Nitrate of soda....	198,800	88,188	88,011	36,972
Nitrate of potash .	24,267	25,278	21,653	22,285
Valonia .....	2,639	3,055	39,476	62,323
Other articles... Value £	..	..	134,581	167,890
Total value of chemicals	..	..	643,376	601,100

## IMPORTS OF OILS FOR MONTH ENDED 31ST JULY.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Cocoa-nut..... Cwt.	25,398	1,065	£ 31,404	£ 1,534
Olive .....	3,323	1,983	116,388	77,335
Palm .....	163,759	90,123	161,205	101,909
Petroleum .....	10,549,235	10,625,318	250,293	209,089
Seed .....	1,151	1,232	28,481	32,484
Train, &c..... Tons	2,364	1,857	45,801	46,461
Turpentine .....	95,958	61,224	143,324	94,390
Other articles ... Value £	..	..	73,603	66,562
Total value of oils ...	..	..	743,559	629,454

## IMPORTS OF RAW MATERIALS FOR NON-TEXTILE INDUSTRIES FOR MONTH ENDED 31ST JULY.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Bark, Peruvian ..	8,009	6,554	£ 27,783	£ 17,778
Bristles.....	393,394	270,015	63,002	46,587
Caoutchouc.....	14,171	16,368	131,883	159,219
Gum:—				
Arabic.....	7,013	3,116	22,488	9,912
Lac, &c.....	4,319	7,649	13,417	32,858
Gutta-percha ....	3,773	1,951	47,531	43,657

IMPORTS OF RAW MATERIALS, &c.—*cont.*

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Hides, raw:—			£	£
Dry..... Cwt.	52,981	33,604	153,753	94,820
Wet..... "	75,871	36,381	175,486	81,167
Ivory..... "	1,198	1,272	55,734	60,021
Manures:—				
Guano..... Tons	2,587	..	15,781	..
Bones..... "	2,544	1,760	12,281	9,140
Paraffin..... Cwt.	18,789	33,556	21,911	1,426
Linen rags..... Tons	3,624	3,328	38,401	30,062
Esparto, &c..... "	14,366	17,081	70,297	77,767
Pulp of wood .... "	9,457	14,237	56,559	76,548
Rosin..... Cwt.	115,027	119,957	25,853	37,977
Tallow and stearin .. "	145,893	174,867	186,232	223,276
Tar..... Barrels	15,477	28,615	9,266	18,965
Wood:—				
Hewn..... Loads	274,052	262,912	688,333	585,900
Sawn..... "	921,956	834,605	2,211,998	1,914,144
Staves..... "	21,062	17,101	70,322	82,326
Mahogany..... Tons	2,804	2,967	24,752	25,063
Other articles.... Value £	..	..	886,277	810,777
Total value .....	..	..	5,009,843	4,492,390

Besides the above, drugs to the value of 38,068*l.* were imported, as against 54,262*l.* in 1889.

## EXPORTS OF METALS (OTHER THAN MACHINERY) FOR MONTH ENDED 31ST JULY.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Brass..... Cwt.	9,967	16,774	42,359	50,302
Copper:—				
Unwrought..... "	72,415	80,825	158,331	243,564
Wrought..... "	39,283	21,111	79,189	75,477
Mixed metal .... "	33,856	48,228	80,616	136,348
Hardware..... Value £	..	..	230,693	246,614
Implements..... "	..	..	109,640	116,625
Iron and steel.... Tons	352,695	391,724	2,343,596	2,414,396
Lead..... "	4,130	4,036	58,717	58,186
Plated wares... Value £	..	..	35,736	31,799
Telegraph wires... "	..	..	32,909	56,575
Tin..... Cwt.	11,768	9,325	55,112	45,780
Zinc..... "	17,373	15,957	12,860	11,839
Other articles .. Value £	..	..	77,456	95,964
Total value .....	..	..	3,303,409	4,117,287

## EXPORTS OF DRUGS AND CHEMICALS FOR MONTH ENDED 31ST JULY.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Alkali..... Cwt.	389,222	491,818	104,834	167,342
Bleaching materials .. "	126,848	145,442	46,218	40,055
Chemical manures. Tons	20,915	36,550	142,242	189,686
Medicines..... Value £	..	..	77,659	94,443
Other articles ... "	..	..	241,622	242,615
Total value .....	..	..	612,575	731,441

## EXPORTS OF MISCELLANEOUS ARTICLES FOR MONTH ENDED 31ST JULY.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Gunpowder..... Lb.	957,969	1,189,800	21,619	27,225
Military stores.. Value £	..	..	76,351	133,637
Candles..... Lb.	1,158,809	1,123,400	20,161	22,410
Caoutchouc..... Value £	..	..	100,946	102,327
Cement..... Tons	62,533	54,393	124,763	113,154
Products of coal Value £	..	..	75,254	91,640
Earthenware ... "	..	..	186,281	214,843
Stoneware..... "	..	..	19,284	16,901
Glass:—				
Plate..... Sq. Ft.	276,665	240,151	18,231	15,406
Flint..... Cwt.	10,588	9,496	21,344	22,191
Bottles..... "	87,941	86,878	43,125	41,331
Other kinds.... "	18,977	14,651	12,742	14,028
Leather:—				
Unwrought .... "	11,051	11,123	106,969	101,948
Wrought..... Value £	..	..	31,749	31,913
Seed oil..... Tons	4,881	4,716	108,091	113,364
Floor cloth..... Sq. Yds.	1,435,600	1,441,800	71,674	73,871
Painters' materials Val. £	..	..	139,633	147,007
Paper..... Cwt.	80,281	86,454	147,129	148,615
Rags..... Tons	4,106	5,299	32,946	40,256
Soap..... Cwt.	46,977	52,705	41,863	54,282
Total value .....	..	..	2,751,161	3,143,229



## Monthly Patent List.

\*The dates given are the dates of the Official Journals in which acceptances of the Complete Specifications are advertised. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the said dates.

### I.—GENERAL PLANT, APPARATUS, AND MACHINERY.

#### APPLICATIONS.

11,042. J. W. Gooch, C. A. Baker, and W. White. Improvements in electric thermometers and thermographs. July 15.

11,082. J. Whitehead. Improvements in and apparatus for supplying fuel to steam boiler and other furnaces. July 16.

11,246. J. Klein. Improved process and apparatus for cooling liquids by means of air. Complete Specification. July 18.

11,262. W. Robertson and D. J. Macdonald. Improved apparatus for compressing air or other fluid. July 19.

11,529. A. Chapman and S. Vickers, jun. Improvements in evaporating apparatus, specially adapted for the extraction of salt from brine. July 23.

11,686. S. M. Lillie. Improvements in evaporating apparatus. Filed July 25. Date applied for December 26, 1889, being date of application in United States. Complete Specification.

11,714. D. Rylands and R. Morant. Improvements in glass-lined tubing. July 26.

11,747. G. U. Haslam and P. Cornish, jun. An improved method of drying and desiccating wet substance and materials. Complete Specification. July 26.

11,807. W. P. Thompson.—From A. Raps, Germany. Improvements in or appertaining to mercurial pumps. July 28.

11,895. L. Johnstone. Improvements in and connected with rotative furnaces or kilns for treating and recovering waste products from raw or partially raw materials, used more especially, and for example, in the manufacture of cement. July 30.

12,004. L. Menetrel. An improved apparatus for washing, lixiviating, or extracting purposes. July 31.

12,079. C. D. Martin. Improvements in valves for chemical liquids. August 1.

12,260. A. W. Ellis. See Class XVII.

12,325. J. Jackson. Improvements in pressure and vacuum gauges. Complete Specification. August 6.

12,421. D. Bethmont. Improvements in apparatus for generating steam or vaporising liquids. August 8.

12,505. S. Seekendorf. An improved drying apparatus for granular, pasty, and similar material, and press for forming the dried material into cakes. Complete Specification. August 9.

12,535. E. Barlow and R. Cunliffe. Improvements in apparatus for drying, calcining, and evaporating substances or materials. August 11.

12,542. E. Guigon. Improvements relating to the circulation of water in surface condensers. August 11.

12,897. G. A. Hart. An improved construction of kiln for drying moist materials. August 16.

### COMPLETE SPECIFICATIONS ACCEPTED.\*

1889.

12,206. W. Tipping. Apparatus for destructing, drying, and calcining. July 23.

12,410. W. L. Wise.—From P. R. Bedlington. Apparatus for indicating temperature and pressure. August 13.

12,871. C. H. Roeckner, F. L. Roeckner, and R. L. Roeckner. Apparatus for concentrating and evaporating liquids, and burning off organic matter. July 23.

14,958. J. Hodgkinson. Mechanical stokers. July 30.

15,124. R. C. A. Banfield.—From C. Linde. Method and means for refrigerating and drying air. July 30.

15,432. R. Marsden and J. Pickard. Transferred to Class V.

16,099. P. Nezeraux. Centrifugal pumps, compressors, and blowers. August 20.

19,222. E. Theisen. Evaporating apparatus. July 23.

1890.

3279. W. H. Skinner. Heating water and other liquids, and fuel for use therefor. August 20.

6136. J. Klein. Apparatus for condensing, heating, or cooling gases or liquids. July 23.

8499. H. Schulze-Berge and F. Schulze-Berge. Air pumps. August 13.

9312. G. Horner. Surface condensers. July 23.

9326. J. O. Woods. Vaporisers. July 23.

9379. W. B. Bull. Means for purifying liquids for prevention of boiler incrustation, ageing liquors, &c. August 13.

9554. L. H. A. W. Sack. Apparatus for automatically drawing off viscous substances. July 30.

9941. R. Haddan.—From E. Passburg. Method and apparatus for drying aqueous matter. Aug. 6.

10,133. P. Abrahamson. Oven and other fire-chamber doors. August 20.

10,398. L. P. Cohen and E. Hermann. Furnaces. August 13.

10,524. T. Gaunt and E. H. Chapp. Evaporating apparatus. August 13.

### II.—FUEL, GAS, AND LIGHT.

#### APPLICATIONS.

11,079. J. Laing. Improvements in the apparatus for destructive distillation of mineral oils. July 16.

11,091. W. R. Hutton. Improvements in apparatus for making coke from coal, and obtaining tar and ammoniacal water and mixed gases therefrom. July 16.

11,106. J. E. Bott, W. Mortimore, and F. H. Lindley. Improvements in gas producers. July 16.

11,195. W. W. Staveley. Improvement in the treatment of crude blast-furnace or coke-oven oils, and application of the oils so treated or produced. July 18.

11,239. W. H. Buckland and G. Myers. Improvements in the composition and manufacture of fuel from coal dust and similar material. July 18.

11,424. G. F. Priestly. Automatic charging and discharging gas retorts. July 22.

11,452. J. Hall. Improvements in the manufacture of coke or in the coking of coal. July 22.

11,514. W. Laezy. Improvements in the manufacture of artificial fuel. July 23.

11,581. F. J. Rowan and H. Armour. Improvements in and connected with retorts for distilling shale or other oil-yielding minerals. July 24.

11,740. S. H. Smith. Improvements in gas-producer furnaces. July 26.

11,757. J. Laing. Improvements in the apparatus for destructive distillation of mineral oils. July 28.

11,784. J. W. C. C. Schirm. Improvements in the method for producing intense light by magnesium or by other glowing materials. Complete Specification. July 28.

12,016. W. Foulis. Improvements in the stirring and drawing of the charge in gas retorts, and in the apparatus employed therefor. July 31.

12,036. I. Lilley and G. H. Hopkins. Improvements in the manufacture of "patent" or compressed fuel, and in machinery or apparatus therefor. Complete Specification. August 1.

12,134. F. V. Hadow. An improvement in treatment of coal for the production of artificial fuel. August 2.

12,217. E. Andrews and C. Miller. Economising, saving, and increasing the heat of fuel, such as coal, coke, wood, turf, and other fuel for heating and drying purposes. August 5.

12,282. C. C. Sykes and G. Blamires. Improvements in apparatus for removing watery vapour from illuminating gas. August 6.

12,580. C. W. Bartholomew and J. Oxley. New or improved apparatus for drawing coke from coke ovens. August 12.

12,638. G. Frank. Improvements in compressed fuel. August 12.

12,639. R. Schimper. Improvements in the manufacture of artificial fuel. August 12.

12,860. J. T. Key. Improvements in the process of and apparatus for manufacturing gas and for the utilisation of the same. Complete Specification. August 16.

12,895. A. M. Hay. Improvements in the process and apparatus for producing oil water-gas. August 16.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

11,624. F. J. Jones. Manufacture of illuminating gas. August 20.

12,266. B. H. Thwaite. Method and apparatus for producing water-gas. August 13.

13,016. J. Dewar and B. Redwood. Distillation of mineral oils, &c., and apparatus therefor. August 20.

14,990. J. R. Wigham. Improving the illuminating power of lighthouses and other places. July 30.

15,409. J. Somerville and S. Cutter. Apparatus for charging and drawing gas retorts. August 6.

15,797. J. R. Paisley. Apparatus for manufacture of water-gas. August 13.

1890.

9733. S. Pitt.—From E. J. Jerzmanowski. Manufacture of illuminating gas. August 6.

#### III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

##### APPLICATIONS.

10,976. T. H. Davis and A. H. Davis. See Class XIII.

11,079. J. Laing. See Class II.

11,757. J. Laing. See Class II.

12,415. E. Boernstein and S. Kleeman. Improvements in the treatment of commercial xylidine for effecting the separation of its various isomeric xylidines. August 8.

#### IV.—COLOURING MATTERS AND DYES.

##### APPLICATIONS.

11,530. B. Willeox.—From The Farbenfabriken vormals F. Bayer and Co., Germany. Improvements in and connected with the manufacture of artificial indigo. July 23.

11,548. J. Imray.—From La Société Anonyme des Matières Colorantes et Produits Chimiques de St. Denis, France. Manufacture of azo-colouring matters derived from azoxyamines. July 23.

11,802. L. Scholvién and L. F. Riedel. Improvements in the manufacture of phenyldimethylpyrazoline. July 28.

12,356. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik, Germany. Improvements in the manufacture and production of colouring matters from dihydroxybenzoic acid. August 7.

12,369. Brooke, Simpson, and Spiller, Limited, and W. S. Simpson. Improvements in the manufacture of blue-black and other azo-colouring matters. August 7.

12,386. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik, Germany. Improvements in and relating to the production and manufacture of new materials for the preparation of dyestuffs. August 7.

12,550. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik, Germany. Improvements in the manufacture and production of new diphenyl derivatives and a new base for use in the preparation of dyestuffs. August 11.

12,715. B. Willeox.—From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture and production of new derivatives of alizarine and its analogues. August 13.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

12,784. O. Imray.—From the Société Anonyme des Matières Colorantes, &c. Colouring matters derived from paratoluidine,  $\alpha$ -metaxylidine, pseudo-eumidine, and mesidine, with tetramethyldiamidobenzhydrol. August 20.

13,217. H. H. Lake.—From A. Leonardt and Co. Colouring matters. August 20.

13,665. B. Willeox.—From The Farbenfabriken vormals F. Bayer and Co. Production of colouring matters. August 6.

14,207. The Clayton Aniline Co., Limited, P. Brunner, and J. Hall. Production of colouring matters. August 6.

14,230. B. Willeox.—From The Farbenfabriken vormals F. Bayer and Co. Manufacture and application of colouring matters. August 6.

16,448. J. Dawson. Manufacture of a grey basic colouring matter soluble in water. August 13.

16,699. S. Pitt.—From L. Casella and Co. Preparation of amidonaphtholsulphonic acid, and of colouring matters therefrom. August 20.

1890.

8389. A. Remy, R. Kramer, and W. Herking. Manufacture of colouring matters. August 6.

#### V.—TEXTILES, COTTON, WOOL, SILK, Etc.

##### APPLICATIONS.

11,005. A. Gratz and B. Gratz. Improvement in the process of treating textile fabrics, and manufacturing bagging from jute butts. Complete Specification. July 15.

11,094. F. Lechner. Improvements in or relating to the manufacture of artificial silk and thread, and in apparatus therefor. July 16.

- 11,986. F. T. Kuott. *See Class XXI.*  
 12,576. J. Sutcliffe and J. Greenwood. An improvement in the treatment of yarn for weaving. August 12.  
 12,689. K. Trobach and G. J. Bruch. *See Class VI.*

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

12,626. G. W. Robertson, D. Black, and J. McGlashan. Treatment of vegetable fibrous material, and apparatus therefor. July 23.

12,975. A. Deru. Process and apparatus for automatically drying and purifying wool and other textiles. July 30.

15,432. R. Marsden and J. Pickard. Condensers for slivers of wool or other fibre. August 6.

15,135. J. E. Richardson. Manufacture of carpets, matting, twine, &c. from esparto grass. July 30.

16,258. J. Schofield. Production of yarn, and machinery therefor. August 20.

1890.

7390. J. Longmore and W. L. Watson. Decortication of rhea, jute, and other fibrous vegetable stems. July 23.

11,005. A. Gratz and B. Gratz. Improvement in the process of treating textile fabrics, and manufacturing bagging from jute butts. August 20.

## VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

## APPLICATIONS.

11,044. M. L. W. Martinot. Improvements in apparatus for washing or bleaching. Complete Specification. July 15.

11,100. A. H. Wardle. Improvements in machinery for dyeing hanks of silk and other yarn. July 16.

11,372. A. Mitscherlich. New process of sizing paper. July 21.

11,580. W. Marriott. Improved mordant for black dyeing. July 24.

11,765. A. Goldthorp. An improved method of dyeing wool, cotton, or any other fibrous substance, in its raw or manufactured state, such as tops, slubbing, yarn on cops or bobbins, yarn in hanks, and cloth in the piece. July 28.

11,873. H. H. Lake.—From The L. Weldon Dyeing-Machine Co., United States. Improvements in yarn-dyeing machines. Complete Specification. July 29.

12,379. H. H. Mott and J. Perry. Improvements in the manufacture of decorative wall papers. August 7.

12,403. W. H. Claus. The production of new blue colouring matters on woollen fibre. August 8.

12,689. K. Trobach and G. J. Bruch. A new or improved process of obtaining and bleaching vegetable fibres. Complete Specification. August 13.

12,900. T. Strahan. Improvements in the process of colouring or decorating paper and other fabrics for wall coverings, and for other purposes. August 16.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

14,246. A. Ashworth. Production of an indigo vat. July 30.

11,938. R. Leigh. Sizes and methods for fixing colours in printing and staining paper, &c. July 23.

15,671. L. Schreiner. Improvements in bleaching and methods for producing bleaching agents. July 30.

16,156. T. Holliday. Producing azo-colours on or in cotton or other textile. August 20.

16,368. A. Greg. Bleaching wood bobbins, reels, &c. August 13.

1890.

11,044. M. L. W. Martinot. Improvements in apparatus for washing or bleaching. August 20.

## VII.—ACIDS, ALKALIS, AND SALTS.

## APPLICATIONS.

11,020. J. R. Wylde, M. J. Hammill, and H. Auer. Improvements in or appertaining to the manufacture of carbonate of strontium. July 15.

11,296. A. G. Haddock and J. Leith. Improvements in the manufacture of alkaline carbonates and by-products, and in arrangement of plant therefor. July 19.

11,529. A. Chapman and S. Vickers. *See Class I.*

11,545. F. M. Lyte and J. G. Tatters. Improvement in the conversion of hard burnt into soft burnt magnesia. July 23.

11,605. P. Kestner. Improvements in the manufacture or production of salts of barium. July 24.

11,638. T. Elliot. An improved process for producing sulphate of calcium. July 25.

11,699. G. Nahsen. *See Class XI.*

11,979. W. B. Giles, F. G. A. Roberts, and A. Boake. Improvements in the production of acid sulphates. July 31.

12,251. M. Netto. Improvements in and relating to the manufacture of sulphide of sodium, and to apparatus therefor. August 5.

12,273. G. E. Davis and A. R. Davis. Improvements in the preparation of sulphurous acid. August 6.

12,274. G. E. Davis and A. R. Davis. The preparation of monohydrated sulphuric acid. August 6.

12,275. G. E. Davis and A. R. Davis. The preparation of anhydrous sulphuric acid. August 6.

12,641. C. T. J. Vautin. An improved process and means for producing the generation of chlorine gas for the chlorination of gold or other ores. Complete Specification. August 12.

12,661. R. Dempster and J. Dempster. Improvements in apparatus connected with the manufacture of sulphate of ammonia. August 13.

12,670. J. Simpson. Improvements in the treatment of certain minerals or slag containing phosphate of lime, for the purpose of obtaining the latter in a concentrated form, with or without the production of alkali as a by-product, and in apparatus therefor. August 13.

12,717. H. B. Yardley. The utilisation of gases which form insoluble or partially insoluble precipitates. August 14.

12,782. J. Anderson. An improved method of preserving alkaline sulphide and similar liquors. August 14.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

13,697. H. Grüneberg, H. Fleming, and W. Siepermann. Preparation of cyanogen alkalis, and apparatus therefor. July 30.

15,531. W. L. Wise.—From Solvay and Co. Apparatus for producing gaseous hydrochloric acid. August 13.

15,649. F. Bale. Obtaining ammonia, chlorine, and hydrochloric acid from ammonium chloride, and apparatus therefor. August 6.

15,655. J. Grossmann. Manufacture of nitrite of soda. August 13.

16,213. W. Shapleigh. Producing chloride of lead. August 13.

1890.

312. J. Gimson and Challinor. Appliance for supporting china while being fired. August 20.

9670. J. C. Steele. Packing chloride of lime, &c. July 30.

## VIII.—GLASS, POTTERY, AND EARTHENWARE.

### APPLICATIONS.

11,279. Gibbons, Hinton, and Co. Improvements in the manufacture of ornamental tiles, chromo, relieve, and figured tiles. July 19.

11,560. A. Schaaf. Improvements in drying stoves for bricks, ceramic materials, and such like. Complete Specification. July 23.

11,651. D. Rylands and A. Husselbee. Improvements in the manufacture of glass tubing. July 25.

12,212. G. F. Chance and Chance Bros. and Co., Lim. Improvements in lears for annealing sheets of glass. August 5.

12,506. T. Webster, sen., and T. Webster, jun. An improved process for making articles of earthenware, such as flower pots, preserve jars, cups, and similar goods of improved form either whole or in section. August 9.

12,665. T. Furnival, S. Furnival, A. Furnival, and C. Toft. An improved method of decorating pottery and glass ware. August 13.

12,794. G. A. Marsden. Improved modes of producing novel effects in the decoration of ceramic ware. August 15.

### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

13,262. H. Dudley-Cooper and J. Selby. Connections for glass or porcelain lined metal tubing. August 20.

14,653. A. J. Clay. Glazed bricks and tiles. July 23.

15,801. Sir H. Doulton and J. Slater. Decorating china, &c. August 13.

16,285. R. B. Baugh. Improvements in the brushing-on process in the manufacture of decorated enamelled metal or glass plates, &c. August 20.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

### APPLICATIONS.

10,918. N. W. Curtis and A. E. Carey. Improvements in machinery or apparatus for crushing, mixing, grinding, and delivering materials for the manufacture of cement. July 14.

10,962. R. G. de Vasson and The Société dite La Suberine. Improvements in the manufacture of plastic compounds for building and other purposes, and in apparatus therefor. July 14.

11,115. J. Thomlinson. Improvements in the manufacture of cement. July 16.

11,245. S. McDougall. Improvements in blocks, tiles, or the like for pavements, sidewalks, floors, platforms, steps, or similar places, and in the manufacture thereof. July 18.

11,443. A. Buecher. An improved process for manufacturing light bricks for building purposes from gypsum, &c. Complete Specification. July 22.

11,479. F. Oschwald. New or improved building material. July 22.

11,796. E. Dupont. Manufacture of compound plates or slabs of cement, plaster, and like material. July 28.

11,839. W. L. Wise.—From Ottweiler Chamotte and Thonwaarenfabriken, vormals L. Jochum, Germany. Manufacture of coke bricks, coke plates, and similar articles. July 29.

11,895. L. Johnstone. See Class I.

12,431. J. S. Palmer. Improvements in the manufacture and production of compounds or materials for covering walls, floors, ceilings, or other surfaces, or for architectural moulding, or other analogous purposes. August 8.

12,607. C. George. Improvements in the manufacture of artificial stone. August 12.

12,640. F. F. von Aimbach. A new or improved process for the production of stony polychrome decorations. August 12.

12,738. J. Bowing. Improvements in the means of drying insoluble substances in a divided state, more especially applicable to the manufacture of Portland and similar cements. August 14.

12,894. W. R. Taylor. Improvements in the manufacture of cement. August 16.

### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

14,600. J. Cockburn. Manufacturing pavement, &c. of rubber or rubber compounds. July 30.

14,963. H. Stantou. Fire-resisting floors for buildings. July 30.

14,997. A. Bishop. Method of burning lime, and kilns therefor. August 6.

15,115. W. Joy. Improved cement kilns; mills for grinding cement clinker; and apparatus for separating the finer and coarser particles of disintegrated chalk, and ground cement clinker; and treating cement. August 20.

1890.

1356. A. J. Boulton.—From W. Deissner. Manufacture of rough, glazed, and coloured facing stones. August 13.

9239. D. Hart. Improvements in bricks. July 23.

## X.—METALLURGY, MIXING, Etc.

### APPLICATIONS.

10,940. H. A. van Vught. A hardening preparation to harden iron all through, being far superior to all others. July 14.

11,012. A. G. Brookes.—From O. Petit, France. Improvements in converting and refining metals, and in cupolas or apparatus employed therein. July 15.

11,328. G. Hilgenstock and J. Massenez, trading as Hoerder Bergwerks und Hütten Verein. An improved method of separating sulphur from sulphureous pig iron. July 19.

11,550. W. Hornsby.—From C. Raleigh, South Africa. Improvements in extracting gold and silver from ores and other substances containing the same. July 23.

11,606. P. Rogers and J. Player. Improvements in machinery or apparatus for coating metal plates or sheets with tin, lead, or other metals or alloys. July 24.

11,649. A. Jepson. Improvements in the manufacture of iron and steel castings. July 25.

11,690. P. U. Askham and W. Wilson. Improvements in electro-magnetic separators for extracting metal from slag and the like. July 26.

11,790. M. W. Ponder. Improvements in the manufacture of tin and other metal ware, and in apparatus therefor. July 28.

11,841. F. Wicks. Improvements in the process of alloying metals by vapour, and particularly by sublimated aluminium chloride. July 29.

11,851. C. Rabache. Improvements in apparatus for the extraction or reduction of zinc and other metals from ores or compounds. Complete Specification. July 29.

11,868. W. W. Keys. Improvements in and relating to a process of and furnace for treating copper and its alloys. Complete Specification. July 29.

11,877. H. H. Lake.—From La Société Vanzetti Sagramoso and Co., Italy. Improvements relating to the manufacture of steel. July 29.

12,040. J. Pointon. An improved method of dephosphorising iron ore. August 1.

12,045. G. A. Jarvis. A new or improved method of treating steel or iron previous to casting the same. August 1.

12,200. W. Beatson. Recovering tin from waste tinned iron or steel. August 5.

12,234. J. H. Darby. Improvements in or relating to the manufacture or iron from iron sand or fragmentary or other iron ores. August 5.

12,342. J. B. Hannay. Improvements in obtaining silver and other precious metals from ores, and in apparatus therefor. August 7.

12,387. J. Ketcher. Improvements in means or apparatus for extracting by amalgamation precious metals from their ores or materials containing them. August 7.

12,432. T. Parker and A. E. Robinson. Improvements in the manufacture or production of zinc from compounds thereof. August 8.

12,553. I. A. F. Bang and M. C. A. Ruffin. Improved process of and means for the separation of tin from other metals, more particularly from scrap tin plate and used preserve tins. Complete Specification. August 11.

12,624. A. M. Clark.—From W. G. Horgan, United States. Improvements in fluxes or solutions for coating metals with lead. August 12.

12,626. L. Mond. Improvements in the manufacture of nickel. August 12.

12,627. L. Mond. Improvements in nickel plating. Complete Specification. August 12.

12,641. C. T. J. Vautin. See Class VII.

12,666. W. Brazell. An improvement relating to annealing tin andterne plates. August 13.

12,716. S. O. Cowper-Coles. Improvements in the deposition of alloys. August 13.

12,831. E. Zappert.—From C. Searneo, Austria. An improved manufacture of material to be employed in ship-building, and in the manufacture of armour plates, and for other purposes. August 15.

12,836. R. E. Shill. Improvements in apparatus for separating precious metals from ores or materials containing them. August 15.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

12,033. H. Bessemer, jun. Production of aluminium and apparatus therefor. July 23.

13,277. M. Gledhill. Manufacture of cast metal ingots, &c., and apparatus therefor. July 30.

14,770. E. J. Railton. Gold screening apparatus, August 20.

15,245. E. Grutzner and O. Koehler. Process and apparatus for condensing the metallic vapours produced in zinc furnaces, and furnace applicable in combination with the said apparatus. August 6.

15,552. T. H. Roberts and T. Underhill. Puddling furnaces and appliances connected therewith. July 30.

15,792. L. Grabau. Apparatus for obtaining metals of the alkalis from molten chlorides. August 20.

16,447. C. A. Burghardt. Manufacture of tin plates. August 6.

16,776A. J. B. Abugaray. Universal furnace for fusing and refining metals, alloys, dross, &c., by the dry process or otherwise. July 23.

#### XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

##### APPLICATIONS.

11,065. G. A. Oncken. Improvements relating to the impregnation of organic, fibrous, and cellular matter, by means of an electric current, and apparatus therefor. July 15.

11,187. A. W. Armstrong. Improvements relating to the manufacture of articles by electro-deposition, and to compositions for use in such manufacture. July 17.

11,319. H. Howard. Improvements in apparatus to be used for electric welding. July 19.

11,533. M. G. Farmer. An improved electrolytic apparatus for forming copper ingots. Complete Specification. July 23.

11,601. A. H. Cowles.—From F. W. Matthiessen, United States. A process for obtaining metals in molten condition from their fused oxides by electrical action. July 24.

11,680. M. G. Farmer. An improved apparatus for producing sheets of metal by electro-deposition. Complete Specification. July 25.

11,699. G. Nahsen. Process for obtaining chlorine and bromine by the aid of electricity. Complete Specification. July 26.

11,842. G. L. Anders and J. R. Shearer. Improvements in the deposition of metals by electrolysis. July 29.

12,562. W. O. Stanley. New or improved means or apparatus for generating electricity. August 11.

12,628. H. E. Fowler. Improvements relating to the welding of metals by electricity. Complete Specification. August 12.

12,830. H. H. Lake.—From G. A. Johnson and S. L. Holdrege, United States. Improvements in secondary batteries and electrodes therefor. August 15.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

7005. A. Schanschieff. Galvanic batteries. July 30.

12,503. F. V. Andersen. Dynamo-electric machines. August 6.

14,871. E. Hopkinson and D. Appleton. Improvements in vessels containing liquids used in electroplating, or the electrolyte of storage batteries. July 30.

14,971. P. J. R. Dujardin. Secondary batteries. August 6.

15,230. Sir W. Vavasour. Electric batteries. August 20.

17,060. T. Paeker. Electrical furnaces and their manipulation. July 30.

19,080. H. T. Cheswright. Plates for secondary or other voltaic batteries. August 13.

1890.

3731. The Mining and General Electric Lamp Company, Limited, and D. G. FitzGerald. Positive elements of secondary batteries, applicable as cathodes in electrolysis. August 13.

4246. Sir C. S. Forbes, Bart. Primary batteries, and switching apparatus therefor. August 13.

7988. C. L. Coffin. Welding metals electrically. August 6.

7989. C. L. Collin. Welding metals by electricity. August 6.  
 7990. C. L. Collin. Electric welding. August 6.  
 7991. C. L. Collin. Electric welding. August 6.  
 7992. C. L. Collin. Electric welding. August 6.  
 7993. C. L. Collin. Welding metals electrically. August 6.  
 7994. C. L. Collin. Welding metals electrically. August 6.  
 9857. M. W. Dewey. Welding, soldering, brazing, &c., by electricity, and apparatus therefor. July 30.  
 10,181. T. M. Foote. Secondary batteries. August 13.  
 10,515. A. J. Jarnan. Connector for the electrodes and cells of electrical batteries. August 13.

## XII.—FATS, OILS, AND SOAP MANUFACTURE.

### APPLICATIONS.

- 11,360. J. Baptista. Facilitating the use of mineral or other non-saponifiable oils in the manufacture of soaps. July 21.  
 11,629. W. N. Hartley and W. E. B. Blenkinsop. Improvements in decolourising vegetable oils in the manufacture of pale drying oils and varnishes, and apparatus therefor. July 24.  
 11,956. G. Tall. Improvements in refining cotton-seed and other fatty oils. July 31.  
 12,000. F. Walton. A process for oxidising and drying vegetable oils. July 31.  
 12,047. C. Wimpess. An improved washing or laundry powder. August 1.  
 12,373. I. Roos. An improved manufacture of saponifiable fatty matter from wool fat. August 7.  
 12,864. E. J. T. Digby. Improvements in the manufacture of soap. August 16.

### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

- 14,914. W. G. Little. Improvement in soap. August 6.  
 16,518. T. Anyon. Composition of soap. August 20.

1890.

9614. P. A. Newton.—From The Chesebrough Manufacturing Company. Manufacture of compressed cakes of soap. July 30.

## XIII.—PAINTS, PIGMENTS, VARNISHES, AND RESINS.

### APPLICATIONS.

- 10,976. T. H. Davis and A. H. Davis. The purification of coal tar and rendering same a suitable paint or coating or iron or steel structures. July 15.  
 11,359. J. Baptista. Facilitating the use of mineral or other non-oxidising oils in compounding paints and paint oils. July 21.  
 11,602. G. Bischof. A process for manufacture of white lead. July 24.  
 11,629. W. N. Hartley and W. E. B. Blenkinsop. See Class XII.  
 11,640. M. N. d'Andria. Improvements in the manufacture of white lead and nitrate of ammonia. July 25.  
 11,780. J. A. Maynard. A novel manufacture of oil paint in sheets. July 28.

- 12,085. J. J. Lee. The application of certain waste materials for the production of a new or improved "mineral white," and improvements in the process of manufacture. August 1.

- 12,123. G. Macaulay-Cruikshank.—From E. Pietzcker, Germany. Improvements in the preparation of rapidly-drying resinous and other oils or varnishes. August 2.

- 12,230. T. Christy. An improved material for use as a substitute for gutta-percha, rubber, oiled, and other transparent tissues, and for other purposes. August 5.

- 12,414. E. V. Gardner. Improvements in the manufacture of white lead, and in apparatus employed in such manufacture. August 8.

- 12,474. J. C. Martin. Improvements in pigments or paints. August 9.

- 16,684 J. S. Fairfax.—From F. Crane, United States. Improvements in or relating to varnishes or other coatings. August 13.

### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

- 14,569. F. Eckstein. Translucent plastic compositions. July 23.

- 14,797. R. Barton. Paints or compositions. July 23.

- 17,236. G. E. Heyl. Insulating compositions for cables. August 20.

1890.

9822. A. J. Boulton.—From W. Kiel. Manufacture of vulcanised plastic compounds. August 6.

9897. J. Gordon. White lead pigments. August 13.

- 10,130. M. Alsberg. Producing minimum. August 6.

- 20,616. G. R. B. Kempton. Blue for laundry and other purposes. August 13.

## XIV.—TANNING, LEATHER, GLUE, AND SIZE.

### APPLICATIONS.

- 11,397. J. M. Pujos. Improvements in tanning. July 21.  
 12,751. J. B. Scammell and P. W. Ellis. Improvements in tanning. August 14.

## XV.—AGRICULTURE AND MANURES.

### APPLICATION.

- 11,800. H. Moon. Improvements in the manufacture of artificial manure. July 28.

### COMPLETE SPECIFICATIONS ACCEPTED.

1890.

575. M. C. Ginster. Manufacture of manures or fertilisers. August 13.

9729. H. H. Lake.—From H. Neuminger. Manufacture of acid phosphates for use as manure. July 23.

## XVI.—SUGARS, STARCHES, GUMS, Etc.

## APPLICATIONS.

11,473. J. Duncan. Improvements in the treatment of molasses or syrup for the purpose of improving its flavour. July 22.

11,612. D. Stewart. Improvements in separating saccharine juices, and in centrifugal apparatus therefor, and applicable for other analogous purposes. July 25.

11,683. J. Duncan. The treatment of sugar beet and other roots for the conservation and concentration or drying thereof. July 25.

12,048. C. Wimpess. An improvement in the manufacture of self-glazing starch. August 1.

12,635. H. H. Lake.—From L. Sterberg, United States. Improvements in sugar refining. Complete Specification. August 12.

## COMPLETE SPECIFICATION ACCEPTED.

1889.

13,555. R. Raeymaeckers. Process and apparatus for concentration of saccharine and other liquids, and obtaining crystals thereof. August 6.

## XVII.—BREWING, WINES, SPIRITS, Etc.

## APPLICATIONS.

11,057. A. M. Clark.—From A. Bohringer, Germany. Improved process for extracting tartaric acid from tartar wine lees and kindred substances. July 15.

11,226. E. Craddock. Improvements in apparatus for the treatment of grain for the production of malt. July 18.

11,249. A. Regel. Improvements in pasteurising apparatus. July 18.

11,554. G. F. Redfern.—From J. F. H. Gronwald, E. H. C. Oehlmann, and G. H. Neuhauss. *See Class XVIII. A.*

11,613. T. Reade. A novel preparation for fining or frothing, or imparting a foam to, beer and other fermented beverages. July 24.

11,672. C. O. Gridley and A. Bishop. Improvements in apparatus for the manufacture of finings and other analogous materials. July 25.

12,260. A. W. Ellis. Improvements in distillation and in apparatus therefor. Complete Specification. August 5.

12,834. F. Sauer. A new beverage termed "Malt wine." August 15.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

14,928. A. Manbré, sen. Manufacture of refined malt and extract from barley malt, or barley malt and amylaceous substances; and treatment of the spent grains for production of cattle food. July 23.

15,064. A. Manbré, sen. Manufacture of methylated spirit, and treatment of the by-products. July 23.

16,093. W. P. Thompson.—From La Société Générale de Maltose. Process for the saccharification and fermentation of amylaceous matter. August 13.

1890.

4982. M. Hanford. Apparatus for cooling and aerating hot worts. August 20.

## XVIII.—CHEMISTRY OF FOODS, SANITARY CHEMISTRY, AND DISINFECTANTS.

## APPLICATIONS.

*A.—Chemistry of Foods.*

10,950. M. Persson. Improvements in or relating to the preparation of condensed cream. Complete Specification. July 14.

10,961. N. Etienne and A. Delhaye. An improved method of preparing peptonised soluble meat and peptone. July 14.

11,554. G. F. Redfern.—From J. F. H. Gronwald, E. H. C. Oehlmann, and G. H. Neuhauss, Germany. Improvements in apparatus for sterilising milk and other liquids, and for drawing off or decanting the same. Complete Specification. July 23.

11,586. V. Tobias and H. Fischer. A liquid chocolate, free from fat, and preservable. Complete Specification. July 24.

12,298. C. Huelser.—From F. J. Chevet, France. Process for making, and composition of, a conserved product for coffee and milk. August 6.

12,310. S. S. Bromhead.—From F. J. Chevet, France. A preparation of maté and milk. August 6.

12,318. J. G. Lorrain.—From F. J. Chevet, France. A new or improved alimentary product, and process of manufacturing the same. August 6.

*B.—Sanitary Chemistry.*

11,048. T. Douglas. The removing from sewers of the gases generated therein, and for the disinfecting and disposing of them. July 15.

12,290. F. Boshardt.—From J. Roland, France. A process and apparatus for treating refuse. August 6.

*C.—Disinfectants.*

12,323. H. H. Lake.—From W. F. Simes, United States. An improved compound for disinfecting and other purposes. Complete Specification. August 6.

## COMPLETE SPECIFICATIONS ACCEPTED.

*B.—Sanitary Chemistry.*

1889.

14,500. T. Jones and J. Jones. Materials for purifying sewage or polluted water from works, &c. July 30.

*C.—Disinfectants.*

1890.

8838. D. C. Mackenzie. Absorbent, deodorising, and disinfectant preparation. August 6.

## XIX.—PAPER, PASTEBOARD, Etc.

## APPLICATIONS.

10,992. A. Morton and F. L. Leech. Improvements in the method of and means for waterproofing paper. July 15.

11,021. J. B. Atherton.—From L. D. Beck, United States. Improvements in the manufacture of Manila paper. July 15.

11,420. J. Lewis. Improvements in paper used for photographic printing. July 22.

11,754. W. Thomson and J. Richards. Improvements in the manufacture of waterproof cardboard, and waterproof cardboard boxes or other receptacles. July 28.



12,139. W. Rogers. Improvements in the preparation or treatment of paper for copying letters, documents, and the like, including such as are done with a type-writing machine, and improvements in chemical compounds, or preparations therefor. Complete Specification. August 2.

12,375. W. Marshall Campbell-Caldender. The manufacture of a new material designed to serve as a substitute for bone or celluloid. Complete Specification. August 7.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1890.

4959. C. Kellner. Boilers for manufacture of sulphite cellulose, and method of heating same. August 6.

5128. C. Kellner. Modifying the properties of fibres used in the manufacture of paper. July 23.

5500. E. G. Wrigley. Manufacture of absorbent sheets, rolls, or pads of paper. August 20.

6158. P. Whiteside. Absorbent writing paper. July 30.

8353. J. Hawke. Manufacture of copying paper. August 6.

#### XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

##### APPLICATIONS.

11,189. W. R. Lake.—From Kalle and Co., Germany. Improvements relating to the preparation of hydrochlorides, hydrobromides, and hydriodides of peptone. July 17.

11,196. G. D. Bowie. Phosphated salt: sal. phosph. July 18.

12,729. H. Cumber. A compound of sugar with the salts of phosphorus for dietetic purposes. August 14.

#### XXI.—PHOTOGRAPHIC PROCESSES AND MATERIALS.

##### APPLICATIONS.

11,104. E. Edwards.—From O. Moh, Germany. An improved method of preparing photographic plates of mica. Complete Specification. July 16.

11,420. J. Lewis. See Class XIX.

11,945. B. J. Edwards. Improvements relating to the manufacture of flexible films for photographic and other purposes, and to apparatus for use in such manufacture. July 30.

11,986. F. T. Knott. An improvement in the application of photography to textile fabrics. July 31.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

11,701. J. Molloch. A glass plate or other transparent substance coated with a non actinic film, and used as a negative for photographic and other purposes. July 23.

1890.

11,104. E. Edwards.—From O. Moh, Germany. An improved method of preparing photographic plates of mica. August 20.

#### XXII.—EXPLOSIVES, MATCHES, Etc.

##### APPLICATIONS.

10,935. A. Hislop.—From J. Storer, Australia. Improvements in the making of wax vestas. July 14.

11,018. W. P. Thompson.—From S. D. Smolianoff, United States. Improvements in explosive compounds. Complete Specification. July 15.

11,202. G. Bruck. Improvements in the manufacture of headless matches. July 18.

11,720. R. J. Jones and G. Worrall, on behalf of Woodhouse and Rawson, United, Limited. Improvements in connexion with electrical fuses. July 26.

11,745. H. J. Haddan.—From J. H. Voelkel, Germany. Improved vacuum drying apparatus for explosive substances. Complete Specification. July 26.

12,094. R. Morris. An electric fuse for firing guns. August 1.

12,785. A. L. Brociner. The manufacture of a new explosive. August 15.

#### COMPLETE SPECIFICATION ACCEPTED.

1890.

8481. P. Ward and E. M. Gregory. Priming and detonating composition. July 23.

# THE JOURNAL

OF THE

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1891.

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Mr. W. C. Young. "On Standard Sperm Candles."

Feb. 2nd.—Dr. W. S. Squire. "The Artificial Production of Cold Theoretically and Practically considered."

## Communication.

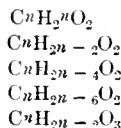
### CONTRIBUTIONS TO THE ANALYSIS OF FATS.

BY J. LEWKOWITSCH, PH.D., F.I.C., F.C.S.

(Read before Section B., British Association, Leeds.)

On an occasion like this it may perhaps not be out of place for me, before entering on the original part of my paper, to give in short outlines a review of the analytical methods which are at present being employed in the analysis of fats. And here one cannot help being struck with the fact that, while the manufactures dealing with fats and oils have been enormously developed, and are playing a most important part in the list of chemical industries, the analytical research into the ultimate composition of them has been sadly neglected. From the ever-memorable researches of Chevreul, which created the scientific basis for these industries, up to a few decades the analytical material at the command of the chemist has been a very scanty one. The examination of physical properties and some chemical methods, perhaps more of a rule of thumb than of a scientific character, have been nearly the only guides for the analyst. The growing adulteration, almost developed to a fine art, especially that of butter, has created a necessity for inventing new methods; and, thanks to this pressure, methods have been found which, though very far yet from allowing an insight into the bewildering and almost inextricable mixtures of fatty acids occurring in nature, have given valuable material into the hands of the chemist, upon which we may hope to build up a complete analytical examination of fats.

The fats and oils are, as well known, the glycerides of fatty acids belonging to the series—



In nature occur exclusively the triglycerides. There has been found by Will and Reimer in old rape oil the diglyceride of erucic acid,  $C_{21}H_{41}(OH)(OC_{22}H_{41}O)_2$ , but this exception is only an apparent one, as it is most likely that the rape oil had undergone that change which is generally known as turning rancid, so that free erucic acid had been formed, while dierucic acid separated as a solid mass. In some cases this process of decomposition may have been progressing further, and the fats may contain a larger amount of free fatty acids, until they may reach as high a percentage as 80—90 per cent., which is known to occur in the case of old palm oil. However, we must still assume that in nature only the triglycerides are found, as proved by the examination of freshly prepared fats.

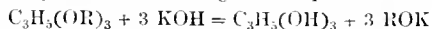
The analytical examination of fats will naturally bear on the examination of the constituents into which they are resolved on undergoing hydrolysis or saponification, namely, into glycerol and fatty acids. The first operation will therefore be to carry out this splitting up into the components; the fatty acids, being mostly insoluble in water will be separated, while the glycerol will remain in aqueous solution.

The exact determination of the glycerol in fats has been, up to a very short time ago, a difficult operation, so much so that it could only be determined approximately; it was extracted by means of a mixture of ether and alcohol, after the neutralised aqueous solution had been boiled down nearly to dryness, from the residual salts. But on evaporating the solution on the water-bath, the glycerol soon reached a concentration when it began to volatilise, which rendered, of course, the result inaccurate. Although this source of error might have been obviated by the somewhat tedious method to free the glycerin from the last traces of ether and alcohol and water by allowing it to stand in a desiccator until the weight remained constant, still, this method necessarily gave inaccurate results, as the glycerin had finally to be incinerated in order to deduct the amount of ashes. But along with the glycerol other organic substances, which would naturally be contained in such a

crudely prepared glycerin, would be burnt at the same time and therefore the result would be found too high.

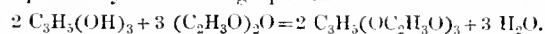
The elegant method of Benedikt and Zsigmondy, which is based on the oxidation of glycerol to oxalic acid by means of potash permanganate will, for this very reason, although permitting a higher grade of accuracy, leave some uncertainty as some organic impurities might also yield oxalic acid.

The indirect method of determining the glycerol by the amount of potash required in saponifying the *neutral fat*, i.e., the triglycerides, according to the equation—



has all the faults of an indirect method and therefore it need not be discussed here.

I proposed therefore to use Benedikt's and Cantor's acetin method, which as I have shown before gives very concordant and accurate results. The method is based on a process expressed by the following equation—



On hydrolysing the triacetin the quantity of alkali required for it and consequently the glycerol may be easily determined. When the fats have been saponified the glycerin may be extracted, as indicated above, with a mixture of ether alcohol, and the latter evaporated and the substance finally dried in the desiccator. It is not necessary to wait until the weight becomes constant, as a percentage of water up to 10 per cent. does not interfere with the acetin process; furthermore, a determination of ashes becomes superfluous. The results I obtained in this way are very satisfactory indeed, and I think this method would recommend itself to the analyst for the estimation of glycerol in wine and beer, where impure crude glycerins are also obtained.

The examination of the fatty acids will in the first instance be directed to the determination of their total amount. If all the fatty acids were insoluble in water it would be easy enough. But as the lower members of the fatty acid series are more or less soluble in water the problem becomes a more complicated one and we are no longer able to apply such exact methods as the mineral analysis gives us at hand. For we must consider that we may have to deal with a mixture of acids of various molecular weights. Therefore recourse has to be taken to the so-called quantitative reactions.

It will perhaps be useful to arrange these reactions under a tabular form in a similar way as it is being done in qualitative analysis of mineral substances (see following table). *a* and *b* are separated by boiling the fatty acids with

#### FATTY ACIDS.

<i>a. Soluble Acids.</i>		<i>b. Insoluble Acids.</i>	
	<i>α-Saturated.</i>		<i>β-Unsaturated.</i>
1.	$C_{17}H_{34}O_2$ yields on oxidation	.....	Dihydroxystearic acid.
2.	$C_{19}H_{38}O_2$ " " .....		Tetrahydroxystearic acid.
3.	$C_{21}H_{42}O_2$ " " .....		Hexahydroxystearic acid.
4.	$C_{17}H_{34}O_3$ " " .....		Trihydroxystearic acid.

water and washing them out repeatedly with boiling water. The insoluble fatty acids are collected on a filter and weighed. The percentage of insoluble acids—the so-called Hehner's value—is a very valuable criterion for the nature of some fats. In the case of fats resembling tallow, Hehner's value will be about 95 per cent., whilst, e.g., for butter the upper limit is 88 per cent. This method enabled chemists to test the butter for the presence of oleomargarine, until the adulterator has found in the cocoa-nut oil a ready means to gauge his adulteration in such a way as to accommodate his mixture to Hehner's test.

It became therefore necessary to examine the soluble fatty acids, and this is done by determining the so-called Reichert's value. This latter is found by saponifying a fat

with caustic alkali, decomposing the soap by means of sulphuric acid and distilling the mixture, when the volatile, water-soluble acids pass over, and may be estimated by titration with caustic potash. This method has been specially devised for the examination of butter fat and represents at present the best means of stating the genuineness of butter. But it is to be feared that the inventiveness of the adulterator will even mar this test by finding fats which, when added to an artificial butter, will produce a Reichert's value approaching that of a genuine butter. Such fats would be, for instance, the glycerides of valeric acid as they are found in porpoise oil.

If we express the amount of fatty acids, both soluble and insoluble, by the number of milligrammes of caustic potash

required for their neutralisation, we shall, on adding these values, find, of course, the number of milligrammes of caustic potash necessary to saturate all the fatty acids contained in a fat. It is evident that this value may be determined at once at the outset, and this constitutes the valuable method devised by Kottstorfer and known to give the so-called Kottstorfer's value, *i.e.*, the number of milligrammes of caustic potash required for the fatty acids of one gramme of fat. This value has also been called the saponification value, not quite correctly perhaps, if by saponification be understood the process of hydrolysing fats and transforming the fatty acids thereof into soap. Most fats contain, for some time after they have been rendered free, fatty acids, and these are at the same time transformed into soaps along with the fats hydrolysed. The Kottstorfer value in most cases comprises therefore the sum of two distinct values, namely, that for neutralising the free fatty acids, and that for saponifying the neutral fats; this latter value has appropriately been called the ether value.

The determination of the free acid in a fat will, therefore, constitute the next step, and is easily carried out by titrating the fat with potash in alcoholic solution, using phenolphthaleïn as an indicator. We shall not find the actual amount of free fatty acids, as we do not know their molecular weight, but we shall have again a value, always expressed in milligrammes of potash, which allows us to approximately calculate the amount of free fatty acids, if we make the assumption that the free fatty acids have the same mean molecular weight as the bulk of fatty acids contained in the fat.

To find the mean molecular weight, we shall best examine the insoluble fatty acids *b*. As they represent, as a rule, a mixture of different acids, we can only determine their mean molecular weight. We find this simply by titrating with caustic potash, and expressing the result in milligrammes of caustic potash per gramme of fatty acid. This value *A*, the so-called acid value, allows us to calculate the molecular weight according to the following proportion:—

$$M : 56 \cdot 1 = 1 : A ; M = \frac{56 \cdot 1}{A}$$

If molecular weights are found, which lay near either end of the following table:—

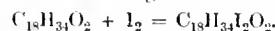
Acid.	Formula.	Mol. W.
Myristic acid .....	$C_{14}H_{28}O_2$	228
Palmitic acid .....	$C_{16}H_{32}O_2$	256
Linolenic acid .....	$C_{18}H_{34}O_2$	278
Linoleic acid .....	$C_{18}H_{32}O_2$	280
Oleic acid .....	$C_{18}H_{34}O_2$	282
Stearic acid .....	$C_{18}H_{36}O_2$	284
Ricinolic acid .....	$C_{18}H_{34}O_3$	298
Erucic acid .....	$C_{22}H_{42}O_2$	338

it will be seen at once in which direction further research into the nature of the acids will have to be carried on. But most of the fats contain fatty acids whose molecular weights range from 256 to 284, and small quantities of ricinolic, &c. acids may easily escape being noticed, if conclusions were to be drawn from the mean molecular weight only.

The fatty acids given in the last table belong to different series, which we may at once divide into two classes, saturated and unsaturated acids, *a* and *b*. The presence of unsaturated fatty acids will best be detected by applying Hübl's reaction—that is, by determining the so-called iodine absorption values. This test adds another valuable method to those enumerated above, and gives, in a great many instances, most indispensable hints as to adulterations, for instance, of tallow with cotton-seed oil.

If the unsaturated fatty acids present besides the saturated acids consisted of oleic acid only, it would be easy

to calculate from the amount of iodine absorbed the exact quantity of oleic acid according to the following equation—



For every gramme of iodine absorbed there must be present  $\frac{284}{254} = 1 \cdot 1102$  grms. oleic acid. But as a rule we must be prepared to pre-suppose the presence of other acids belonging to the other series of unsaturated acids, and their separation from the saturated acids and from one another becomes therefore necessary.

The solubility of the lead salts of the unsaturated fatty acids in ether, which does not dissolve the lead salts of the saturated fatty acids, admits in the first instance of the separation of the unsaturated fatty acids from those belonging to the series  $C_{18}H_{34}O_2$ . It has hitherto been supposed that this solvent allows the complete separation of the acids *a* and *b*, but, as I shall show later on, this may be doubted. However, for practical purposes, we may assume here that the separation is a complete one. The acids *a* will have to be resolved by fractional crystallisation of their lead—or barium—or magnesium salts into their single constituents. The determination of their mean molecular weight will serve as a convenient guide. If a value *M* be found lying between 256 and 284—the mol. weights for palmitic and stearic acids—and there is reason to assume that these two acids only are present, their actual amount *x* and *y* may be determined from the following two equations—

$$x + y = 100.$$

$$2 \cdot 56 x + 2 \cdot 84 y = M.$$

If therefore the fatty acids *b* contain exclusively oleic, palmitic, and stearic acids, it would be quite possible to determine their absolute amounts. Of course, the iodine absorption value does not indicate at all the nature of the unsaturated fatty acids, which may belong to one or more of the four different series represented in the table sub-numbers 1–4 by four acids actually found in some fats. An elegant method to at least qualitatively prove the presence of these acids has been given by Hazura. His method is based on the fact that the unsaturated acids, like perhaps all unsaturated derivatives of the fatty series, are oxidised in alkaline solution, by means of potash permanganate in such a way that each  $CH = CH$  group is transformed into a  $CH(OH) - (OH)CH$  group. Thus oleic acid would yield on oxidation dihydroxystearic acid, &c., as indicated in the table. The resulting acids are well characterised, and may be separated in a convenient way, as has been shown by Hazura.

At the hand of a practical example the usefulness of the above-mentioned methods may be clearly demonstrated. I examined the fat extracted from the Sawarri nuts, and found the following figures:—

Hegner's value .....	96.9 per cent. (insoluble acids)
Reichert's value .....	0.65 per cent. (soluble acids)
Kottstorfer's value .....	199.51
Mean molecular weight of the insoluble acids .....	272.82
Free acids (Mol. W. 272.8) .....	2.4 per cent.
Glycerol .....	9.1 "
Iodine value of the fatty acids .....	51.5

The low Reichert's value and the high Hegner's value show that the amount of volatile soluble acids may be neglected. The mean molecular weight, 272.82, seems to point to the presence of oleic, palmitic, and stearic acids only. In order to resolve the insoluble fatty acids into their components, they were transformed into their lead salts by double decomposition of the neutral sodium salts with lead acetate, and repeatedly extracted with ether. It was, however, found that the ether did not dissolve the lead salts of the unsaturated acids completely, for the insoluble lead salts yielded, even after a great many extractions, on being decomposed with sulphuric acid, fatty acids which showed by their capacity of absorbing iodine that they

still contained a certain amount of unsaturated fatty acids. As further extraction seemed to prove of no use, inasmuch as the iodine value of the fatty acids constituting the insoluble lead salts remained constant at 5.8, I had to renounce the hope of bringing the separation to an end in this way. It was thought possible to concentrate, so to speak, by fractional crystallisation of the acids yielding the insoluble lead salts, the unsaturated acid into one fraction, and therefore the insoluble lead salts were decomposed by means of sulphuric acid, and repeatedly boiled with water slightly acidulated with this acid in order to remove the last traces of lead salts, which, by being enveloped in the free fatty acid, easily escaped decomposition.

The free acids were dissolved in absolute alcohol, and yielded seven consecutive crystallisations; the filtrate from the last crystallisation was freed from alcohol by heating on the water-bath, thereby giving an eighth and last quantity of acids. The melting points of these eight fractions were as follows:—

Fraction.	Melting Point.	Solidifying Point.
	°C.	°
1st	60.0–61.0	58.3
2nd	61.2–65.5	60.0
3rd	61.0–61.5	60.0
4th	61.0–63.0	60.0
5th	61.0–62.0	58.3
6th	56.5–59.5	54.5
7th	57.5–60.0	55.5
8th	46.5–50.0	44.4

The iodine absorption values of the first, fourth, and eighth fraction were 1.66, 1.65, and 25.05.

The first two values lie within the experimental errors of the method, and may therefore be neglected; the last value, however, demonstrates clearly that the unsaturated acid accumulated in the last fraction. The determination of the mean molecular weight of the three fractions (first, fourth, and eighth) showed for the—

1st fraction .....	258.4
4th „ .....	262.0
8th „ .....	309.0

pointing to the explanation that the first fraction is nearly pure palmitic acid, which in the higher fractions is being partly replaced by stearic acid. A further study of these acids by means of fractional crystallisation of their lead or magnesium salts will give a final answer as to their ultimate composition.

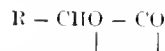
The soluble lead salts were decomposed in the same way, the liquid acids repeatedly washed and freed from the last traces of water. After having been kept for some time in a desiccator the liquid acids became of a butter-like consistency; from which, however, it was not possible to separate the solid constituent. The mean molecular weight of the acids was found at 297.5, the iodine value 68.85, thus proving, in addition to the physical properties of the acid, that the liquid acids contained besides the supposed oleic acid (molecular weight 282, iodine value 90), one or more other acids. The oxidation of the acids by means of potash permanganate according to Hazura was expected to give a better insight into the composition of these fatty acids. As the iodine value has been found so low it was hardly likely that acids of the series  $C_nH_{2n-4}O_2$  and  $C_nH_{2n-6}O_2$  would be present. The detection of any hydroxylated fatty acids will be mentioned later on.

30 grms. of the unsaturated acids—as I will call them—were dissolved in 36 cc. caustic potash of 1.27 sp. gr. and the soap dissolved in 2 litres of distilled water. To this solution was gradually added a 1½ per cent. solution of potash permanganate until the liquor remained pink for some time; 2,500 cc. of the permanganate solution was added. I hasten

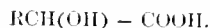
to explain that this *modus operandi* is against Hazura's prescriptions, whose papers I knew at that time from abstracts only. Hazura prescribes in his later publications that 30 grms. of the acids, neutralised as above, should be mixed with 2 litres of a 1½ per cent. solution of potash permanganate, and that after standing for 10 minutes the surplus of permanganate should be reduced by means of sulphurous acid. Therefore it will be easily explained why I found considerably quantities—over 5 grms.—of dibasic water-soluble acids.

The slight excess of potash permanganate was removed by means of sulphurous acid, and the potash salts decomposed by sulphuric acid, when a precipitate of insoluble acids was obtained. These were collected and dried under a desiccator; their weight was 28 grms.

By means of purified ether these acids were extracted four times, 2 litres being used each time. Thus, four extracts were obtained, while the insoluble acids remained behind. The first extract amounted to about 18 grms., the substance was jelly-like, dissolved readily in alcohol, but did not crystallise from it. As the substance did not absorb iodine, it was first considered to be a saturated fatty acid, the lead salt of which had been dissolved by ether. But on further examination it was found that it was no acid, for when shaken up with water and titrated with decinormal potash after adding a little phenolphthalein, it was not neutralised at once, the pink colour only gradually disappearing after some time. Alcoholic potash, however, saponified the substance readily, giving a saponification value of 271.2. The fatty acid obtained from the soap gave an acid value of 182.5, corresponding to a molecular weight of 305. These figures, on account of the small quantities with which I had to carry out my experiments, do not admit of certain conclusions, and this much seems only proven that the substance is an inner anhydride of a hydroxylated fatty acid of the general formula—



pointing to the existence of an acid of the formula—



The second, third and fourth extracts were nothing else than small quantities of the bulk of the acids which are not altogether insoluble in ether, and were later on added to them.

The bulk of the acids was repeatedly boiled out with water without, however, losing in weight, whereby the absence of linolic and isolinolic acids was proved. The acids could therefore, providing the original acids contained 18 atoms of carbon, only consist of dihydroxystearic, trihydroxystearic, and sativic acids. On repeatedly recrystallising them from absolute alcohol it was, however, found that only one acid was present, all fractions yielding acids of a melting point lying between 125° and 130°. The last filtrate was evaporated to dryness and gave as residue a dark-coloured acid melting between 117° and 119°. The absence of sativic acid,  $C_{18}H_{32}(OH)_2O_2$ , melting point 173°, and also of isotrihydroxystearic acid,  $C_{18}H_{34}(OH)_3O_2$ , melting point 110°–111°, was therefore proved. The oxidised acid could only be dihydroxystearic acid,  $C_{18}H_{34}(OH)_2O_2$ , melting point 137°, mixed perhaps with small quantities of trihydroxystearic acid, melting point 140°–142°. The determination of the molecular weight and an elementary analysis could easily decide this question. The acid was therefore recrystallised from absolute alcohol, and the crystals showing every sign of purity, melting point 132°–133°, analysed.

0.4059 grm. gave 0.4224 grm.  $H_2O$  and 1.008 grm.  $CO_2$ , wherefrom—

Experiment.	Theory for $C_{18}H_{34}(OH)_2O_2$ .	Theory for $C_{18}H_{32}(OH)_2O_2$ .
C	67.70	68.3
H	11.56	11.8
		65.06
		10.84

The molecular weight, determined by titration with half normal caustic potash, was found, for a monobasic acid, to be 316·19; theory requires for dihydroxystearic acid, 316. The acid may therefore be considered as pure dihydroxystearic acid formed from oleic acid.

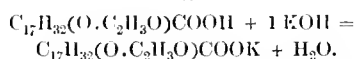
The water-soluble acids were obtained by neutralising the sulphuric acid after the dihydroxystearic acid had been filtered off and boiling the liquor down until potassium sulphate began to separate. The solution was now acidulated and the dibasic acid extracted with ether. After repeated crystallisation from water (charcoal having been used to remove colouring substances) an acid was obtained of the melting point of 126°–130°.

On titrating with caustic potash, an acid value of 622 was found corresponding, for the unavoidable assumption that a dibasic acid was present, to the molecular weight 180·2. Azelaic acid, melting point 106°, has the molecular weight 188; suberic acid, melting point 140, has the molecular weight 174; it is therefore not to be doubted that I had in my hands a mixture of azelaic and suberic acids.

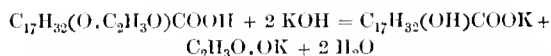
I hope to get a fresh supply of the somewhat rare Sawarri nuts in order to repeat the oxidation and determine the nature of the supposed hydroxylated fatty acid.

For the detection and determination of hydroxylated fatty acids (No. 4 of our table) Benedikt had proposed a method based on the principle that hydroxylated fatty acids, on being heated with acetic anhydride, exchange the hydrogen of their hydroxyl group or groups with the radicle of acetic acid, whilst those acids which do not contain any hydroxyl group remain unaltered when subjected to the same operation. On neutralising and saponifying such hydroxylated acids Benedikt found two values, expressed in milligrammes of caustic potash, the difference of which yielded his "acetyl value." As an illustration he gave the following two equations, *e.g.*, for ricinolic acid—

I.



II.



I have, however, recently shown (Proc. Chem. Soc. 1890, 72, 91; this Journal, 1890, 660), that Benedikt's method may lead to erroneous results, as all fatty acids on being boiled with acetic anhydride yielded the anhydrides of the fatty acids, and I proved that Benedikt's "acid values" obtained according to the first equation had no quantitative meaning at all.

In my first note in the Proceedings I had only shown that working after Benedikt one may even get acetyl values for oleic, palmitic, and stearic acids, which, of course, ought to be an impossibility.

As I gave no explanation in my first note, it called forth a reply from Benedikt,\* who, without waiting for my second note, simply declared my results to be wrong. The importance of the method requires further explanation and proof on my part. Before giving these I shall have to refute Benedikt's alleged explanations for my supposed errors. Benedikt assumes two causes of error: firstly, that the acetic anhydride I used has not been pure; secondly, that I did not wash the fatty acids free from acetic anhydride before titration.

As to the purity of the acetic anhydride I used I may state that it had the following boiling point:—

	Degrees.	Per Cent.
Beginning of boiling at .....	134	
Distilled from .....	134–135	3
" .....	136	7
" .....	136·5	16
" .....	137	28
" .....	137·5	46
" .....	138	60
" .....	138·5	79
" .....	139	90

\* Chem. Zeit. 1890, No. 51.

The remaining 10 per cent. were only slightly coloured.

On hydrolysing the acetic anhydride by means of caustic potash, I found the following saponification values:—

1. 7·303 grms. were placed with a large quantity of water into a beaker and titrated with half normal caustic potash; used 285·95 cc., corresponding to a saponification value of 1080; theory requires 1100.

2. 2·5520 grms. were titrated in the same way; used 98·2 cc. half normal potash. Saponification value found, 1087.

3. 1·1516 grms. were saponified by means of half-normal alcoholic potash; used 43·75 cc. Saponification value found, 1066.

These analytical results entirely do away with Benedikt's first objection.

As to the second assumption of Benedikt that I did not wash the resulting fatty acids until free from acetic anhydride, I have to say that I did not explicitly state that this had to be done; for such, indeed, would have been the very first objection I should have raised to myself in order to explain the unexpected results. As a matter of fact, the substances had been washed repeatedly with boiling water, and the precaution was even used not to test for any acid with litmus paper, but with a very sensitive litmus solution. A complete refutation will moreover be found in the figures given in my second note.

But even granting for a moment the absurd supposition that the oleic, palmitic, and stearic acids I mentioned in my first note had not been washed free from acetic anhydride, it is easy to show that the values given by me cannot be explained in this way by Benedikt. From the saponification values I found and from the theoretical saponification values of acetic anhydride, of oleic, palmitic, and stearic acids\* it can be calculated according to the three following equations, how large the percentage of any admixed acetic anhydride might have been.

$$\text{For oleic acid.} \dots 1,100 \frac{x}{100} + 198 \cdot 9 \frac{100-x}{100} = 242, x = 4 \cdot 78 \text{ per cent.}$$

$$\text{For palmitic acid.} \dots 1,100 \frac{y}{100} + 219 \cdot 1 \frac{100-y}{100} = 226 \cdot 13, y = 0 \cdot 79 \text{ per cent.}$$

$$\text{For stearic acid.} \dots 1,100 \frac{z}{100} + 197 \cdot 5 \frac{100-z}{100} = 221 \cdot 18, z = 2 \cdot 62 \text{ per cent.}$$

Assuming, further, that these quantities of acetic anhydride have been left in the "acetylated" products, which were, according to Benedikt, nothing else but the free oleic, palmitic, and stearic acids, and making in addition the (absurd) assumption that any such acetic anhydride has not been hydrolysed, while the "acid value" was being determined in alcoholic solution, we may calculate what ought to have been the "acid values" of such mixtures of oleic, palmitic, and stearic acids with acetic anhydride. We should find—

$$\text{For oleic acid } (100 - 4 \cdot 78) \times 198 \cdot 9 = 189 \cdot 39.$$

$$\text{For palmitic acid } (100 - 0 \cdot 79) \times 219 \cdot 1 = 217 \cdot 37.$$

$$\text{For stearic acid } (100 - 2 \cdot 62) \times 197 \cdot 5 = 192 \cdot 32.$$

While the corresponding figures actually found by me were given as 116·5; 143·3; 138·39.

To remove every doubt I boiled 10 grms. of the oleic anhydride, which had been analysed, four more times with water, 30 minutes each time. 1·1472 grms. of this oleic anhydride shaken up with 25 cc. water, took 0·3 cc. one-tenth normal potash, when it became pink. The wash-waters were slightly acid and required (1) 12·8 cc.; (2) 4·0 cc.; (3) 2·36 cc.; (4) 1·12 cc. decinormal potash; but the wash-water from 10 grms. of the oleic acid, from which I had started, took, treated twice in the same way, 2·9 cc. and 0·8 cc. decinormal potash. I will make another concession, although an inadmissible one, namely, that the acid found in the wash-waters was hydrolysed acetic anhydride which had been retained in the oleic anhydride. I used altogether 12·8 + 4 + 2·36 + 1·12 cc. = 20·28 cc. decinormal caustic potash equal to 0·1138 gm. KOH which

\* For the acids the saponification values are of course identical with their acid values, 198·9, 219·1, 197·5.



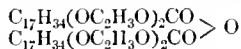
corresponds to 0.1217 grm. acetic acid or 0.1034 grm. acetic anhydride, or in other words the oleic anhydride *might* have retained 1.034 per cent. acetic anhydride. Previously I had granted for an equally inadmissible concession 4.78 per cent. anhydride.

The crucial experiment with a mixture of oleic anhydride and acetic anhydride was to give a final answer. To 1.8225 grm. oleic anhydride were added 0.1342 grm. acetic anhydride and this mixture of 93.15 per cent. oleic anhydride and 6.85 per cent. acetic anhydride was shaken up with 25 cc. water, and titrated with one-tenth normal potash. When 27.3 cc. had been added the pink colour appeared, while a perfect emulsion had been formed. The colour disappeared very soon and it took 4.8 cc. more to restore the pink tint; this disappeared very soon again and the mixture took another 1.2 cc. decinormal potash, when the pink colour stayed for about 20 minutes. The above 0.1342 grm. acetic anhydride would require theoretically for complete hydrolysis 38 cc. one-tenth normal KOH, while 33.3 cc. had been used; after 20 minutes 1.3 cc.; after two hours, 6.6 cc.; and after another hour, 5.2 cc. more were required to show an alkaline reaction.

Benedikt adduces as a proof for the correctness of his explanation and observation all the determinations of "acetyl values" that have been carried out by himself and by Hazura in the latter's researches on hydroxylated fatty acids. Hazura had found in all cases "acid values" for the "acetylated" fatty acids, which approximatively agreed with those required by theory. According to my views these "acid values" have only been found because the "acetylated" substances, which were the anhydrides of the acetylated substances, were partly hydrolysed on being titrated in *alcoholic* solution by means of half-normal potash, and this hydrolysis ceased when an equilibrium was established; therefore these figures prove nothing whatever.

In order to give further experimental proof I prepared dihydroxystearic acid from pure oleic acid by oxidising it by means of potash permanganate in alkaline solution. The dihydroxystearic acid was repeatedly recrystallised from alcohol; melting point, determination of the molecular weight, and elementary analysis proved the purity of the substance. 30 grms. of this acid were boiled with 25 grms. acetic anhydride, the mixture was poured into boiling water, and the separated oily layer repeatedly boiled and washed with water until a few cc. of the wash-water had no action on a sensitive litmus solution. The oil was then freed from the last traces of water and filtered in a drying oven. This substance was to be, according to my views, diacetyl-hydroxystearic anhydride, containing perhaps a small quantity of the corresponding acid, which might have been formed by slight hydrolysis of the anhydride.

1.5813 grms. of the substance required for complete saponification, 22.10 cc. of an alcoholic half-normal potash solution corresponding to a saponification value of 392. As the formula—



requires theoretically 430, while the free acid—



requires 420, this result cannot give any decision as to the question at issue, besides showing that either of these two substances had been formed. The substance was oily, viscous, and did not crystallise, so that a purification had not been attempted. But the following two experiments give a decisive answer:—

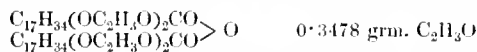
I.—1.3272 grms. were shaken up with 25 cc. water and after adding phenolphthalein titrated with decinormal potash. When 0.2 cc. had been added, the mixture became pink; this colour disappeared after one minute, and after the following times, 2, 2, 1, 3, 6, 9, 16, 60 minutes, the following quantities of decinormal potash added were used up 0.4 cc.; 1.1 cc.; 1.1 cc.; 1.1; 1.0; 1.0; 1.0; 1.0.

II.—(a.) 3.9557 grms. of the substance were dissolved in absolute alcohol and titrated with half-normal potash in a way exactly corresponding to that used by Benedikt while determining the "acid value" of the "acetylated" hydroxylated fatty acids. 13.35 cc. half-normal potash were used, yielding an "acid value" of 94.66.

(b.) 1.2858 grms. titrated in the same way required 4.4 cc. half-normal potash corresponding to an "acid value" of 95.9.

An acid of the formula  $C_{17}H_{34}(OC_2H_3O)_2.COOH$  would theoretically require 140. If we were to draw conclusions according to Benedikt we should find from the proportion of the saponification value to the acid value 392 : 95.2 or about 4 : 1 that dihydroxystearic acid contains besides the carboxyl group *three* hydroxyl groups, which is of course absurd. This proves clearly that the "acid values" in this case and such like have no quantitative meaning whatever, indicating only that hydrolysis has taken place.

Benedikt's principle requires some modification to be of use in the analysis of fats. The hydroxylated fatty acids on boiling with acetic anhydride will certainly become acetylated, but at the same time the acetylated acids will be transformed into their anhydrides. It will, therefore, only be safe to infer the presence of hydroxylated fatty acids in a fat if the acid value of the original fatty acids and the saponification value of the "acetylated" fatty acids—*i.e.*, the anhydrides—show a difference large enough to exclude any error naturally adhering to this method. If the presence of hydroxylated acid be proved, the real "acetyl value" will best be found by actually estimating the amount of acetic acid (resp. "acetyl") which has been taken up by the fatty acids. I propose for this purpose a method closely resembling that of Reichert, namely, to distil over the acetic acid formed by complete saponification and estimate it by titration with decinormal potash. The determination of the acetic acid can be conveniently combined with that of the saponification value; care has only to be taken that sulphuric acid be used for titrating back the excess of alcoholic potash and for acidulating the solution of soap. Before adding the sulphuric acid to set the fatty acids free, it will be best to evaporate the alcohol in order to prevent later on formation of acetic ether. In this way I estimated the "acetyl" split off by complete saponification of the above-mentioned 1.5813 grm. In order to neutralise the filtrate, 76.6 cc.  $\frac{1}{10}$  normal potash were used corresponding to 0.3165 grm. acetyl ( $C_2H_3O$ ), while theory requires for 1.5813 grm.—

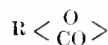


This method has the additional advantage of enabling us to prove the acetic acid "in substance."

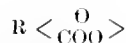
The unsaturated fatty acids of the Sawarri fat (see above) showed an acid value of 188.5; after having been boiled with acetic anhydride the anhydrides gave a saponification value of 230.4, proving the presence of hydroxylated fatty acids. I determined the amount of acetic acid by distillation and found in the distillate a quantity of it corresponding to 8.4 cc. decinormal potash for 3.364 grms., that is 2.5 cc. for one gramme. If we express, as I propose to do, the acetyl value by the number of milligrammes of caustic potash that will be required to saturate the acetic acid which one gramme of the acetylated fatty acid—*i.e.* of the acetylated anhydride—will split off on being saponified, the acetyl value will be in this case  $0.25 \times 56.1 = 14.03$ .

The action of acetic anhydride on organic acids with the production of anhydrides seems to be a general one. Anschütz (Liebig's Annalen, 226, 12) has already studied the inter-action of acetic anhydride with succinic, camphoric, benzoic, *o*-phthalic, and diphenic acids, and found in each case that the acids were partly transformed into their anhydrides. I found the same holds good of sebacic acid.

In conclusion, I may point out that, by adopting my views, Benedikt will easily get over a difficulty which the explanation of the fact that the "acid values" of hydroxylated fatty acids are found lower than the theory requires, seems to offer him.\* He thinks that a small percentage of inner anhydrides of the formula—

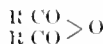


(in consequence of a clerical error the paper gives the formula—



\* Chem. Zeit. 1890, No. 51.

might have been formed, but excludes altogether the possibility of the formation of the normal anhydrides—



My explanation by no means precludes the simultaneous formation of inner anhydrides, but it does not postulate it.

## Obituary.

PROFESSOR THOMAS CARNELLEY, D.Sc., F.C.S.,  
A MEMBER OF THE SOCIETY OF CHEMICAL INDUSTRY.

On the 27th of August an unexpected blow was sustained by the scientific world through the sudden death of Dr. Thomas Carnelley. Only in his thirty-eighth year, full of hope and promise, and with bright expectations of a further harvest of the fruits of research and literary work, he himself, the worker and investigator, was quickly removed from the field.

Educated at the Owens College, Manchester, where he became Demonstrator and Assistant Lecturer in Chemistry, he thence proceeded as Professor of Chemistry to Firth College, Sheffield, and later on to University College, Dundee.

On the death of Professor Brazier he was, two years ago, appointed to the Chair of Chemistry at the University of Aberdeen.

His most striking investigations were those which embodied the further development of the periodic system of Newlands and Mendeléeff. Up to the time of his decease his mind was concentrated upon a great work on the chemical and physical constants in which he was tracing out relations and uniformities not previously detected. With a mind singularly apt and powerful in tracing scientific analogies, great things might have been further anticipated of him had he been spared. Carnelley's work and investigations on sanitary chemistry were of great interest, and his last research published in conjunction with Mr. Frew on "The Antiseptic Powers of Isomeric Organic Compounds," was of considerable value, not only viewed from the standpoint of pure, but also equally from that of applied chemistry. Among the more useful of his researches may be enumerated the following: "The Results of Passing the Mixed Vapours of Bisulphide of Carbon and Alcohol through a Red-hot Tube," "The Action of Water and Various Saline Solutions on Copper," "On High Melting Points, with special reference to those of Metallic Salts," "Colorimetric Methods of determining small Quantities of Iron and Copper," "Tolyl Phenyl, a New Hydrocarbon," "The Action of Heat on Mercuric Chloride under Low Pressures," "A New Form of Pyrometer," "Oxidation of Di-tolyl," "Brominated Derivatives of Diphenyl, Tollyl-benzene, and Ditollyl," "Amidodiphenylsulphonic Acid and Azo-dyes from Diphenyl," "The Derivatives of Tollyl-benzene," "The Solubility of Isomeric Organic Compounds, and of Mixtures of Sodium and Potassium Nitrates, and the Relation of Solubility to Fusibility," "The Dehydration of Metallic Hydroxides by Heat, with special reference to the Polymerisation of the Oxides and to the Periodic Law."

Let any chemist or chemical technologist read these researches, which form a selection of those recorded in the Journal of the Chemical Society during the last 12 years, and he must come to the conclusion so aptly expressed by a contemporary, that "Thomas Carnelley was a man whom the scientific world could ill spare."

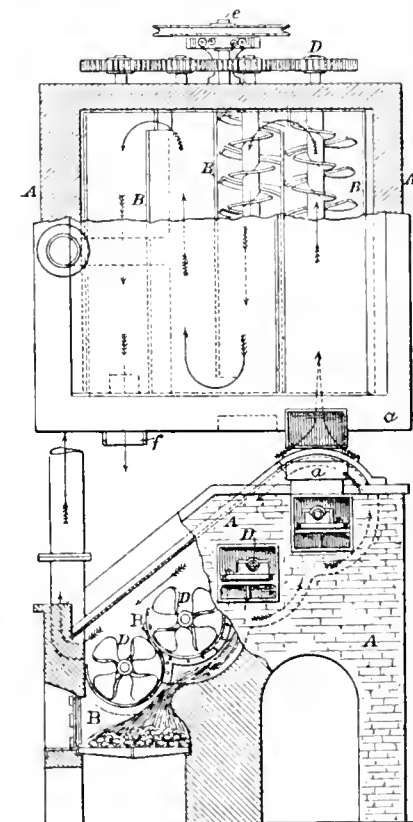
## Journal and Patent\* Literature.

### I.—GENERAL PLANT, APPARATUS, AND MACHINERY.

#### PATENTS.

*Improved Means for Drying or Desiccating Substances or Materials.* P. Cornish, Leyton. Eng. Pat. 6102, April 9, 1889. 8d.

A is a brickwork furnace in which the cylindrical troughs B are set, slightly inclined; each is provided with a "screw creeper," D, of the usual construction, driven by the pulley e and gearing, revolving alternately in opposite directions. The cylinders are heated in the manner shown.



DESICCATION APPARATUS.

The material to be dried is introduced through the hopper *a*, and is passed through the series of cylinders, falling from one to the other, and is finally discharged at *f*, dried by the heated air of the furnace passing both under and over the drying cylinders as shown.—C. C. H.

\* Any of these specifications may be obtained by post, by remitting the cost price, plus postage, to Mr. H. Reader Lack, Comptroller of the Patent Office, Southampton Buildings, Chancery Lane, London, W.C. The amount of postage may be calculated as follows:—

If the price does not exceed 8d. ....	1d.
Above 8d., and not exceeding 1s. 6d. ....	1d.
" 1s. 6d., " " 2s. 4d. ....	1½d.
" 2s. 4d., " " 3s. 4d. ....	2d.

*Improvements in Apparatus for delivering Measured Quantities of Liquid.* F. E. MacMahon, London. Eng. Pat. 13,208, August 21, 1889. 6d.

This invention is an improvement on Eng. Pat. 12,644 of 1888, in which the liquid to be measured was received into, and delivered from a cylinder, the piston of which was allowed to travel a certain distance, dependent on the quantity of liquid to be delivered. Here the apparatus consists of a measuring cylinder and a piston and cock so connected therewith, that in one position of the plug it allows the measuring vessel to fill and the piston to rise; and in another position it allows the measuring vessel to empty by a spout, and the piston to fall, the travel of the piston being regulated by controlling stops according to the amount to be delivered.—E. S.

*Improvements in Apparatus for Softening, Purifying, and Filtering Water.* J. Wright, London. Eng. Pat. 1946, February 5, 1890. 8d.

As upright rectangular tank of considerable height is divided into three parts by vertical partitions. At the top of the whole is placed a regulating cistern dividing the incoming water into two portions. A portion is admitted into one of the sections above described, in which it is mixed with lime, whilst another portion—the bulk of the water treated—is admitted into the mixing division, into which also the lime water and a solution of caustic soda from a separate tank enters. Passing down the mixing portion, the treated water enters the bottom of the remaining division of the apparatus, through which it ascends. The solid matter is arrested on depositing shelves and the partially clarified water, after passing through a tubular filtering apparatus, is delivered at the top of the tank. The incoming water effects the agitation of the lime water as well as the mixture of the reagents with the unpurified water, by means of agitating chains worked by means of small water-wheels.—C. C. H.

*Apparatus for Condensing, Heating or Cooling Gaseous or Liquid Substances.* J. Klein, Frankenthal, Germany. Eng. Pat. 6136, April 22, 1890. 8d.

This apparatus is adapted both to condense and cool gaseous or liquid substances, the object being to increase as much as possible the area of the cold or heat-producing surface (as the case may be), and also to allow of the inner part of the apparatus being readily cleaned.

The apparatus is arranged within a tank, the side walls of which form the frame supporting the cast-iron plates of which the apparatus is constructed. These plates have projecting edges, and when fitted tightly together, form a series of hollow spaces, and are securely connected with the head wall of the tank by connecting rods. When thus fitted together, the whole is surrounded by the water, for the purpose of more thoroughly effecting the condensation of the steam, or the more perfect absorption of the heat of any hot gases or fluids that may be conveyed to the apparatus.

To secure a higher degree of vacuum in the steam chambers, a fine spray, issuing from a valve placed at the end of the apparatus in one of the terminal chambers, and operated externally, is further employed.—E. S.

*Improvements in Filters.* W. P. Thompson, Liverpool. From J. A. Bowden, Detroit, U.S.A. Eng. Pat. 8568, June 3, 1890. 8d.

THE filter can be constructed either in an upright or a horizontal form. In the upright form it is composed of two compartments, one above the other, partially filled with sand or other filtering medium. Upon the bottom of each compartment is a conical-shaped diaphragm, supported clear of the bottom by a perforated screen. The water to be filtered flows down through the sand, the perforated

screen, and beneath the inclined diaphragm to the lower filter bed, and escapes by an outlet pipe. To clean the beds, the water is admitted from below, in each compartment, and causes the sand to rise with it. The sand in the centre flows down the inclined diaphragm to supply the place of that removed, and thus a complete circulation is established, and the bed thoroughly washed. The drawings attached to the specification show both forms of filter. There are six claims.—E. S.

*Apparatus for Automatically Drawing off Viscous Substances.* L. H. A. W. Sack, Berlin, Germany. Eng. Pat. 9554, June 20, 1890. 6d.

This invention is intended to effect the automatic removal from vessels of viscous substances, as syrups, honey, mustard, thick oil colours, and lubricating substances, whilst at the same time the residuum in the vessel is protected from being covered with dust or a film. On the surface of the viscous fluid in the vessel is a plate which bears down upon it under the action of a spiral spring enclosed in a telescopic tube, the upper end of the tube and spring pressing firmly against the cover, which is kept in position by any suitable means. The fluid is drawn off through a plug at the bottom of the vessel.

There are four claims.—E. S.

*An Improved Method and Apparatus for Drying Aqueous Matters.* R. Haddan, London. From E. Passburg, Breslau, Germany. Eng. Pat. 9941, June 26, 1890. 6d.

This invention is an improvement on Eng. Pat. 6326 of 1884, and consists in drying aqueous materials *in vacuo* by means of heat reservoirs in juxtaposition with the matters to be dried, by which means the heat may be transferred to the reservoirs whilst the matters are in juxtaposition therewith, and the temperature of the reservoirs can be maintained for an indefinite time before they are allowed to cool down under the influence of conduction and radiation. An air-tight chamber, connected by a pipe with a condenser and air-pump, has inside it steam compartments formed of metal frames with sheets welded to them on either side. Each side is perforated, and the two holes are connected respectively with the inlet and outlet steam pipes. The apparatus is fed through a door, and metallic trays carrying the substance to be dried are placed on the steam chambers, outside which a vacuum is established. The door is then closed, and the air-pump is worked to remove the vapour from the apparatus; as soon as the material begins to dry steam is shut off to avoid overheating. A gauge and thermometer are attached.—E. S.

## II.—FUEL, GAS, AND LIGHT.

*The Loomis Process of making Gas for Fuel.* R. N. Oakman, jun. The Engineer, August 8, 1890, 107.

By this process three kinds of gas, viz., producer-, water- and carburetted water-gas may be produced for lighting purposes. The paper deals with the two former. The fuel used is a bituminous coal, the slack working the best. The producer-gas passes from the generator through a 20-ft. multitubular boiler, wherein its heat is utilised in producing steam for the water-gas. The alternating operations are each carried on for the same length of time—from 4 to 6 minutes. This is not always the case, however, as the generator is sometimes worked on producer-gas alone, in which case steam is admitted at various points during this operation. For the maximum production of water-gas from a ton of coal more than half a ton is consumed in making producer-gas. The water-gas made by this process is said to possess a strong

odour, due to the presence of about 3 per cent. of olefiant gas, which, however, is omitted from the following two analyses:—

	Water-Gas.	Producer-Gas.
	Per Cent.	Per Cent.
Carbonic acid.....	4.5	7.4
Carbonic oxide.....	36.6	22.5
Hydrogen .....	57.4	13.2
Nitrogen .....	1.5	56.9

The author recommends the production and application of the two gases jointly. The economy is pointed out, but Lowthian Bell states that the use of gaseous fuel represents 30 per cent. loss, and it is only because with gas fuel regeneration is possible that it becomes economical. Solid fuel under proper conditions is the most economical of all.—A. W.

*Testing the Relative Value of Different Coals.* W. Kent.  
Eng. and Min. J. 50, 76—77.

THIS paper contains a description, with full results, of some practical boiler tests of some samples of coal. The author points out the mistake in supposing that the results obtained by testing coals in a boiler under the same conditions represent the relative values of the same coals if they are to be used for a different kind of boiler or for other purposes. Moreover, the figures obtained in tests with the same boiler under the same conditions and with the same fireman, &c. are likely to lead to erroneous conclusions unless they are corrected by the "efficiency of the boiler" as shown during those tests. The first tests of two samples of coal, A and C, gave the water evaporated at 100° C. per pound of coal as 9.311 and 9.302 lb., respectively—a ratio of 100:99.9. The evaporation per pound of combustible, however, was 10.167 and 10.311 lb., or a ratio of 98.60:100. Evidently the conditions, although identical in each case, were more favourable to sample C than A. Consequently if the conditions were such that the efficiency of A was equal to that of C, then the corrected value of C would be to that of A as only 98.5:100 obtained as follows—

$$\therefore 100:98.6::99.9:98.50.$$

A second set of tests was made with a view to obtain increased efficiency by reducing the grate surface to such an extent that the ratio of heating to grate surface was raised from 39.8 to 46.9:1. The actual evaporation in these tests was for A, 9.419 lb., and for C, 9.495 lb., or an apparent relative value of 100:100.81, thus in this instance showing coal C to be superior to coal A, and reversing the result of the first test. The efficiency of the boiler was increased in each case to 10.214 and 10.466 respectively, or a ratio of 97.59:100. Making the correction therefore as before—

$$\therefore 100:97.59::100.81:98.36$$

the relative value of C to A becomes 98.36:100, which agrees well with the corrected figure in the first tests, 98.50.

The author does not recommend this correction as always giving the practical fuel value of a coal, because in many cases increased efficiency could only be obtained at the expense of the capacity of the boiler. He suggests, however, the following rule:—"If in a comparative test of two coals of approximately similar chemical constitution, the apparent relative fuel value as shown by the boiler test differs less than the difference of boiler efficiency in the two tests, then the apparent relative values should be corrected for the difference in boiler efficiency." He is not aware of other experimenters having made use of these corrections, but believes that within the above limits they should be applied in tests made for commercial purposes.—A. W.

*On the Manufacture and Utility of Naphtha Candles.*  
Dingl. Polyt. J. 276, 563—565.

THE so-called naphtha candles were recently shown at an exhibition in St. Petersburg, and although at one time great hopes were entertained of them, it was found that they were not suitable for use.

They consist chiefly of a mixture of ammonia-soap, stearic acid, and mineral oil, this mixture becoming a solid mass, which can be moulded into candles. Such candles, however, smell unpleasantly, as the mineral oil gradually evaporates away. The candles exhibited were made from a mixture composed of 65 per cent. of stearic acid, 30 per cent. of mineral oil, 5 per cent. of water, and about 0.8 per cent. of ammonia. An analysis, made about a month after the candles were manufactured, showed from 10—12 per cent. of mineral oil, 82—85 per cent. of stearic acid, 4.5—5 per cent. of water and 0.6—0.75 per cent. of ammonia. To diminish the evaporation of the mineral oil the candles were coated with various kinds of varnishes, but this, besides being an expensive, was not a satisfactory device. The appearance of the candles was also bad, as they were yellow coloured with white spots. They burned with an unsteady sooty flame and satisfactory photometric observations could not be obtained.—W. M.

PATENTS.

*Improvements in the Manufacture of Gas for Illuminating and other Purposes.* M. H. Strong, Brooklyn, U.S.A.  
Eng. Pat. 2249, June 9, 1877. (Second Edition.) 8d.

A CENTRAL narrow vertical chamber provided with a hopper and feeding apparatus at the top, and with a fire grate and closed ash pit at the bottom, communicates over a baffle-wall with a wide chamber filled with loose brickwork, the lowest part of which is connected with the chimney. A shallow chamber likewise provided with a hopper may optionally be placed on the other side of the central chamber and connected with it. The whole mass is surrounded with non-conducting material and enclosed in a metal casing. The central chamber is heated to a sufficient temperature by an internal fire which is supplied with air by a jet exhauster in the chimney and by a properly regulated air passage in the upper part of the chambers; the air supply is then cut off and the chimney closed by a valve; steam is then admitted into the chimney and becomes superheated in the regenerative chamber. Finely ground coal or liquid hydrocarbon is then introduced in a fine shower into the central chamber, and is volatilised and acted upon by the steam, the resulting gases are further "carburised" by passing through the bed of ignited fuel on the grate, and are drawn off by a pipe opening into the ashpit. The gases may be enriched in illuminating constituents by passing them up again through bituminous coal supplied into the third chamber before withdrawal.—S. B. A. A.

*A New and Improved Carburetted Apparatus for Enriching or Generating Illuminating Gas from Volatile Liquid Hydrocarbons.* H. Howell and L. Smith, London.  
Eng. Pat. 14,686, September 18, 1889. 8d.

THE apparatus is intended for automatically impregnating coal gas or air with hydrocarbon vapour so that the mixture may burn without smoke or smell and without depositing the enriching vapours under normal variations of temperature. It consists of a vertical cylinder divided by a horizontal partition into two compartments, the upper and smaller forming a reservoir for the hydrocarbon and the lower constituting the enriching chamber; a central vent pipe passes through the partition and reaches almost to the top and bottom of the apparatus, and a syphon having its mouth opening near the bottom of the upper compartment has its longer limb either turned up to form a trap or projecting into a small shallow dish immediately under the partition. The enriching chamber contains a series of alternately convex and concave perforated partitions covered with some absorbent fabric except for portions of their surface at the circumference and centre respectively. A shallow

annular pan is fixed near the bottom, the intervening space being occupied by incomplete cylindrical partitions covered with the absorbent fabric. When the hydrocarbon is charged into the reservoir it is conveyed by the syphon into the shallow dish which overflows and saturates the partitions, the drainage from which accumulates in the lower pan which finally overflows, forming a well on the floor of the chamber (the "vent well") which soon seals the vent pipe. The syphon then ceases to act and the reservoir may be filled. Gas or air is admitted above the vent well, passes around the cylindrical partitions, through the central space, round and between the convex and concave partitions, and is drawn off from the upper end of the enriching chamber. As soon as the liquid in the vent well has evaporated sufficiently to unseal the bottom of the vent pipe the syphon again acts and the partitions are drenched with the liquid from the reservoir. A section and sectional plan of the apparatus are shown in the specification.—S. B. A. A.

*Improvements in Internal Combustion Regenerative Thermomotors some of which said Improvements are Applicable to Gas and Hot Air Engines.* J. Hargreaves, Widnes. Eng. Pat. 14,789, September 19, 1889. 8d.

The improvements consist in the first place in the use of two slide valves to regulate the entrance of air into the air pump, its passage into the cooler end of the regenerator, and the exit of the spent gases from the working cylinder after passing through the regenerator, the slide valves being actuated either by two eccentrics or by one eccentric with rocking levers operating at different angles. Secondly it is claimed that the seats of slide valves may be kept true so as not to require frequent re-surfacing, by coupling the slide stems directly to the eccentric straps or levers without pivoting, the slides acquiring in this way an oscillating lateral motion which causes them to wear more uniformly. Lastly, to prevent the accumulation of tarry matter in the working cylinder of thermo-motors when liquid fuel is used, a groove is formed around the working piston at the point which comes opposite the scraper when the piston is at the inner portion of its stroke and the scraper is made with diagonal passages along its face so as to allow any accumulated matter to pass through. The details are fully illustrated in the specification. (Compare this Journal, 1888, 30.)

—S. B. A. A.

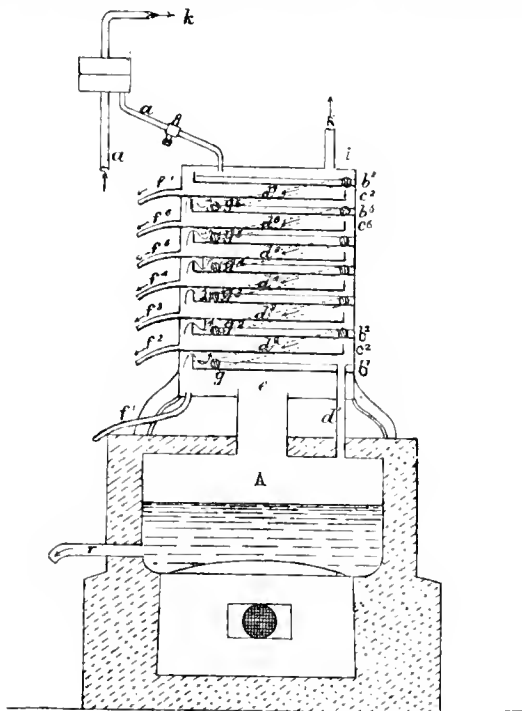
*Improvements in the Manufacture of Illuminating Gas.* S. Pitt, Sutton. From E. J. Jerzmanowski, New York, U.S.A. Eng. Pat. 9733, June 23, 1890. 6d.

The difficulty generally experienced in the utilisation of low-grade illuminating oils such as crude oil, for enriching gas by the ordinary process of conducting the gas and the illuminant together through heated "fixing retorts," is that the illuminant comes in contact with surfaces continually cooled by the incoming gas and is therefore less thoroughly vaporised and more likely to deposit. In the present invention the oil is conveyed by a pipe which enters the retort from the discharging end and passes along two-thirds of the length of the retort before allowing the oil to escape into the interior. The heated and vaporised oil then turns back upon its course together with the gas, and passes into the delivery pipes. The connections are such that the oil may be heated before entering the retort pipe by passing through pipes in the brickwork around the retorts or through suitable superheaters. An arrangement of steam pipes is shown for chilling the oil supply pipes in cases of over-heating and for keeping the pipes clear.—S. B. A. A.

### III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

*Apparatus for Fractional Distillation of Crude Naphtha and Naphtha Residues.* Schuchow and Intschik. Dingl. Polyt. J. 276, 465—466.

The apparatus consists of a boiler A, heated from below, and of a vertical series of pans above, the current of vapour and gases rising from the boiler A, through the column of pans in a zig-zag manner, meeting all along its route a flow of liquid crude naphtha entering the apparatus from above through the pipe *a*. This liquid, being previously used in a condenser as a cooling material, is already warmed. Entering through the pipe *i* it falls on the uppermost pan, then by the pipe *d'* to the pan next below, &c. It naturally meets



DISTILLATION OF CRUDE NAPHTHA.

at the upper end of the apparatus the lightest parts of the naphtha vapour from the boiler A, and being itself here the coldest condenses them. The lower down the liquid flows, the higher becomes the temperature of the apparatus, the heavier are the gases it meets, and gradually yields its volatile elements. Thus only the heaviest part of this liquid naphtha, or in other words only the residues reach the boiler A by the pipe *d'*. On the other hand, this very liquid flow serves for fractionating off the vapour rising from A. Entering the space *e*, the latter comes into contact with the bottom of pan *b*, cooled by its liquid contents, condenses and falls on the tray, and flows out through *f' f*. Uncondensed vapours are forced to dive under partition *g*, into the mass of liquid naphtha, thus heating it, yielding to it some heavy vapours and entering the space between *b<sub>1</sub>*, *d<sub>2</sub>*, and *b<sub>2</sub>*; here again it comes in contact with the cooler bottom of pan *b<sub>2</sub>*, condenses and falls on the tray *d<sub>2</sub>* and flows out through *f<sub>2</sub>*, &c. This process may be continued according to the number of pans. The lightest gases which could not be condensed in the apparatus are carried away through the pipe *i* and condensed separately. The boiler is heated by pulverised residues.

The principal results of using this apparatus in practice are as follows: the average difference of temperature between each two neighbouring pans is 30° C., which makes for

seven pans a total difference of  $210^{\circ}\text{C}$ . at the upper and the lower ends of the apparatus. Thus it may contain at the same time the lightest and the heaviest distillation products. The volume of the daily output is 27 times the capacity of the apparatus, which makes in one case 105,000 kilos., the proportion of the naphtha residues being only 40 per cent., while the best apparatus in common use leave not less than 60 per cent. of residues.—N. W. T.

*On the Condensation of Benzene Vapour and of Acetylene under the Influence of the Glow-Discharge.* P. Schutzenberger. *Compt. Rend.* **110**, 889—892.

BENZENE vapour was introduced into the annular space between two concentric tubes by means of a semi-capillary tube, the end of which was then sealed with the blow-pipe. A glow discharge was then caused to pass in the enclosed space by means of sulphuric acid electrodes (as in the Berthelot ozone apparatus). The liquid benzene in the bottom of the tube gradually evaporated and condensed on the active surfaces in the form of a layer of a transparent light yellow substance. Although the author believes that no oxygen was present in the tube at the beginning of the experiment, the substance contained oxygen. The author concludes that *glass under these conditions is permeable*. The time during which the experiment lasted was variable, the inner tube often breaking; no tube withstands the passage of the current for more than six hours when the annular space is filled with benzene vapour, or for more than a few minutes when the space is entirely exhausted.

The amount of oxygen absorbed varied from 0.7 per cent. to 7 per cent. of the product formed (12 analyses). In one case the deposit on the internal and external surfaces was analysed separately and gave the following results:—Deposit on internal surface, C = 90.54 per cent., H = 7.30 per cent., O = 2.10 per cent.; deposit on external surface, C = 87.14 per cent., H = 7.48 per cent., O = 7.0 per cent.

The author regards the following experiment on acetylene as conclusive. The annular space was not sealed in this case, but closed by a surface of mercury. A volume of 250 cc. of the gas was gradually introduced and condensed by means of the glow discharge; the residue of acetylene was then measured, and pure dry hydrogen was introduced and the glow discharge continued for 12 hours. At the beginning of this period an absorption took place corresponding to the volume of the acetylene in the apparatus, after which no further absorption was observed.

The condensed product contained C = 75.0 per cent., H = 8.9 per cent., O = 16.1 per cent. This corresponds to an absorption of 2 centigrammes of water from the exterior. This product does not absorb oxygen from the air. The appearance and weight of the glass are not sensibly altered. After the combustion of the substance, a very slight superficial deposit of alkaline salt on the glass is observed, not weighing more than from 1 to 2 milligrammes. —P. J. H.

*The Action of Litharge on Toluene: Production of Benzene.* C. Vincent. *Compt. Rend.* **110**, 907—908.

BENK and Van Dorp, and after them Lorenz (Ber. **7**, 1096) have shown that stilbene, diphenyl, anthracene, and a liquid hydrocarbon are produced by the action of litharge on toluene at a dull red heat. The author has investigated the reaction at different temperatures. The apparatus consisted of a long iron tube heated by a row of regulated gas burners, connected to a condenser and a wash-bottle containing baryta water. The iron tube was filled with litharge in scales, and toluene was introduced drop by drop into the end of the tube until all the litharge was reduced.

The author obtained the following results—

(1.) At a temperature a little below  $335^{\circ}$  (the melting point of lead) a simple oxidation of the side chain took place, giving rise to benzene and carbonic acid. The oily liquid which distilled over consisted only of the excess of unattacked toluene mixed with 10 per cent. of its weight of benzene.

(2.) At a higher temperature, less benzene is formed, the chief product being stilbene, mixed with other hydrocarbons. The principal reaction is—



(3.) At a low red heat, besides the above compounds, their condensation products are also formed—diphenyl, phenanthrene, anthracene, &c.

(4.) The production of diphenyl is not due to the presence of benzene as an impurity in the toluene, but to the benzene formed by the reaction.—P. J. H.

# PATENT.

*A New or Improved Process for the Purification of Mineral Oils and for Producing Alcohol.* W. P. Thompson, Liverpool. From P. Marix, Paris, France, and N. Notkin, Moscow, Russia. Eng. Pat. 13,653, August 29, 1889. 6d.

NAPHTHA and mineral oils are usually purified by means of strong sulphuric acid, whereby a "black acid pitch" is obtained. The inventor proposes to make use of this "pitch" by mixing with it various vegetable matters, such as wood, straw, paper, &c., which yield glucose when acted on by sulphuric acid. These vegetable matters are either introduced into the oil before treatment with acid or incorporated with the "pitch" thus produced. The mixture is then treated with boiling water for some time, the solution neutralised, filtered, concentrated and fermented. The inventor also proposes to use the pitch, as usually obtained, for absorbing ethylene from illuminating gas, thus forming ethyl sulphuric acid, which when distilled with water yields alcohol and sulphuric acid.—A. L. S.

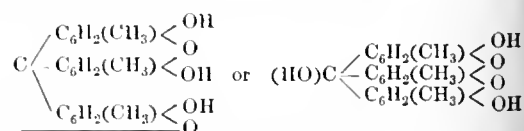
## IV.—COLOURING MATTERS AND DYES.

*The so-called Trisulphonic Acid.* C. Loring Jackson and G. T. Hartshorn. Ber. **23**, 2143.

THE authors formerly reported the discovery by them of a method of preparing aniline trisulphonic acid (this Journal, 1888, 669). They now find to their surprise and sorrow that what they obtained was simply sulphanilic acid, and that consequently aniline trisulphonic acid has yet to be discovered.

*Homofluoresceïn.* E. Grimaux. *Compt. Rend.* **110**, 1074—1076.

ON heating a mixture of oreinol, chloroform, and soda, Schwartz, in 1880, obtained a crystalline compound to which he assigned the formula  $\text{C}_{23}\text{H}_{15}\text{O}_5$ , regarding it as trimethylfluoresceïn, a homologue of fluoresceïn, which he named homofluoresceïn. Schwartz assumed that the body was formed by the condensation of 1 mol. of methylphthalic acid with 2 mols. of oreinol, the methylphthalic acid, according to his view, being produced by the replacement of two OH-groups in oreinol by two COOH-groups—a view which seems to the author difficult of acceptance, as he considers that the reaction would agree better with the formation of an aurin of the empirical formula  $\text{C}_{22}\text{H}_{15}\text{O}_5$  and the constitution—



As Nencki in 1882, by acting on oreinol with formic acid and zinc chloride, prepared an aurin of the above constitution, the author concluded that Schwartz's homofluoresceïn and this body must be identical, and made



experiments to prove that such was the case. Schwartz's compound was prepared by the published method, being obtained in the form of the soda-derivative, which was twice crystallised from acetic acid; red needles with green reflex were so obtained, containing acetic acid of crystallisation; on drying at 100° the compound lost 31.3 per cent. of acetic acid, Schwartz giving the loss at 31.4 to 31.7 per cent. Nencki's oreinol-aurin was crystallised in a similar way from acetic acid, needles of precisely the same appearance as Schwartz's compound being obtained; on drying at 100° they lost 32.2 per cent. of acetic acid. The two compounds were now closely examined, identical reactions being in all cases observed, for example, both compounds melt at 300°; heated in test tubes, both compounds at a high temperature decompose with separation of carbon and evolution of red fumes; both yield barium salts sparingly soluble in cold water; the alkaline solutions of both compounds are intensely fluorescent.

The compound obtained by Schwartz by nitrifying homofluorescein and acting on the nitro-derivative with ammonia, and to which that chemist gave the formula  $C_{22}H_{20}N_{10}O_{10}$ , assuming that it was a pentanitrodiazamidocompound, when referred to the aurin-type, will be seen to be a hexanitrodiamido-derivative of oreinol-aurin of the formula  $C_{22}H_{10}(NO_2)_6(NH_2)_2O_3 \cdot 11H_2O$ , Schwartz's analytical figures confirming this.

Using resorcinol in place of oreinol in Schwartz's experiment a red-brown solution is obtained, from which acids precipitate a yellow powder identical with the resaurin prepared by Nencki by heating together to 140° resorcinol, formic acid, and zinc chloride.

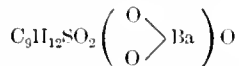
It is interesting to note that whilst the alkaline solutions of resorcinol-aurin are devoid of fluorescence, those of the corresponding oreinol-compound are extremely fluorescent, but conversely, whilst resorcinol-phthalein in alkaline solution is richly fluorescent, oreinol-phthalein, as stated by E. Fischer, is non-fluorescent.—E. B.

*On Amethyleamphophenolsulphone and its Tetra-nitro derivative: a Yellow Colouring Matter.* P. Cazeneuve. *Compt. Rend.* 110, 961—964.

IN a previous note (this Journal, 1890, 647; see also 1889, 815) a certain class of camphor derivatives called *campho-sulphophenols* was described. The first member of the series yields, on splitting off the methyl group, the body *amethyleamphophenolsulphone*. This body is also formed according to the equation—

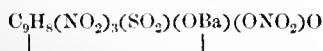


The substance crystallises in white scales, soluble in water and alcohol, insoluble in benzene, ether, chloroform, and carbon disulphide. It contains two hydroxyl groups, the one phenolic, the other corresponding to a secondary alcohol. Baryta water gives a precipitate corresponding to the formula—



A monacetic ester is obtained by heating to ebullition with acetic anhydride; the diacetic ester by boiling for an hour with acetic anhydride and sodium acetate. Heated with phenylhydrazine on the water-bath, a liquid compound (difficult to purify) is slowly formed. Hence it follows that the ketonic group of camphor still persists in this compound.

Added gradually to five parts of fuming nitric acid at a temperature of from 0° to 10° the body dissolves without any evolution of gas. A precipitate forms on addition of ice water which, on recrystallisation from alcohol, was found to be the tetranitro derivative,  $C_9H_{10}(NO_2)_4SO_2O_3$ , or more probably  $C_9H_9(NO_2)_3(SO_2)(OH)(ONO_2)O$ . This body is only slightly soluble in water, more soluble in alcohol. It melts at 87° and solidifies at 80°. It distils above 200°, but not without decomposition, and explodes when projected into a red hot crucible. It is a dibasic acid, and gives crystalline salts of an orange-yellow colour, all of them soluble, even those of the alkalis. The barium salt has the formula—



It is obtained in orange crystals containing two molecules of water; the water may be driven off at 100°, leaving a red anhydrous salt of the colour of chromic acid, which readily absorbs moisture from the air and again becomes orange yellow. The nitro body and all its salts dye wool and silk of a splendid yellow or orange colour without the use of a mordant. It is the first nitro colouring matter of the terpene series, of which the fundamental hydrocarbon can only be, according to the author, a hydride of propylbenzene.—P. J. H.

## PATENTS.

*A New Manufacture of Violet and Blue Azo Dyes.* H. J. Haddan, London. From the "Farbenfabriken vorm. F. Bayer and Co.," Elberfeld, Germany. Eng. Pat. 9510, August 10, 1885; amended March 27, 1890. 6d.

THE patentee confines himself to the use of the tetrazo compound of *ortho* tolidine in the production of the above colouring matters (see this Journal, 1886, 427).—O. H.

*Improvements in the Manufacture of Azo Dyes.* J. Y. Johnson, London. From the "Farbenfabriken vorm. F. Bayer and Co.," Elberfeld, Germany. Eng. Pat. 14,424, November 24, 1885; amended March 27, 1890. 6d.

THE patentee disclaims "the use of the Alpha-mono-sulpho acid and the Gamma-disulpho-acid of Beta-naphthylamine;" also the use of "benzyl ethers of the diamidophenols" as stated in the first claim, restricting himself to the "ethyl methyl or amyl ethers." Compare the abstract of original specification, this Journal, 1886, 428.—O. H.

*The Production of Paranitrobenzyl-sulpho Acid, and also Azo Colouring Matters from the Amidobenzylsulpho Acid corresponding to this Nitro-compound.* A. Bang, Leeds. From Dahl and Co., Barmen, Germany. Eng. Pat. 7588, May 7, 1889. Reprint. 4d.

THIS specification has already been abstracted (this Journal, 1889, 611). The amendment made by order of the Comptroller prior to the sealing of the patent is to the effect that the patentee is "well aware that it has been previously proposed to use sulpho acids of amines in the production of mixed azo colours."—T. A. L.

*The Preparation of New Sulphonic Acids suitable for the Manufacture of Colouring Matters.* The Clayton Aniline Co., Limited, and C. Dreyfus, Manchester. Eng. Pat. 10,934, July 6, 1889. 6d.

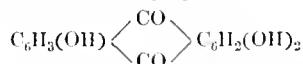
A METHOD for the production of sulphonic acids from phenyl- and tolyl- $\beta$ -naphthylamine. Zimmer (Ger. Pat. 45,940) describes the production of a new sulphonic acid of phenyl- $\beta$ -naphthylamine. This, according to the present specification, is a mixture of two monosulphonic acids which can be separated by the difference in solubility of their ammonium salts in water. The less soluble one called the A acid forms a sodium salt crystallising in needles free from water, which do not dissolve to fluorescent solutions in water or alcohol, whilst the more soluble one called the B acid forms a sodium salt crystallising in plates containing 3 molecules of water, soluble in water or alcohol to fluorescent solution, 100 kilos. of phenyl- $\beta$ -naphthylamine are dissolved in 400 kilos. of sulphuric acid monohydrate below 50° C., and allowed to stand two days at 15°—20°. The melt is then poured into 1,600 litres of water and boiled till the sulphonic acids separate out as a granular powder. The precipitate is filtered off, washed and suspended in 1,600 litres of water, sufficient ammonia being added to dissolve it. After filtering, the solution is allowed to cool, when the ammonium salt of the A acid separates out. A further quantity can be obtained by evaporating the liquor to one quarter its bulk. The sodium salt of the B acid is precipitated from the filtrate by the addition of



caustic soda or sodium chloride. The quantities of the two acids vary according to the temperature. The lower the temperature the larger the quantity of the A acid. Under the conditions above-mentioned about 2 parts of A and 3 parts of B are obtained. In a similar manner two isomeric acids are obtained from *o*-tolyl- $\beta$ -naphthylamine, which can be separated by means of the calcium salts.—F. A. L.

*Improvements in the Production of Orange-Red Colouring Matters from Flavopurpurin.* O. Imray, London. From the "Farbwerke vormals Meister, Lucius and Brünig," Höchst-on-the-Maine, Germany. Eng. Pat. 11,666, July 22, 1889. 6d.

A process for the production of two nitro-compounds designated  $\alpha$  and  $\beta$  from flavopurpurin—



The  $\alpha$ -compound is produced by the action of nitric acid on flavopurpurin in presence of concentrated sulphuric acid, whereas if glacial acetic acid be used the product will be the  $\beta$ -compound, the latter being a valuable colouring matter and preferable to  $\beta$ -nitro-alizarin on account of its dyeing a yellowish shade. It is obtained by mixing 1 kilo. of finely powdered flavopurpurin with 4—5 kilos. of glacial acetic acid, heating the mixture to 30°—35° C. and slowly adding 0.45 kilo. of nitric acid containing 60 per cent. of  $\text{HNO}_3$ . The temperature is then raised to 40°—45° for about an hour when the nitro-flavopurpurin is filtered off and washed. It dissolves with difficulty in ordinary solvents but crystallises from glacial acetic acid in ochre-yellow needles of melting point 265°. It dyes an orange tint on fibres mordanted with alumina. On reduction it is converted into the corresponding amido-compound which crystallises from acetic acid in brownish crystals and the salts of which are decomposed by washing with water.

—T. A. L.

*An Improved Manufacture of Compounds for Dyeing, Consisting of a Mixture of Nitrite with Basic Amido-azo Compounds, or with the Nitro-Compounds of Aromatic Monamines in the Form of a Paste, and the Application of such Paste for Diazotising.* O. Imray, London. From the "Farbwerke vormals Meister, Lucius and Brünig," Höchst-on-the-Maine, Germany. Eng. Pat. 12,031, July 29, 1889. 6d.

The patent describes the production of a paste for the use of dyers consisting of a mixture of equal molecules of nitrite and of one of the following bases, amido-azo-benzene, amido-azo-toluene, amido-azo-xylene, *m*-nitraniline, *p*-nitraniline, or *m* nitro-*p*-toluidine. For the formation of azo-colouring matters on the cotton fibre it is only necessary that the component which is not to be diazotised should be first fixed on the fibre and the material then passed through the diazo-bath obtained by adding the paste above described to a slight excess of hydrochloric acid.—T. A. L.

*Improvements in or Connected with the Manufacture and Production of Colouring Matters or Dyestuffs.* B. Wilcox, London. From the "Farbenfabriken vorm. F. Bayer and Co.," Elberfeld, Germany. Eng. Pat. 13,665, August 29, 1889. 6d.

The two naphthol disulphonic acids described in Ger. Pats. 40,571 and 44,079, when fused with potash or soda yield dihydroxynaphthalenemonosulphonic acids and trihydroxynaphthalenes. These substances combine in an alkaline solution with the salts of diazo and tetrazo compounds, yielding a variety of azo-colouring matters.—T. A. L.

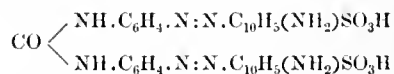
*Improvements in the Manufacture and Production of Colouring Matters or Dyestuffs.* The Clayton Aniline Co., Limited, P. Brunner and J. Hall, Manchester. Eng. Pat. 14,207, September 9, 1889. 6d.

The bases obtained by the action of sulphur on *p*-toluidine are dissolved in sulphuric acid and diazotised with sodium

nitrite in sulphuric acid. This solution is then combined with an alkaline solution of the sodium salt of the sulphonic acids of the above-mentioned bases, and the whole, after being stirred for some time, is raised to the boil. From the solution thus obtained the colouring matter is precipitated by adding salt, filter-pressed and dried. It dyes unmordanted cotton yellow. The yellow solution is turned deep red by the addition of an excess of caustic soda. In place of the bases first mentioned the sulphonic acids may be employed in a similar manner.—T. A. L.

*Improvements in the Manufacture and Production of Disazo Colouring Matters.* J. Y. Johnson, London. From the "Badische Anilin und Soda Fabrik," Ludwigshaven, Germany. Eng. Pat. 14,222, September 9, 1889. 6d.

The colouring matter described, which dyes unmordanted cotton salmon to orange-brown shades, is produced by the action of phosgene on *p*-amido-benzene-azo-naphthionic acid, and has the formula—



The following method is given for its preparation:—15 kilos. of finely powdered *p*-amido-acetanilide are dissolved in 45 kilos. of hydrochloric acid (containing 32 per cent. of  $\text{HCl}$ ), 200 litres of water, and 200 kilos. of ice, and diazotised with 7 kilos. of sodium nitrite in 21 litres of water. The diazo solution is run into 360 litres of water containing 31 kilos. of sodium naphthionate and 36 kilos. of calcined soda. When the azo compound is completely formed it is filtered off and boiled for some time with dilute caustic soda. By this means the acetyl group is split off, and the sodium salt of *p*-amido-benzene-azo-naphthionic acid is formed and crystallises out on cooling, when it is filter-pressed. To convert it into the disazo compound it is suspended in 450 litres of water, 600 kilos. of ice are added, and phosgene passed through the liquid until it shows an acid reaction. The colouring matter which is insoluble in the acid liquid separates out, and after filter-pressing is mixed with sufficient sodium carbonate to convert it into the sodium salt, and dried.—T. A. L.

*Improvements in the Manufacture, Production and Application of Colouring Matters.* B. Wilcox, London. From the "Farbenfabriken vorm. F. Bayer and Co.," Elberfeld, Germany. Eng. Pat. 14,230, September 9, 1889. 6d.

Moxo- and di-nitroso compounds of the dihydroxynaphthalenes are produced by the action of nitrous acid on the latter, and yield colouring matters which dye mordanted fibres. The employment of the (1:8) dihydroxynaphthalene is disclaimed. An aqueous solution of 10 kilos. of ( $\beta$ 1,  $\beta$ 4) dihydroxynaphthalene (obtained by fusing  $\beta$ -naphthol- $\beta$ -sulphonic acid (Schäffer) with potash) in dilute alkali, is treated with 4.5 kilos. of sodium nitrite, cooled with ice and acetic acid run in until the yellow nitroso compound is completely formed. This is then salted out, filtered, and used as a paste. Wool mordanted with aluminium sulphate, when boiled with it, is dyed a deep dark brown to a black. For cotton-printing chromium acetate is preferably used.

—T. A. L.

*Improvements in the Production of Amido-ory-naphthalin Disulphonic Acid and of Amido-diory-naphthalin Monosulphonic Acid, and the Manufacture of their Diazo-compounds, and of Azo Colouring Matters therefrom.* O. Imray, London. From the "Farbwerke vormals Meister, Lucius and Brünig," Höchst-on-the-Maine, Germany. Eng. Pat. 15,175, September 26, 1889. 6d.

By fusing the  $\beta$ -naphthylamine-trisulphonic acid corresponding to the  $\beta$ -naphthol-trisulphonic acid described in Eng. Pat. 2544 of 1882, with caustic alkalis, two of the sulphonic

groups may be successively replaced by hydroxyl, forming, firstly, amido-hydroxynaphthalene disulphonic acid, and secondly, amido-dihydroxynaphthalene monosulphonic acid. Both these compounds may be diazotised and combined with phenols or amines, or they will react with tetrazo compounds, yielding colouring matters in both cases.

—T. A. L.

*Improvements in the Production of Amido-naphthol-mono-sulphonic Acids, and the Manufacture of their Diazo Compounds and Azo Colouring Matters therefrom.* O. Imray, London. From the "Farbwerke vormals Meister, Lucius, and Brüning," Höchst-on-the-Maine, Germany. Eng. Pat. 15,176, September 26, 1889. 6d.

THE  $\beta$ -naphthylamine disulphonic acids corresponding to the  $\beta$ -naphthol disulphonic acids R and Y described in Eng. Pat. 1715 of 1878, when heated with caustic alkalis to 200°–280° yield amido-naphthol-mono-sulphonic acids which differ from the isomeric acids hitherto known in that they yield diazo compounds. The acids can also be combined with various tetrazo compounds, giving blackish-violet to blue-black dyestuffs. The following table gives some of the reactions of the two acids produced:—

—	R.	Y.
Solution of neutral salts in water.	Violet fluorescence.	Blue.
With ferric chloride.	Dark blue colouration, turning to tan colour.	Dirty claret-red colouration.
With chloride of lime.	Light yellowish brown colouration, which disappears rapidly on adding an excess.	Dark reddish brown colouration, which disappears gradually on adding an excess.
Diazo compound.	Reddish orange.	Canary yellow.
Combination of the diazo compound with R salt* in an alkaline solution.	Claret red.	Violet-black.

\* R salt is the sodium salt of  $\beta$ -naphthol disulphonic acid.

—T. A. L.

*Improvements in the Manufacture of Colouring Matters or Dyes.* A. Remy, R. Kramer, and W. Herking, Neuwied-Weissenthurm, Germany. Eng. Pat. 8389, May 30, 1890. 4d.

A PROCESS for the precipitation of the metallic double salts of the alkylated and non-alkylated mono- and diamido-benzophenones and their sulphonic acids, and also of the tetra-alkylated diamidothiobenzophenones by dissolving them in hot weak acid, adding metallic salts, such as zinc chloride or common salt, and allowing the solution to cool. These double salts, or the original ketones, are then to be reduced with zinc and hydrochloric acid, and the hydrols thus formed are precipitated as double metallic salts by means of zinc chloride and common salt, giving colouring matters which dye cotton mordanted with tannin and tartar emetic blue to reddish-violet shades. Further, the hydrols may be employed as dyestuffs in an alcoholic solution by using them together with a mineral or organic acid.—T. A. L.

## V.—TEXTILES: COTTON, WOOL, SILK, Etc.

*Thermo-Chemical Investigations on Textile Fibres (Wool and Cotton).* L. Vignon. Compt. Rend. **110**, 909–910.

THE author who has previously shown the value of thermo-chemical investigations in the case of silk (this Journal, 1890, 771), now applies the same method to wool and cotton.

The absolute weight of each sample of cotton wool and cotton thread was determined by drying, and it was afterwards exposed to the air of the laboratory so that it might take up its normal amount of moisture and acquire the temperature of the calorimeter. It was then plunged into the calorimeter and the evolution of heat observed.

*Wool.*—Unspun wool and wool in threads was treated by potash, soda, hydrochloric acid and sulphuric acid.

The weight of substance taken varied from 10 to 12 grms.; the volume of the reagent was in all cases 500 cc.

In every case a distinct evolution of heat took place which ceased after five minutes. The results have been calculated for 100 grms. of substance and also for a molecular formula,  $C_{88}H_{119}N_{27}O_{27}S_3$  (= 2,111).

### QUANTITIES OF HEAT EXPRESSED IN CALORIES EVOLVED BY TREATING WOOL WITH DIFFERENT REAGENTS AT A TEMPERATURE OF 11°–12° C.

Reagent.	Woollen Thread, Unbleached.		Unspun and Unbleached Wool.	
	For 100 Grms.	For $C_{88}H_{119}N_{27}O_{27}S_3$ .	For 100 Grms.	For $C_{88}H_{119}N_{27}O_{27}S_3$ .
KHO (1 litre = KHO).....	1.16	24.50	1.37	28.90
NaHO (1 litre = NaHO) .....	1.15	24.30	1.10	23.20
HCl (1 litre = HCl) .....	0.95	20.05	1.00	21.10
H <sub>2</sub> SO <sub>4</sub> (1 litre = $\frac{1}{2}$ H <sub>2</sub> SO <sub>4</sub> ).....	0.99	20.90	1.05	22.20

Considering the difficulty of the experiment, these figures agree remarkably well.

*Cotton.*—Unbleached cotton thread and bleached cotton wool were experimented on. A weight of from 9 to 13 grms.

was taken and immersed in 500 cc. of the solution of the reagent. Cotton is permeated by the liquid more slowly than wool, nevertheless, by agitation, the total evolution of heat was made to take place in from 7 to 8 minutes.

QUANTITIES OF HEAT (EXPRESSED IN CALORIES) EVOLVED BY TREATING COTTON WITH VARIOUS REAGENTS AT A TEMPERATURE OF 11°—12° C.

Reagents.	Cotton Thread, Unbleached.		Cotton Wool, Bleached.	
	For 100 Grms.	C <sub>6</sub> H <sub>10</sub> O <sub>5</sub> .	For 100 Grms.	For C <sub>6</sub> H <sub>10</sub> O <sub>5</sub> .
KHO (1 litre = KHO).....	0·80	1·30	1·40	2·27
NaHO (1 litre = NaHO) .....	0·65	1·05	1·35	2·20
HCl (1 litre = HCl) .....	0·40	0·65	0·40	0·65
H <sub>2</sub> SO <sub>4</sub> (1 litre = $\frac{1}{2}$ H <sub>2</sub> SO <sub>4</sub> ).....	0·38	0·60	0·36	0·58

Bleached cotton evolves more heat with the alkalis than unbleached cotton. This is probably due to the formation of oxy-cellulose during the bleaching.

The author proposes to show in a future note how these and further results bear on the theory of dyeing.—P. J. H.

#### PATENTS.

*An Improved Method of Treating Cotton Fibres, or Masses of Cotton Fibres Preparatory to being Worked Up or Made into Yarns for Weaving or Manufacturing Purposes, applicable also to Fibres, Animal or Vegetable, other than Cotton.* R. S. Burn, Stockport. Eng. Pat. 9904, June 17, 1889. 11d.

THE object of this invention is to subject compressed masses of fibres, especially raw cotton as taken from the bales, to the action of currents of air or of "aeriform fluids" at pressure, in order to loosen them, &c. This has hitherto generally been done in various ways, viz., by beating, blowing and seutching machines, and the principle of this invention is to avoid as completely as possible such rough and harsh treatments which are injurious to the fibres. The cotton taken from the bales is brought into chambers, the inner walls of which have either a smooth or a corrugated or ribbed surface which the author proposes in some cases to cover with vulcanised india-rubber or other strong but soft and elastic material. This serves to make the concussions between walls and fibres as little injurious to the latter as possible. The form of the chambers is that of a cylinder or of a truncated cone. When the fibres are placed into the chambers, strong currents of air are produced in the chambers in various directions, and these currents of air, by producing a quick and violent motion of the masses of fibres, open them up. A pressure of several atmospheres may first be applied, but as the process of treatment goes on, and the masses of fibres become more and more separated, and take a light feathery free and open form, the pressure is gradually reduced so that dust, dirt and other impurities may fall to the bottom of the chamber, whilst the fibres are removed from the chamber by a gentle current of air which drives them through a tube at the top, the valve of which has been opened for the purpose. The time of treatment and pressure vary according to the nature of the masses of fibres to be operated upon.

Instead of air at ordinary temperature it may be useful in some cases to apply currents of hot air or of gases which have a bleaching or purifying effect upon the fibres.—H. S.

*Improvements in the Cleansing or Washing of Wool and like Animal Fibres, and in Apparatus employed therein.* A. Ambler, S. Ambler, and F. Ambler, Wilsden. Eng. Pat. 13,626, August 29, 1889. 11d.

THIS invention consists in applying for wool-washing purposes a closed conduit or channel which has abrupt changes of direction. Into this channel, wool and washing liquid are introduced simultaneously and continuously, the changes of direction effecting an intimate admixture of both, and so a thorough washing and cleansing of the wool. The liquid and dirt are separated from the wool fibres by means of

a straining process, gauze, wirework, grids, &c. being introduced into various parts of the channel for this purpose. The abrupt changes of direction may practically be applied by giving the closed conduit a zig-zag form.—H. S.

*Improvements in the Treatment of Fibres and Fibrous Materials, made of China-grass, Flax, Hemp, and the like Fibres, by which means such can be more easily Cleansed and Bleached.* K. T. Sutherland and G. Esdaile, Manchester. Eng. Pat. 15,333, September 30, 1889. 4d.

THE material is placed in a weak solution of carbonate of soda mixed with mineral oil or wax (1 pint of oil or  $\frac{1}{2}$  lb. of wax to 8 gallons of the solution). The material is worked in this solution (which is used "hot or boiling") from  $\frac{1}{2}$  to 3 hours, according to the state of the fibres, then wrung out, washed in water and dried. When the material is dry, it is placed for some time in paraffin oil, then wrung out and passed into a hot or cold weak solution of sodium carbonate in which it is left from ten minutes to half an hour. Afterwards the material is squeezed out and passed into a vat containing hot or cold water, in which it is steeped, beaten, squeezed or hammered, until it becomes cleansed and bleached. If the result be not satisfactory, the process can be repeated from the steeping in paraffin oil, until the material is completely bleached and cleansed. The advantages of this method of treatment are stated to be that the materials are more easily cleansed and bleached without fear of injuring the fibre, and that they are not liable to "ent"; that the fibre may be spun in short or longer lengths as required; that it will "flock"; and that the waste is less in spinning the fibre in short lengths.—H. S.

#### VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

*Fastness to Light of Substantive Cotton Dyes.* O. Müller. Oest. Woll. u. Leinen-Ind. 6, 288.

SAMPLES of cotton fabrics, dyed with various substantive dyes, were exposed to the light for 14 days with the results given below. The following dyes remained unaltered: Benzoazurine 3 G (treated, after dyeing, with copper sulphate), Chrysamin G and R, Brilliant yellow, Chrysophenin, Hessian yellow, Cureumine S, Mikado orange G, R, and RR, Brahma orange, and Diamine yellow N.

There were slightly altered: Congo eorinth G, B, Benzoazurine 3 G G (treated, after dyeing, with copper sulphate and soaped), Azo-violet, Congo brown G, Benzo-brown G, B, NB, Benzo-black-blue, Mikado brown G, B, Salmon red, Diamine red NO, N, Diamine blue 3 R, Fast pink B and G.

The following were destroyed: Congo red (all shades), Brilliant Congo, Hessian purple NG, B, NB, Benzopurpurin B, 4 B, 6 B, 10 B, Deltapurpurin 5 B, G, Benzoazurine 3 G, G, Azo-blue, Rosazurin BG, Heliotrope, Hessian violet,

Thiazol yellow, Sulphonazurine, Diamine blue B, St. Denis red, Brahma red, Violet-black.

The effect of the treatment with a boiling solution of copper sulphate of cotton goods dyed with Benzoazurin (this Journal, 1890, 289) varies according as the goods have been rinsed or not after dyeing. Repeated rinsing of the dyed cloth before treatment with the copper salt renders the action of the latter useless, the colours then being as little fast as when untreated.—E. B.

## PATENTS.

*Improvements in Bleaching, Disinfecting, Purifying and Preserving Substances, and Apparatus therefor.* G. Brownen, London, and J. Gamgee, Wimbledon. Eng. Pat. 9763, June 13, 1889. 8d.

See under XVIII. C., page 883.

*Improvements in the Manufacture, Production, and Application of Colouring Matters.* B. Willcox, London. From the "Farbenfabriken vorm. F. Bayer and Co.," Elberfeld, Germany. Eng. Pat. 14,230, September 9, 1889. 6d.

See under IV., page 854.

*Improvements in the Method of Preparing Colours for Making Writing Inks.* R. Ashton, Manchester. Eng. Pat. 14,388, September 12, 1889. 6d.

PAPER, vegetable parchment, gelatin, sugar or other suitable carrier is to be coated with a strong solution of a coal-tar dyestuff, dried and cut into portions of a convenient size. When required for use one or more of these pieces is placed in an inkstand with a little water, thus furnishing a writing ink of the same colour as that with which the material was originally coated.—T. A. L.

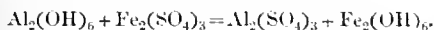
## VII.—ACIDS, ALKALIS, AND SALTS.

*Some Observations on the Relations of the Ferric and Aluminium Hydroxides and of some Iron and Aluminium Salts to each other.* E. A. Schneider. Ber. 23, 1349—1354.

### A.—RELATIONS OF ALUMINIUM HYDROXIDE TO FERRIC SALT SOLUTIONS.

#### I.—Aluminium Hydroxide and Ferric Sulphate Solutions.

Freshly precipitated aluminium hydroxide precipitates all iron from neutral solutions of ferric sulphate at the boiling as well as at lower temperatures. This, however, cannot be explained by the equation—



but it is known that ferric sulphate solutions dissociate easily into basic salt and free acid, and if such solutions be boiled with aluminium hydroxide, the sulphuric acid which becomes free by dissociation is neutralised by the aluminium hydroxide. The ferric sulphate solution thereby becomes more and more basic, and an accelerated precipitation of basic iron salt is the result.

100 cc. of a weak basic ferric sulphate solution containing 0.5126 grm. of  $\text{Fe}_2\text{O}_3$  and 0.6596 grm. of  $\text{SO}_3$  were boiled with 0.7460 grm. of aluminium hydroxide for an hour and a quarter (1 mol. of  $\text{Fe}_2\text{O}_3$  : 1.4 mols. of  $\text{Al}_2\text{O}_3$ ). The filtrate contained:—grm. of  $\text{Fe}_2\text{O}_3$ , 0.1893 grm. of  $\text{Al}_2\text{O}_3$ , and 0.3980 grm. of  $\text{SO}_3$ , whilst it should have contained 0.6596 grm. if the reaction had been in accordance with the above equation; moreover, the iron is completely precipitated by the aluminium hydroxide, not only at the boiling, but

also at lower temperatures. Dissociation also in this case takes place, but it is preceded by a catalytic influence of the aluminium hydroxide. Time has little influence when the reaction is made at lower temperatures, e.g., two portions of a weak basic ferric sulphate solution, each containing 0.2068 grm. of  $\text{Fe}_2\text{O}_3$ , were gently heated each with 0.7460 grm. of aluminium hydroxide (1 mol. of  $\text{Fe}_2\text{O}_3$  : 3.6 mols. of  $\text{Al}_2\text{O}_3$ ), the first portion for 24 hours, the second only for half an hour. In both cases the filtrate was free from iron; in the first case it contained 0.0904 grm. of  $\text{Al}_2\text{O}_3$ ; in the second 0.0770 grm. of  $\text{Al}_2\text{O}_3$  (as sulphate). The influence of dilution on the reaction made at lower temperatures is in accordance with the fact that with increasing dilution the dissociation of the ferric sulphate continuously increases, and that therefore the same quantities of aluminium hydroxide precipitate larger quantities of iron, e.g., 25 cc. of a ferric sulphate solution which contained 0.2507 grm. of  $\text{Fe}_2\text{O}_3$  were gently heated with 0.7857 grm. of aluminium hydroxide for half an hour (3.2 mols. of  $\text{Al}_2\text{O}_3$  : 1 mol. of  $\text{Fe}_2\text{O}_3$ ); the filtrate contained 0.0773 grm. of  $\text{Fe}_2\text{O}_3$ . But the filtrate of a ferric sulphate solution, diluted to 2,000 cc., containing the same quantity of ferric oxide and treated as before with aluminium hydroxide, contained only 0.0072 grm. of  $\text{Fe}_2\text{O}_3$ . The process varies when the reaction is made at boiling heat. All iron is equally precipitated, in concentrated as well as in dilute solutions. The quantities of alumina, being in solution as aluminium sulphate, are in most cases almost the same, as the following figures will show:—

25 cc. of Ferric Sulphate Solution contain 0.2068 grm. $\text{Fe}_2\text{O}_3$ . The following cc. of water are added to every 25 ccm. of Ferric Sulphate Solution.		Quantities of $\text{Al}_2\text{O}_3$ in Solution after boiling.	No $\text{Fe}_2\text{O}_3$ found in the Solutions.
	Ccm.	Grm.	
I.	25	0.0760	
II.	100	0.0769	
III.	225	0.0768	
IV.	1175	0.0648	
V.	1175	0.0760	

In all these instances the substance was boiled for an hour and a quarter. In the experiments I. to IV. the quantity of aluminium hydroxide was 0.7460 grm. = 0.4888 grm.  $\text{Al}_2\text{O}_3$ , therefore we have approximately the proportion  $3\frac{1}{2}$  mols. of  $\text{Al}_2\text{O}_3$  : 1 mol. of  $\text{Fe}_2\text{O}_3$ . In the experiment V. the proportion was: 1  $\frac{1}{2}$  mol.  $\text{Al}_2\text{O}_3$  : 1 mol.  $\text{Fe}_2\text{O}_3$ .

#### II.—Aluminium Hydroxide and Ferric Chloride Solutions.

Freshly precipitated aluminium hydroxide is quickly and completely dissolved to a brown-red solution at ordinary temperature in neutral and basic ferric chloride solutions. This solution immediately coagulates when very small quantities of sulphuric acid are present, and the precipitate contains various quantities of ferric and of aluminium hydroxide, sometimes even the entire quantity of them, according to the duration of the reaction and the concentration of the ferric chloride solution. The dissociation of the ferric chloride solution begins here because of the catalytic influence of the present aluminium hydroxide, the hydrochloric acid which becomes free combines with the aluminium hydroxide, forming aluminium chloride, and the ferric hydroxide remains in solution in its colloid modification.

Aluminium hydroxide has the same relations to ferric nitrate solutions as to the just described ferric chloride solutions.

### B.—RELATIONS OF FERRIC HYDROXIDE TO ALUMINIUM SALT SOLUTIONS.

#### I.—Ferric Hydroxide and Aluminium Sulphate Solutions.

Freshly precipitated ferric hydroxide is dissolved easily and in considerable quantities in concentrated aluminium sulphate solutions. A weak basic aluminium sulphate solution, containing 80.7 grms. of  $\text{Al}_2\text{O}_3$  and 0.1287 grm. of

$\text{Fe}_2\text{O}_3$  per litre, was heated for some time on the water-bath with dry ferric hydroxide, until a precipitate was obtained on the cooling of the liquid. The resulting dark brown-red solution contained 66.73 grms.  $\text{Al}_2\text{O}_3$  and 6.62 grms.  $\text{Fe}_2\text{O}_3$  per litre, and was evaporated to dryness without being decomposed. By subsequently adding water a basic salt was precipitated, showing approximately the following composition:  $\text{SO}_3, 3\text{Fe}_2\text{O}_3, 3\text{H}_2\text{O}$ . The formation of this compound cannot result but from the influence of a part of the sulphuric acid which was combined with the alumina, whilst the alumina becoming free, is dissolved in the aluminium sulphate which is present in excess. In this case ferric hydroxide acts as a basis which is stronger than aluminium hydroxide.

## II.—Ferric Hydroxide and Aluminium Chloride Solutions.

Freshly precipitated ferric hydroxide is dissolved in neutral aluminium chloride solutions in considerable quantities, if gently heated for some length of time. If, however, a part remains suspended in the solution, and is collected by means of a filter and washed, then the water always assumes a yellow colour. The ferric hydroxide thus collected on the filter now easily dissolves in larger quantities of water, forming an intensely red-brown solution, showing fluorescence. The results of the experiments were as follows:—

100 cc. of an aluminium chloride solution which contained 0.858 gm. of  $\text{Al}_2\text{O}_3$ , dissolved a quantity of ferric hydroxide containing 0.820 gm. of  $\text{Fe}_2\text{O}_3$ ; 0.752 gm. of  $\text{Fe}_2\text{O}_3$  were coagulated by a minute quantity of sulphuric acid. The resulting solution appeared light yellow. 0.717 gm. of  $\text{Al}_2\text{O}_3$  remained dissolved, and 0.141 gm. of  $\text{Al}_2\text{O}_3$  were precipitated. 100 cc. of the same solution, treated with the same quantity of ferric hydroxide, and diluted with water to 1,000 cc., dissolved a quantity of ferric hydroxide containing 1.747 grms. of  $\text{Fe}_2\text{O}_3$ ; 1.691 grms. of  $\text{Fe}_2\text{O}_3$  were coagulated by a minute quantity of sulphuric acid. The resulting solution appeared dark brown, 0.818 gm. of  $\text{Al}_2\text{O}_3$  remained dissolved, and 0.040 gm. of  $\text{Al}_2\text{O}_3$  were precipitated. A strong basic aluminium chloride solution (1 mol. of  $\text{Al}_2\text{O}_3$ ; 7 mols. of  $\text{HCl}$ ) showed the same relations to ferric hydroxide as described. The author adds that he has hitherto not found a satisfactory explanation of these experiments.

## III.—Ferric Hydroxide and Aluminium Nitrate Solutions.

Aluminium nitrate differs from aluminium chloride inasmuch as solutions of the first, containing the same quantities of alumina as the aluminium chloride solutions used in the above-described experiments, dissolve only very small quantities of ferric hydroxide. But when ferric hydroxide is mixed with an aluminium nitrate solution of the said concentration, and water afterwards added, then a considerable quantity of ferric hydroxide is dissolved. 100 cc. of an aluminium nitrate solution, containing 0.858 gm. of  $\text{Al}_2\text{O}_3$ , dissolved only 0.024 gm. of  $\text{Fe}_2\text{O}_3$ , after having been gently heated for some length of time. This solution was not coagulated by a minute quantity of sulphuric acid. But 100 cc. of the same aluminium nitrate solution, treated with the same quantity of ferric hydroxide, and diluted to 1,000 cc., dissolved a quantity of ferric hydroxide containing 1.148 grms. of  $\text{Fe}_2\text{O}_3$ . That part of the ferric hydroxide which remained unaffected after being washed with much water, gave a solution of colloid modification.

The author finally adds some remarks upon the relations of aluminium chloride solutions to very small quantities of sulphuric acid. It is known that aluminium hydroxide will be precipitated from basic aluminium chloride solutions if the minutest quantities of sulphuric acid be added. The author considers it a new item that acid aluminium chloride solutions are also coagulated by sulphuric acid, e.g., an acid aluminium chloride solution (1 mol.  $\text{Al}_2\text{O}_3$ ; 7.2 mols.  $\text{HCl}$ ), containing 0.1894 gm.  $\text{Al}_2\text{O}_3$  in 400 cc., gave a precipitate of 0.0486 gm. of  $\text{Al}_2\text{O}_3$  on adding a very minute quantity of sulphuric acid, this being 25.6 per cent. of the total quantity of alumina present. Moreover, the author finds that solutions of aluminium

chloride coagulate in larger quantities by sulphuric acid if they have been prepared from aluminium hydroxide which was kept for some time in ammonia water, than if they have been prepared from freshly-precipitated aluminium hydroxide.—H. S.

## The Action of Hydrogen Dioxide on the Oxides of Manganese. A. Gorgen. *Compt. Rend.* **110**, 857—859.

See under XXIII., page 895.

## On Soda-Alum. E. Augé. *Compt. Rend.* **110**, 1139—1140.

THE author shows how unreliable are the data given in chemical text-books and treatises with respect to the properties of soda-alum.

Statements.	Facts.
(1.) Soda-alum is exceedingly efflorescent and falls to powder after a few days' exposure (all authors).	(1.) Soda-alum is but slightly efflorescent and keeps without alteration for months.
(2.) Its solubility is considerable. 100 parts water dissolve 110 parts soda-alum at 16° C. ( <i>Dictionnaire de Wurtz</i> ).	(2.) 100 parts water dissolve 51 parts soda-alum at 16° C.
(3.) A solution of soda-alum boiled loses its property of crystallisation ( <i>Muspratt, Wagner, &amp;c.</i> ).	(3.) A solution of soda-alum may be boiled indefinitely without losing the property of crystallisation.

If a solution containing aluminium sulphate and sodium sulphate in the proper proportions is boiled down to above 38° B. (hot); such a solution on cooling yields an amorphous paste in which even microscopically no crystals can be observed. The formation of this amorphous mass is solely due to the high concentration of the liquor, and not a function of the temperature. If same solution is cooled down before sp. gr. 38° B. is reached crystals of soda-alum are obtained. The amorphous mass between 7° and 25° C. parts gradually with its mother-liquor and is transformed into crystals of  $\text{Al}_2\text{O}_3, 3\text{SO}_3, \text{Na}_2\text{O}, \text{SO}_3, 24\text{aq.}$  For layers 5 cm. in thickness the crystallisation is complete in three days. Above 28° C. the transformation into a crystalline mass is extremely slow, at about 0° C. crystals of soda-alum are formed accompanied by crystals of sodium sulphate. This latter phenomenon explains statement No. 1, the soda-alum obtained in the laboratory having been mixed with sulphate of soda.

According to the foregoing the manufacturer must concentrate the solution of the two sulphates up to 39°—43° B., then cool and drain the amorphous mass, the mother-liquor draining off, representing about quarter of the whole mass, will carry nearly all impurities away; after the crystallisation is complete at about 15° C. the crystals are dried. (See abstract of *Eug. Pat.* 11,975, on next page.)—K. M.

## On the Oxides of Manganese obtained in the Wet Way. Second Part: Manganous Acid. A. Gorgen. *Compt. Rend.* **110**, 1134—1137. (See this Journal, 1889, 541 and 774.)

A CLOSE study of the methods of preparation of binoxide of manganese and of the oxides obtained has led the author to the conclusion that it is practically impossible to isolate the pure hydrate of manganese peroxide. The best way to obtain the hydrate consists in mixing cold and strongly acidified solutions of nitrate of manganese and permanganate of soda; but even by this method it is impossible to obtain hydrated binoxide of manganese free from manganous oxide.

The hydrated binoxide or manganous acid reddens blue litmus and combines with soluble basic oxides producing manganites; it decomposes carbonates, acetates, and even sulphates. The composition of the manganite of potassium obtained from the decomposition of the acetate by the hydrate of the peroxide is expressed by the formula  $25(\text{MnO}_2)_2\text{K}_2\text{O}$ , and the corresponding soda compound by

$25(\text{MnO}_2)\text{Na}_2\text{O}$ ; while the manganites obtained in presence of acetates and sulphates vary from  $3(\text{MnO}_2)\text{K}_2\text{O}$  to  $9(\text{MnO}_2)\text{K}_2\text{O}$ . The anhydrous binioxide acts as an indifferent oxide; the presence of manganous hydrate, however, seems to lend it an acid character. An interesting reaction was observed by the author. If to an alkaline (potash) solution of permanganate of potash which does not appreciably decompose when cold or on boiling manganous hydrate or still better manganite of potash  $5(\text{MnO}_2)\text{K}_2\text{O}$  is added, these two compounds are dissolved without evolution of oxygen and the solution turns a green colour. The lower oxides are evidently oxidised and the permanganate reduced to manganate.—K. M.

*New Mode of obtaining Crystalline Metallic Oxychlorides; the Oxychlorides of Copper.* G. Rousseau. *Compt. Rend.* **110**, 1261–1264.

In a previous paper the author has shown that a crystalline ferric oxychloride may be obtained by heating a very strong solution of ferric chloride with the carbonate of an alkaline earth. He finds, contrary to his first expectations, that the method is not general, but confined to a small number of chlorides which are either extremely soluble in water or form hydrates of slight complexity capable of undergoing aqueous fusion. In the contrary case amorphous precipitates are obtained, as for instance from solutions of aluminium chloride. In accordance with Berthelot's principle of maximum work the decomposition of such chlorides is only possible when the heat of combination of the metal with chlorine does not exceed its heat of combination with oxygen by more than 4.8 cal., i.e., the difference between the heats of formation of hydrochloric acid (diluted) and water (in the liquid state). This at once limits the reaction to the chlorides of tin, titanium, antimony, bismuth, magnesium, zinc, and iron. Certain exceptions are, however, apparent; thus the formation of atacamite,  $3\text{CuO}, \text{CuCl}_2, \text{H}_2\text{O}$ , takes place easily under the most varied conditions although the difference between the heat of formation of cupric chloride and cupric oxide is greater than 4.8 cal. being, in fact, 6.8 cal. Berthelot explains this, however, by showing that although it is inexplicable from the heat of formation of  $3\text{CuO}, \text{CuCl}_2$  which is insignificant, yet an adequate cause is found in the heat of hydration of this salt, which is 10.9 cal., a quantity large enough to determine the reaction. Starting from these considerations one would expect to obtain oxychlorides of different compositions stable under different conditions of equilibrium, by the use of small quantities of water and higher temperatures, thus tending to dissociate the hydrated chloride and cause the formation of a notable proportion of the anhydrous salt. The author therefore experimented on the action of calcium carbonate, in the form of marble, on cupric chloride,  $\text{CuCl}_2, 2\text{H}_2\text{O}$ , when heated in sealed tubes at  $150^\circ\text{--}200^\circ\text{C}$ . After 48 hours a small quantity of a new crystalline oxychloride,  $\text{CuCl}_2, \text{CuO}, \text{H}_2\text{O}$ , was obtained; it was easily decomposed by water and was freed from the excess of unaltered cupric chloride by dissolving out the latter with boiling alcohol; by using a higher temperature the yield was greater and at  $250^\circ\text{C}$ . in the presence of natural magnesium carbonate (giöbertite) the crystals became larger ( $5 \times 2\text{ mm.}$ ). The new oxychloride forms yellowish green hexagonal tables which lose their water of crystallisation above  $250^\circ\text{C}$ . while retaining their shape; at dull red heat they decompose. When brought into contact with water they lose their transparency and their tint alters to pure green; at the same time hydrochloric acid is set free and they are found to be converted into atacamite (*v.s.*). The same reaction occurs when the salt dehydrated at  $100^\circ\text{C}$ . is used; a green colloidal mass is first formed which gives a pseudo solution, clear by transmitted, turbid by reflected light, and precipitated by the addition of an alkaline lorde.

By increasing the quantity of water used with the original ferric chloride to the proportion corresponding to the formula  $\text{CuCl}_2, 5\text{H}_2\text{O}$ , the new oxychloride is no longer modified, and on exceeding this limit and heating for three days at  $180^\circ\text{--}200^\circ\text{C}$ . atacamite is obtained identical with the natural variety; this constitutes a new method of pro-

ducing this substance essentially different from that employed by Debray. More diluted liquids give poor results. Work on similar basic salts is in progress.—B. B.

## PATENTS.

*Improvements in the Manufacture of Soda-Alum.* E. Augé, Montpellier, France. Eng. Pat. 11,975, July 27, 1889. 6d.

In the usual way of manufacturing soda-alum, solutions of equivalent parts of aluminium sulphate and sodium sulphate are heated to a temperature not exceeding  $60^\circ\text{C}$ ., and allowed to crystallise. The improvement consists in establishing the degree of concentration of the final liquor; this should not exceed at any stage of the process a density of about 70 Tw. at  $50^\circ\text{C}$ ., otherwise no crystallised alum will be obtained; whilst on the other hand a lower density interferes considerably with the yield of crystals. The required concentration can be obtained approximately by using a solution containing the molecular weight of aluminium sulphate per litre at  $50^\circ\text{--}60^\circ\text{C}$ ., and suspending therein the required quantity of pure anhydrous sodium sulphate. When all the sodium salt is dissolved, the clear liquor of 70° Tw. is allowed to cool in crystallising vessels, the crystals separated, whilst the mother-liquor returns to the process. (See also preceding page.)—H. A.

*Improvements in Generators for making Carbonic Acid Gas.* E. S. Chavasse, Ombersley. Eng. Pat. 12,346, August 3, 1889. 8d.

The object of this invention is to render the working of the generator automatic. Besides a pump for the supply of the whitening and water there are mechanical stirrers for the intimate mixing of the carbonate with sulphuric acid, the supply of which is regulated automatically by the height of the gas-holder in the following way. The top of the holder is connected by means of a chain leading over pulleys to a vertical rack, provided with slides and moving in guides. The rack is geared with a lever arm in the form of a toothed sector; the arm is centred on a pin and the other end formed as a small toothed sector to gear with a similar toothed sector on the plug of an earthenware tap on the vitriol supply pipe. When the holder is rising, the rack is set in downward motion, and so checks the tap; when the holder is falling, the supply of vitriol is in the corresponding way augmented; and when the holder is at its lowest point, the lever will fall out of gear altogether and shut off entirely the supply of vitriol. At this juncture the generator may be emptied of its spent contents by means of a sluice valve at the bottom, and carbonic acid from the holder will simultaneously enter into the generator (by means of a pipe provided with a back-pressure valve), thus preventing access of air and subsequent dilution of the carbonic acid.—H. A.

*Improvements in or Connected with the Manufacture of Acetic Acid, and in Apparatus therefor.* J. Farmer, Pollokshields. Eng. Pat. 13,264, August 22, 1889. 11d.

In the usual way of manufacturing acetic acid, acetate of lime is decomposed in a still with hydrochloric acid, and the escaping acetic acid passed through purifiers, which retain the tarry matters. These purifiers are liable to block up, and in consequence of the pressure exercised there is a frequent escape of acetic acid from the joints of the still. To obviate this nuisance the still is provided with two lids, an inside lid, made of gun-metal or stone, and an outside stone lid, jointed over the whole top. The inside lid is supported on its vertical lip in an annular gutter, jointed to the top-outside part of the still and luted with the purifying liquor. In distilling off the acetic acid from the charge, it passes through the seal (which may consist of pure acetate of lime and hydrochloric acid, with some paraffin on the outer seal), and from hence through the space between the two lids to the condenser, without exercising any considerable pressure.

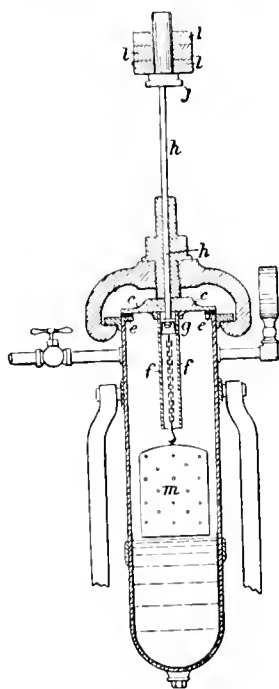
Towards the end of the reaction the charge is steamed up; this is effected by means of a perforated copper pipe with stuffing-box and fittings for its withdrawal from the still, when not in use; the choking up of the perforations with mud is thus obviated.

The specification contains the drawings and description of another still, in which facilities are given for the cleaning out of the annular gutter.

The application of the improvements to an ordinary acetic acid still is also described and illustrated.—H. A.

*An Apparatus for Generating Carbonic Acid Gas under Pressure, to be used in Forcing or Raising Fermented Liquors from one Vessel to another.* C. Estcourt, Manchester. Eng. Pat. 13,309, August 23, 1889. 8d.

In this apparatus the supply of carbonate or bicarbonate is regulated automatically by the pressure of carbonic acid in the vessel. The generator is a cylindrical vessel with hemispherical bottom and a lid on the top. The pressure



THE GENERATION OF CARBONIC ACID.

is made to act on the piston, *g*, of a small cylinder, *f*, open at the bottom, which is fixed to the under part of the lid *c*. On the piston *g* is suspended a cage *m* containing the carbonate, which dips in the acid. The piston rod *h* extends through the lid, and is provided on its upper part with a support for weights *l*, whereby the pressure of gas to be obtained is capable of being adjusted and regulated to the greatest nicety.—H. A.

*Improvements in Treating Liquid Acid Residues containing Iron to obtain Products therefrom.* M. N. d'Andria, Manchester. Eng. Pat. 13,575, August 28, 1889. 4d.

The liquid acid residues from cleaning iron, previous to tinning, galvanising, wire-drawing, &c. consist of sulphate of iron and sulphuric acid. It is proposed to utilise this for the manufacture of iron pigment and magnesium sulphate. Milk of magnesia is added and the iron thus precipitated oxidised with a blast of air. After undergoing the usual processes the iron oxide will be available as a pigment, whereas the filtered liquor is boiled down for the manufacture of magnesium sulphate.—H. A.

*Improvements in the Preparation of Alkaline Cyanides and in Apparatus therefor.* H. Grüneberg, Köln, H. Flemming, Kalk, and W. Siepermann, Elberfeld, Germany. Eng. Pat. 13,697, August 30, 1889. 8d.

The invention consists in passing ammonia over a mixture of alkaline carbonate and charcoal at a dark red heat, and subsequently raising the temperature to a bright red in an atmosphere free from ammonia. The operation is performed in a series of vertical retorts, which are set in a furnace in such a way that the fire gases strike first on the lower part of the retorts, heating this to bright red, and in returning, the higher part to dark red. The ammonia is supplied from the top of the retort by means of a vertically adjustable pipe, which is introduced almost as far as the bright red part of the retort. The liquid products of the reaction are collected in a mutual receiver. The remaining mass in the retorts is boiled out with water and the cyanide separated in the usual way—or, in the case of potassium cyanide precipitated with sodium carbonate.—H. A.

*An Improved Process for Recovering Valuable Substances from the Mother-Liquor Resulting from Treatment of Copper Ores.* E. de Cuyper, Perennes-lez-Binche, Belgium. Eng. Pat. 13,726, August 30, 1889. 8d.

The object of this invention is the separation and utilisation of the zinc, sulphate of sodium, and iron contained in the liquors resulting from the treatment of copper ores by chlorination.

The liquor is evaporated down to a pasty condition in special chambers, by causing a fine spray (caused by a blast or agitation) of the liquor to meet the gases from a gas furnace. Hydrochloric acid and chlorine will be given off and a mixture of ferric oxide, oxychloride of zinc, sulphate and chloride of sodium remains. Water will dissolve the latter three constituents; zinc is precipitated by addition of carbonate or sulphide of sodium. It is proposed to treat the solution of chloride and sulphate of sodium with sulphuric acid and allow the sulphate of sodium to crystallise, or else to treat with bicarbonate of ammonium for the manufacture of alkali.

Instead of boiling down to pasty condition the liquor may be evaporated to 40° B. and the sulphate of sodium and ferric chloride fished out as they separate. The fished salts are calcined, the sulphate of sodium dissolved out and obtained by cooling down the solution to -3° C. The mother-liquor from the fished salts contain the zinc, which is precipitated as described before.—H. A.

*Improvements in Apparatus for Evaporating Brine and the like under a Vacuum.* W. Young, Droitwich. Eng. Pat. 13,814, September 2, 1889. 6d.

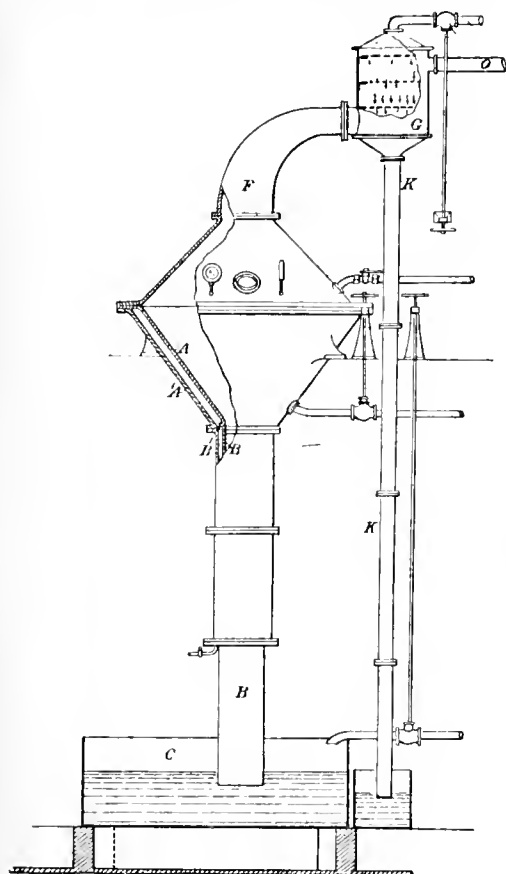
BRINE is evaporated under vacuum and the salt collected by means of mechanical rakes, or an endless screw, and deposited in pockets or boxes, which are in direct communication with the evaporating vessel. The boxes are provided with tight-fitting doors, which allow of the occasional removal of the salt.—H. A.

*Improvements in and Connected with Apparatus for Evaporating Brine and the like in Vacuum.* W. Young, Droitwich. Eng. Pat. 13,816, September 2, 1889. 8d.

The object of this invention is to produce salt in a continuous manner with economy of fuel and employment of unskilled labour. The apparatus consists of a conical vessel *A*, provided with a cone-shaped lid, and with appliances common to most vacuum pans. The bottom cone is narrowed down to a pipe *B*, 24 ft. long, dipping into a pan *C* fed with brine and preferably kept warm by waste heat from any available source. Part of the tube and the lower cone are surrounded with a steam-jacket, *A*<sup>1</sup>, *B*<sup>1</sup>. The upper cone is connected by a swan-neck *F* with the condenser *G*, terminating at the bottom in a pipe *K*, which passes down to a water tank; the height of this pipe should



of course be such that the water within will be below the bottom of the condenser. The condenser is connected with an air-pump at Q. In starting, the brine is drawn by the



THE EVAPORATION OF BRINE.

pump up the 24-ft. pipe until it reaches the middle of the boiling chamber, and is kept at this level by the vacuum—as is the case in Torricelli's experiment. Steam is now admitted in the jacket, and the vapours given off by the brine are condensed; as the brine becomes more concentrated, salt will separate and drop down the column of liquor in the tank below, and fresh brine will rise up in its place. The salt is continuously removed out of the bottom pan by means of rakes. It is obvious that the brine level in the bottom pan will influence the brine level in the boiling chamber.—H. A.

*Improvements in obtaining Ammonia, Chlorine, and Hydrochloric Acid from Ammonium Chloride, and in the Apparatus Employed therein.* F. Bale, Droitwich. Eng. Pat. 15,649, October 5, 1889. 8d.

Oxide of manganese in powder is suspended in molten chloride of zinc at a temperature below 350°C. and powdered chloride of ammonium introduced in the mixture. Ammonia is given off, and a chloride of manganese or an oxychloride is formed. The last traces of ammonia are eliminated by passing cold and dry air through the mixture. The temperature is then raised and hot and dry air introduced; the chloride or oxychloride is thus decomposed into chlorine (the first gases contain 12–20 per cent. of Cl) and oxide of manganese regenerated. When steam is used in place of dry air, hydrochloric acid is given off. The last traces of chlorine or hydrochloric acid are removed by a current of cold air and a new quantity of ammonium chloride added as before.

The process is carried out in retorts lined with ganister and “a layer of the fused chloride of a metal, whose fusion point is considerably above the temperature required for these reactions.” The retorts are worked in series and alternate ways of working are described. Chloride of zinc can be substituted by any other chloride or metal which is not affected by hydrochloric acid or ammonia vapour. In place of oxide of manganese other oxides may be used.

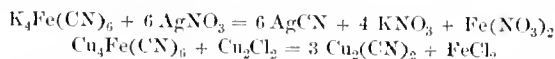
—H. A.

*A New or Improved Process for the Recovery or Regeneration of Nitric Acid Employed in the Manufacture of Chlorine.* W. Wolters, St. Petersburg, Russia. Eng. Pat. 16,609, October 21, 1889. 4d.

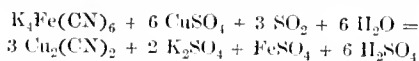
THE weak point in the manufacture of chlorine by the action of hydrochloric acid and nitric acid on manganese is the recovery of the nitric acid, considerable quantities of which are lost in calcining the nitrate of manganese liquor and in the subsequent conversion of the hyponitric acid vapours into nitric acid, by means of air and water. It is proposed to distil off the excess of nitric acid from the still liquor, using the weaker fractions for condensing the hydrochloric acid used in the process, and the stronger fractions, along with air, for the conversion of the hyponitric acid set free in the decomposition of the residual “nitrate of manganese sesquioxide” with steam or air.—H. A.

*Improvements in the Manufacture or Production of Cyanide Compounds from Ferro-Cyanide Compounds.* E. Bergmann, Hamburg, Germany. Eng. Pat. 4095, March 15, 1890. 6d.

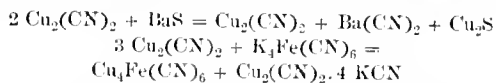
THIS invention effects (1) the manufacture of cyanides by the action of silver or copper salts in dilute solution on ferrocyanides, which are dissolved or suspended in boiling water. The silver or copper taking part in the reaction should be sufficient to combine with the whole of the cyanogen present; and a small quantity of free acid is requisite to further the reaction—



When eupric salts are used, it is necessary to add a reducing agent, such as sulphites,  $\text{SO}_2$ , &c. to promote the reaction—



(2) the cyanide of silver or copper obtained by any of these reactions is available for the manufacture of double salts, in digesting them with sulphides of the alkalis or alkaline earthy metals, or with an alkaline ferrocyanide—



(3.) In the former reaction the solution of copper-barium cyanide can be utilised for the manufacture of other double salts. The copper ferrocyanide produced in the latter equation is available for manufacturing new quantities of cyanide of copper, as given above.—H. A.

*A New or Improved Process for Manufacturing Bleaching Powder and Caustic Soda.* J. D. Pennock and J. A. Bradburn, Syracuse, U.S.A. Eng. Pat. 8090, May 23, 1890. 4d.

THE object of this invention is the regeneration of the nitric acid employed in manufacturing chlorine, and the utilisation of the nitrate of soda obtained therein for the manufacture of caustic soda.

Salt and nitric acid are introduced in a still, and on heating the whole of the chlorine of the salt is given off as a mixture of nitrosyl chloride and chlorine, which is converted into

pure chlorine by passing it through nitric acid, holding dioxide of manganese in suspension; nitrate of manganese is obtained thereby as a by-product.

The hot still liquor, consisting of a saturated solution of nitrate of soda, undecomposed salt, and nitric acid, is run off: half of the nitre is deposited on cooling, and the mother-liquor used over again for a new batch. The nitre so obtained is available for the manufacture of caustic soda, for which purpose it is furnaceed at a moderate red heat with 2—3 times its weight of oxide of iron in a current of air. In lixiviating the resulting mass with hot water, a solution of caustic soda of a density of 50°—77° Tw. is obtained.

The nitric acid introduced in the process is regenerated by passing the hot waste gases from the revolver first over an oxidising substance ( $MnO_2$ , manganites, manganates, permanganates), and afterwards over the nitrate of manganese solution obtained in the washing of the chlorine, which is evaporated down to a plastic condition, when  $MnO_2$  will remain, and the nitrous compounds are condensed as nitric acid after the usual treatment with air and steam.—H. A.

*Improvements in Apparatus for the Condensation of Nitric Acid and other Distillation Products.* O. Guttman, London. Eng. Pat. 8915, June 9, 1890. 6d.

THE condensation is effected by passing the vapours through one or more series of vertical earthenware pipes, connected on top and bottom by means of bends, the bottom bends being provided with traps and branch pipes, which lead in one main and a common receiver. The acid is condensed by surface cooling and collected continuously from each bend. The undecomposed acid passes from the last earthenware pipe through a coke tower, as usual.—H. A.

*Improvements relating to the Manufacture of Acid Phosphates for use as Artificial Manure.* H. H. Lake, London. From A. Memminger, Charleston, U.S.A. Eng. Pat. 9129, June 12, 1890. 4d.

IN the usual way of manufacturing superphosphates, the finely-ground phosphatic rock is mixed with the requisite quantity of sulphuric acid of 50° B. and allowed to dry. The duration of the drying operation depends greatly on the quality of the rock, and the object of this invention is to reduce the time of drying and thus to make phosphates of any origin suitable for the manufacture of superphosphates.

This is effected by an addition of about 5 per cent. or more of fluor spar to the rock, grinding the two materials together to pass through a 40-mesh sieve.—H. A.

*Improvements in the Packing or Putting-up of Chloride of Lime and other Substances.* T. C. Steele, Glasgow. Eng. Pat. 9670, June 21, 1890. 4d.

THE package is made of paper, cardboard, or thin wood, and rendered gas- and water-tight by dipping it in molten wax, or a mixture of the same with rosin, pitch, gums, or drying oils.—H. A.

## VIII.—GLASS, POTTERY, AND EARTHENWARE.

*Alteration of the Coefficient of Compressibility of Glass at different Temperatures.* E. H. Amagat. Compt. Rend. 110, 1246—1249.

THE author has been led to determine the coefficient of compressibility of both ordinary soda- and lead-glasses at various temperatures by the need for accurate knowledge of possible alterations, as demonstrated by his own researches, and the conflicting statements current on the subject. The problem was attacked by observing the alterations in volume of a long glass cylinder subjected to high pressure at known temperatures. Among the difficulties encountered was the tendency of the cylinder to act as an ultra-sensitive thermometer, if made large, so as to give measurable readings, while the use of a small cylinder necessitated such high pressures that permanent deformation was apt to result. The instrument adopted consisted of what was practically a piezometer enclosed in a steel cylinder filled with mercury, which was jacketed, so that the intermediate annular space could be filled with ice, steam, or the vapour of methyl benzoate (boiling point 200° C.), according to the temperature needed. The readings of the instrument were regulated so that they ranged over a comparatively short scale (to avoid error due to imperfect calibration), which was kept at a constant temperature by a water jacket.

The following table shows the results obtained; the numbers are arbitrary, representing divisions on the scale.

Temperature.	Piezometer No. 1. (Glass.)		Piezometer No. 2. (Glass.)		Piezometer No. 3. (Flint Glass.)	
	Divisions on the Scale.	Alteration per 100 Divisions.	Divisions on the Scale.	Alteration per 100 Divisions.	Divisions on the Scale.	Alteration per 100 Divisions.
0	202.7	2.95	205.7	2.71	182.8	4.10
100	208.7		211.3		190.3	
200	215.5		218.6		203.0	

The increase in the variation with rise of temperature is here manifest; it is quite appreciable for ordinary glass and distinctly greater for flint glass. It is in all cases somewhat higher than the value arrived at by Guillaume in his work on thermometers of precision, but the instruments he used were of hard glass. Although the existence of this variation

is thus well established, it is in any case extremely small, and would generally be negligible. Thus, the error experienced in estimating a volume at 200° C. under a pressure of 1,000 atmospheres would be only 0.00028 if this correction were ignored.—B. B.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

*Manufacture of Slag Cement.* J. Grosclaude. Dingl. Polyt. J. 275, 433—445.

THE trade in slag cement has recently grown considerably and its manufacture is now practised both in England and abroad. Besides being sold under its proper name it sometimes come into the market under the inappropriate titles of puzzolana and Portland cements. In the first place it is necessary to distinguish it sharply both from Portland cement proper, and from untreated slag often used as a sophistication for hydraulic cements. Slag cement is of the puzzolana order, *i.e.*, it is a mixture of active siliceous matter and lime which unite and set on the addition of water; the active siliceous matter is ordinary blast-furnace slag granulated and ground to bring it into a suitable condition and the lime is the ordinary substance carefully slaked. Taking the raw materials and their treatment systematically we have:—

(1.) *Granulation of the Slag.*—This is effected by quenching the molten slag with water and is absolutely essential for the production of a cement of any value; the precise change is not well understood. It may be regarded as analogous to the change produced in sulphur by fusing and quenching it where an altered molecular condition is preserved by sudden cooling; in this case the less complex molecule formed at a higher temperature would be prevented

from aggregating as it would were the cooling slow and retained in the same state when cold. A contrary hypothesis may be advanced to the effect that combinations existing at the higher temperature are decomposed by quenching and thus resolve themselves into simpler substances. In either case there is little doubt that granulation produces a state of greater molecular simplicity and activity, rendering the double silicate of lime and alumina constituting blast-furnace slag, active and capable of playing the same part that puzzolana does in cements made from that substance.

(2.) *Composition of the Slag.*—Besides its molecular condition, the ultimate composition of the slag is of great importance. Experience has shown that the oxygen of the silica, together with that of the alumina, should have to that of the lime approximately the relation 2:1. *Tetmajer*, who has worked considerably at the subject, is of opinion that a slag in which the relation of lime to silica is lower than unity—

$$\left( \frac{\text{CaO}}{\text{SiO}_2} < 1 \right)$$

is not available for cement making. He fixes the best composition as: lime 46, silica 30, alumina 16. Certain slags contain a notable proportion of calcium sulphide. Some of this is decomposed on granulation, but enough remains as a rule to give the cement a greenish colour due to the trace of iron present when allowed to set in water or damp air. It does not appear, however, that any harmful action can be ascribed thereto. The following table exhibits the composition of a few typical slags:—

—	Marnaval.	Saulnes.	Choizeux.	Hartzburg.	Middlesbrough.	Bilbao.
CaO .....	48.00	47.20	45.11	48.59	32.26	47.30
SiO <sub>2</sub> .....	30.50	31.65	26.88	30.72	31.65	32.90
Al <sub>2</sub> O <sub>3</sub> .....	19.50	17.00	24.12	16.40	25.30	13.25
FeO .....	0.85	0.65	0.44	0.43	0.10	0.46
MgO .....	0.75	1.36	1.09	1.28	3.54	1.37
CaS .....	..	..	1.86	2.16	1.42	3.42
MnO .....	0.40	0.85	0.50	Trace	0.36	1.13
Undetermined .....	..	1.29	..	0.42	5.37	0.17
	100.00	100.00	100.00	100.00	100.00	100.00

(3.) *Slaking the Lime.*—The common plan is to dip the lime contained in baskets bodily into water, but a preferable method is to use a special apparatus consisting of cylindrical vessels, in which the lime in pieces about the size of an egg is placed, enclosed in an outer cylindrical case fitted with a pressure gauge and safety valve. The whole being closed, water is admitted by sprinklers and the slaking goes on regularly, the lime being exposed to an evenly damp atmosphere, while at the same time local excess of water is avoided inasmuch as any volatilised during slaking falls back into the space between the vessel containing the lime and the outer cylinder, and does not drop back direct on to the lime itself. A pressure of 5—6 atmospheres is generally reached.

(4.) *Drying the Granulated Slag.*—Several methods have been proposed; the most obvious, *viz.*, exposing the slag, spread out in layers 6—7 cm. deep to flue gases, permits an output of 100 kilos. per sq. metre in 24 hours, with an expenditure of 6—7 kilos. per centner of dried slag.

*Ruelle* has devised an ingenious arrangement consisting of a long slightly tapering drum, capable of being rotated, within which is a screw by which the slag is carried from the end where it is fed into that nearer the furnace, the drying being thus systematic. Surrounding this drum is another coned in the reverse direction, and also provided with a screw, so that the dried and heated slag is gradually

carried back to the end whence it started, imparting its heat to the inner drum during its passage.

Such an apparatus will turn out about 25 tons of dried slag in 24 hours, with a coal consumption of 6 kilos. per 100 kilos. of dried product, and an expenditure of 6—7 horse power.

*Raty* has made an attempt, worth recording, to dispense with the necessity for such drying apparatus. He quenches the slag, with precaution, so as to avoid leaving any part semi-fused or plastic, while at the same time as nearly as possible the whole of the water is volatilised. Any final drying necessary is effected by passing molten slag through a pipe buried in a heap of the granulated product.

(5.) *Grinding the Granulated Slag.*—This is effected in an ordinary edge-runner, which, though rather wasteful of power, is best fitted for this particular purpose.

(6.) *Mixing the Slag and Lime.*—A mill, consisting of a cylinder with its inner surface shaped into a series of depressions and elevations and containing a number of iron balls, is used. The mingling is very perfect, while the fineness of the finished product may be gathered from the fact that it leaves a residue of 8—10 per cent. on a sieve of 5,000 meshes per sq. cm.; on the attainment of this degree of fineness, much of the quality of the cement depends.

The lime used is generally an ordinary "fat" lime, but it seems likely that a poorer lime would be better, there being less risk of the cement cracking if used in air.

It sometimes happens owing to the composition of the slag being not quite suitable that cement made from it sets badly and shows cracks and a tendency to disintegrate.

These evils may be overcome by mixing a little precipitated silica and alumina with it, the former being prepared by treating a portion of the slag with hydrochloric acid, while the latter may be obtained by precipitating the filtrate from the silica with lime.

(7.) *Strength of Slag Cement.*—The results about to be given are taken from the publications of the *Écoles des Ponts et Chaussées* (No. 3405, September 7, 1888), and refer to a cement made at the Donjeux factory from Marnaval slag and "fat" lime. The composition of the cement was:—

Sand .....	0.25
Silica .....	23.85
Alumina .....	13.95
Ferric oxide .....	1.10
Lime .....	51.40
Magnesia .....	1.95
Sulphuric anhydride .....	0.45
Loss on ignition .....	7.05
	<hr/> 100.00

When sifted it left on a sieve of 324 meshes per sq. cm., 0.7 per cent. residue, on 900 meshes 0.8 per cent., and on 5,000 meshes 21.5 per cent., the total residue amounting to 23 per cent. When gauged with 28 per cent. of water, it began to set in  $1\frac{1}{4}$  hours, and setting was complete at the end of 3 hours.

The following table shows the mean results of the mechanical tests:—

	In Tension.			In Compression.		
	Kilos. per sq. cm.			Kilos. per sq. cm.		
	7 Days.	28 Days.	84 Days.	7 Days.	28 Days.	84 Days.
Neat cement .....	21.87	26.88	31.15	275.7	377.3	462.3
Cement + Sand .....	14.93	26.03	29.23	180.0	260.2	319.2
(? 1:3)						

It must be admitted that this is about the best sample, the behaviour of which is recorded in the paper above mentioned, but it serves to show what quality may be attained. It has been alleged that slag cement fails when used for sea work, but it has been used for harbour work both at Bremen and Middlesbrough.

In judging a slag cement from the analytical data it affords, it must be remembered that the criteria of excellence that are in vogue for Portland cement do not apply to it.

Thus a good Portland cement of low specific gravity is almost a contradiction in terms, whereas a good slag cement may have quite a small density.

A detailed statement of costs for erecting the necessary plant and carrying out the manufacture are given in the original.—B. B.

## X.—METALLURGY.

*The Dry Assay of Tin Ores.* Part 1. H. O. Hofman. *Technical Quarterly*, 3, 112—143.

See under XXIII., page 899.

*The Estimation of Phosphorus in the "Basic" Siemens Steel Bath.* W. Galbraith. Paper read before the Iron and Steel Institute, May 1890.

It is usual to judge of the extent to which the oxidation of the phosphorus has proceeded by withdrawing a sample, hammering down, quenching in water and examining the fracture, which should be bright and crystalline and have a characteristic streaky appearance if the phosphorus be present in quantity. The appearance is apt to vary, however, with the size of the sample, the amount of hammering and the temperature at which it is quenched. The following chemical test is rapid and more reliable: 0.20 grm. of the drillings are dissolved in 3 cc. of aqua regia in a small flask, 3 cc. of water are added and enough ammonia to precipitate the whole of the iron. The precipitate is next dissolved in nitric acid added drop by drop until the liquid is distinctly clear. 10 drops of ammonium molybdate, prepared according to Fresenius, are then added, the liquid is transferred to a small bottle 2 in. high by  $\frac{1}{2}$  in. in diameter, diluted to a mark on the bottle and well shaken. Similar operations conducted with a standard steel containing say .05 per cent. of phosphorus, and the opacities were compared. The comparison becomes more simple if a series of bottles be prepared containing from .01 per cent. to .10 per cent. of phosphorus. The molybdic solution is made by dissolving 50 grms. of molybdic acid in a mixture of 100 cc. of ammonia and 100 cc. of water and pouring the liquid into 750 cc. of nitric acid (sp. gr. 1.20). It is not advisable to arrest a basic Bessemer blow sufficiently long to allow of this test being made, but the analysis may easily be performed between each blow and any phosphoric heat detected.—H. K. T.

*Aluminium in Carburetted Iron.* W. J. Keep. Paper read before the Iron and Steel Institute, May 1890.

THE author first describes the method used by him in making mechanical tests, he then gives the effect of aluminium on various kinds of iron and steel. With commercially pure iron in the form of Robert-Bessemer ingots, the addition of 0.25 per cent. of aluminium diminishes deflection by decreasing the set and elasticity. Rigidity is increased, the grain is closer and more uniform, and the metal is improved as a structural material. Remelted wrought iron containing 2.45 per cent. of aluminium makes a good weld and after being forged can be bent nearly double. Its grain is like that of fine steel. Wrought iron with the above percentage of aluminium can be easily cast. With a higher percentage of carbon, as in steels, aluminium slightly increases the hardness, owing to some of the graphite being expelled, since, with low percentages of carbon, aluminium does not convert the combined carbon into graphite, but expels some of the existing graphite. White cast iron is greatly improved by aluminium since the carbon is reduced in amount and part of it is converted into graphite, thus lessening the hardness of the metal and increasing its rigidity and elasticity. The author points out that if the graphite of grey cast iron could be produced by aluminium instead of by silicon the metal would be better for most purposes. Even at the present price of aluminium the addition is generally advantageous (compare this *Journal* 1888, 752).—H. K. T.

*Certain Chemical Phenomena in the Manufacture of Steel.* W. Galbraith. Paper read before the Iron and Steel Institute, May 1890.

IN the manufacture of soft steel either by the Bessemer or Siemens process, especially where a basic lining is used, there is often experienced a difficulty in keeping the metal

quiet in the mould. This is usually accompanied by a difficulty in teeming, a considerable quantity of metal being left in the ladle. The author considers that these difficulties are due to oxide of iron disseminated through the metal, especially as they are accompanied by a metallic-looking slag high in iron, by a large evolution of gas, and by an excessive consumption of spiegeleisen or ferro-manganese before the carbon can be increased. In support of this view it frequently happens that metal will teem badly whilst showing nothing abnormal on ordinary analysis, whilst on the other hand the effect of an oxidising slag in rendering steel infusible is well known. In further support of his view the author quotes Müller's experiments on the secretion of gases in iron and steel (Iron, September 14, 1883), in which it was found that basic steels contain an abnormal amount of carbonic oxide, the latter being produced at the expense of the oxides in the metal when the oxidising slag was removed. Another effect of a slag containing much oxide of iron is that the iron is oxidised more rapidly in proportion to the amount of phosphorus removed. In order to avoid the injurious effect of the basic slag on the spiegeleisen and to obtain a better control of the carbon in the metal, the steel after teeming is run into a second ladle until the basic slag begins to come, when the stream is stopped, at the same time some siliceous slag, similar to ordinary Siemens slag, together with the necessary alloys, is added.—H. K. T.

#### *Steel Rails considered Chemically and Mechanically.*

C. P. Sandberg. Proc. Inst. Mech. Eng. July 29, 1890. Industries, 9, 97—101.

The subject is discussed and tabulated analytical data given, under the following headings:—

Safety and durability.

Favourable effect of silicon.

Experiments on high silicon steel rails.

Hardness dependent on carbon and phosphorus.

Heavier and harder rails for safety.

The remaining headings have no direct connexion with applied chemistry.

*The Critical Points of Iron and Steel.* F. Osmond. The Iron and Steel Institute, May 1889. (See also this Journal, 1890, 517—518.)

The paper dealt with experiments carried out for the examination of the rate of cooling of samples of iron and steel of different composition with a view to determine their critical points.

The samples operated upon were in the form of bars, into one end of which one of the junctions of Le Chatelier's thermo-electric pyrometer (Journal de Physique, VI, Jan. 187) was soldered. The couple of the latter was composed of a cast platinum and a platinum-iridium wire, and the opposite ends were connected with a Deprez and D'Arsonval aperiodic mirror galvanometer.

The pyrometer was calibrated by means of the melting point of potassium sulphate (1,015°) and the boiling point of ammonium chloride (340°), and it was assumed that the temperatures, up to 1,500°, were proportional to the electromotive forces of the couple. In experimenting, the metallic bar, with wires attached, was placed in a porcelain tube closed with corks and heated to the required temperature. The furnace and tube were then allowed to cool spontaneously and the times at which the index passed each division of the scale were recorded by means of a chronograph. The temperatures were then plotted as abscissæ, and the times included between the passage of the index over two consecutive divisions of the scale as ordinates. The different critical points are indicated by the letters  $a_1$ ,  $a_2$ ,  $a_3$ , &c. —  $a_1$  representing the lowest temperature.

The first experiments were made on samples of iron and steel with increasing proportions of carbon, the other constituents remaining, as far as possible, constant. The following table gives the nature and composition of the different samples:—

	Electrolytic Iron.	Extra Mild Steel, basic Open-Hearth.	Mild Steel, basic Bessemer.	Fairly Mild Steel, acid Open-Hearth.	Hard Crucible Steel.	Swedish White Pig Iron.
Carbon.....	0.08	0.160	0.290	0.570	1.250	4.100
Silicon.....	..	0.012	0.060	0.085	0.190	0.220
Sulphur.....	..	0.021	0.060	0.020	0.010	0.040
Phosphorus.....	..	0.029	0.052	0.050	0.021	0.018
Manganese.....	..	0.110	0.270	0.230	0.100	0.120

The following arrests were obtained:—

	$a_3$ .	$a_2$ .	$a_1$ .
Electrolytic iron..	885° (abrupt and very long).	750°—690° (very gradual).	660° (almost imperceptible).
Extra mild steel..	845°—800° (gradual).	755°—710° (gradual).	680°—645° (gradual).
Mild steel.....	780°—690°		680°—640°
Fairly mild steel..	750°—661°		661°
Hard steel.....	860° (very slight).	..	674° (very long).
White pig iron....	..	..	695° (short).

The above results show that the delay in cooling  $a_1$ , which is almost imperceptible in electrolytic iron, goes on increasing with the proportion of carbon until it assumes enormous proportions in hard steel. Thus it is a function of the proportion of carbon, and is identical with the phenomenon discovered by Barrett in 1873, and named by him "recalcence."

The station  $a_1$ , which increases in duration with the proportion of carbon in steels, diminishes on passing from

hard steel to white pig iron, hence there is little change of state at this point, and the carbon at all temperatures is either in the form of hardening carbon or of cement carbon. With regard to the other points of rest in electrolytic iron, there is a sudden arrest,  $a_3$  at 855°, and a more gradual one,  $a_2$ , at 750°—690°. In extra-mild steel the same stations are observed,  $a_2$  remaining the same,  $a_3$  being somewhat lowered. In mild steel,  $a_3$  is still lower, and becomes confounded with  $a_2$ , so that there is a single gradual slackening,  $a_3-2$ . In medium steel, the point  $a_3-2$  continues to descend and joins on to  $a_1$ . Finally, in hard steel, the three critical points are merged into one, and  $a_3-2-1$  at 674°.

The point  $a_3$ , since it is most distinct in pure iron, probably represents a change of molecular state of the element, the change being analogous to that which is known to take place in the case of sulphur, silicon, &c. The point  $a_2$  may represent a distinct molecular modification of iron, but as it is not sharply defined, even in electrolytic iron, the author preferred to regard it provisionally as the end of  $a_3$ . Thus, below  $a_2$ , iron possesses the molecular form  $\alpha$ , whilst above  $a_3$  it assumes the allotropic form  $\beta$ . At intermediate stations it is a mixture of  $\alpha$  and  $\beta$ . The action of carbon on iron can now be easily summarised. Carbon, in the form of hardening carbon, maintains iron in the  $\beta$  condition, during slow cooling, down to a temperature which is lower

the greater the quantity of carbon present. The points  $a_3$ ,  $a_2$ , and  $a_1$  correspond with a number of changes already observed in the physical properties of iron, such as recalcence, variation in specific heats, appearance and disappearance of magnetism, and changes in rate of dilatation and electrical resistance.

**Influence of the Initial Temperature and of the Rate of Cooling on the Position of the Critical Points. Theory of Hardening.**—If samples be cooled from successively higher temperatures it is found that the temperature of recalcence is proportionally lowered. The temperature of recalcence is also lowered the more rapid the cooling. If the sample is cooled in water no retardation is observed. Thus, if the cooling is fairly rapid the changes produced during slow cooling do not take place, and the hardened steel consists of iron and carbon in the state in which they existed at high temperatures. In order to see if the heat which had not been evolved was really latent in the steel, equal weights of annealed and hardened steel were dissolved in the double copper ammonium chloride. The following rises in temperature were obtained:—

	Annealed.	Hardened.
Medium steel .....	2°366	2°477
Hard steel .....	2°148	2°369
White pig iron .....	1°665	1°878

Hence the hardened metal contains an excess of heat over the annealed one. The influence of time on the change of state during cooling is in accord with general chemistry.

With regard to the share of iron and carbon in communicating the usual properties of hardened steel to the metal, the author considered that they are mainly due to the iron and that hardened steel contains the iron in the  $\beta$  condition. In order to prove this, steel was hardened at a temperature between  $a_3-2$  and  $a_1$  that is, at a temperature at which the change (during cooling) of iron from the  $\beta$  state to the  $\alpha$  is well advanced, whilst the combination of the carbon with the iron has not yet commenced. The metal so obtained was soft under the file, yet when treated with nitric acid it gave the reaction for hardening carbon. On the other hand mild steel may be hardened by heating to a temperature above  $a_3$ , and rendering the cooling from this point extremely rapid by plunging the metal into a freezing mixture. Under these circumstances the tensile strength increases from 26·6 tons to 61·5 tons per square inch, the elongation decreasing from 30 to 4 per cent. This enormous diminution in malleability could not be due to the ·22 per cent. of carbon which the steel contained, but is due to the iron being in the  $\beta$  condition. The influence of carbon is the same as that of rapid cooling, and hinders the conversion of iron from the  $\beta$  to the  $\alpha$  condition.

**Phenomena observed on heating the same samples between 600° and 1,100°.**—On heating samples of iron and steel the same critical points were observed as on cooling, but they were uniformly lower. The difference in  $a_3$  for heating and cooling is least in electrolytic iron. These considerations explain the fact observed by Brinell that the temperature to which steel must be heated to enable it to be hardened and the temperature at which it suffices to cool it, are not identical.

**Heating and cooling below 600°. Theory of Tempering.**—Annealed steel does not show any critical points below 600°. Hardened steel on heating begins to evolve heat at 200°, so that there is an acceleration in the rate of heating when this temperature is reached. The maximum liberation of heat is reached at 350°, and it ceases at 520°. A further evolution of heat occurs at 660°—680°. Hence the iron and carbon in hardened steel are in a state of unstable equilibrium similar to that of plastic sulphur, and during tempering the  $\beta$  iron returns to the  $\alpha$  state and the hardening (uncombined) carbon to the condition of iron carbide.

**Influence of Foreign Metals. Boron.**—A sample from Roberts-Austen was examined. There were two liberations of heat, viz.,  $\frac{a}{m} r_3$  of which the maximum is produced between 815° and 805°;  $a r_2 + \frac{(m-1)a}{m} r_3$  of which the maximum is produced between 738° and 725°, each station being lowered. The two liberations of heat are fairly equal. This action is analogous to that of carbon.

**Nickel.**—The sample showed a single station,  $a_3-2-1$ , at 650°. In a steel containing the same amount of carbon,  $a_1$  would be quite distinct; thus, the nickel has considerably lowered the charge of iron and of carbon.

**Manganese.**—The temperature of the allotropic change of iron and of recalcence is lowered in proportion as the percentage of manganese is increased. Ferro-manganese only shows traces of arrestation. Thus manganese has the same effect as carbon in maintaining the iron in the  $\beta$  condition. In ferro-manganese and highly manganiferous steels all the iron is in the  $\beta$  condition, a condition similar to that of iron at a temperature above 860°. This explains why these alloys are not magnetic, since iron above 860° cannot be magnetised.

**Copper.**—The samples were prepared by Dr. Edwin J. Ball and Mr. Arthur Wingham (Jour. Iron and Steel Inst. 1889, 123). With 0·847 per cent. of copper the three points,  $a_3$ ,  $a_2$ , and  $a_1$  are quite distinct, but  $a_3$  is lowered some 40 or 50 degrees. With a greater proportion of copper,  $a_3$  becomes confounded with  $a_2$ , and  $a_1$  descends. The effect therefore of copper is the same as that of the previous metals but it is not so energetic, 4 per cent. of copper having the same effect as 0·2 per cent. of carbon.

**Chromium.**—Three samples were examined with 1·0, 2·0, and 10—12 per cent. of chromium. With the first the maximum  $a_3-2$  is slightly higher than with steel containing the same amount of carbon. The remaining two samples, cooled from 1,100°, show only one perturbation  $a_3-2-1$  in the form of a highly accentuated recalcence. The third sample cooled from 800°; recalcenes at the exceptionally high temperature of 744°. Hence it appears that the effort of chromium is to raise the critical point  $a_1$  and its action is of an opposite nature to that of manganese, and is equivalent to annealing. If chromium steels unite in a remarkable manner certain opposite properties and are but slightly brittle in proportion to their hardness, we must assume that their hardness is due to the chromium itself, and their consistency to the absence of  $\beta$  iron. The influence of the initial temperature on the portion of  $a_1$  is greater in chrome steels than in carbon steels.

**Tungsten, 0·26 to 1·53 per cent.**—The points  $a_3$  and  $a_2$  retain their original positions, but  $a_1$  is lowered to 530°. Thus tungsten has no action on the change of iron per se, but it has a hardening action through the agency of the carbon which it has a tendency to retain in the form of hardening carbon. This action is peculiar to tungsten. The lowering of  $a_1$  only takes place when the initial temperature is sufficiently high. If cooled from 750°  $a_1$  appears at 670°.

**Silicon.**—The samples were supplied by Mr. Hadfield, and consisted of an extended series in which the silicon was the only variable (Iron and Steel Institute, September 1889). With increase of silicon  $a_3$  is raised and approaches its position in pure iron. It eventually disappears altogether and is not found even when the metal is heated to 1,400°. Hence the effect of silicon is to keep the iron in the  $\alpha$  condition.

**Arsenic.**—The effect of this element is similar to that of silicon in tending to raise  $a_3$  and to diminish its intensity;  $a_2$  and  $a_1$  are not affected.

**Phosphorus.**—Two samples were examined, a sample of puddled iron ( $P=0\cdot38$  per cent.) and one of basic Bessemer metal (before afterblow,  $P=1\cdot35$  per cent.). The curve for the puddled iron is exactly similar to that of electrolytic iron,  $a_3$  being gradual and not abrupt. Probably in this sample the phosphorus was chiefly in the form of phosphate

of iron. The Bessemer metal gave only one very indistinct point ( $a_1$ ) at  $680^\circ$ . The effect of phosphorus is therefore similar to that of silicon.

**Sulphur.**—A single sample of basic Bessemer steel (S=0.28 per cent.) was examined and its curve compared with that for a steel containing the same amount of carbon and manganese. The stations  $a_{3-2}$  and  $a_1$  were found to be raised. The action of sulphur appears to be similar to that of silicon and phosphorus.

**General Conclusions on the Influence of Foreign Elements.**—Roberts-Austen has found that the effect of foreign elements on the properties of gold is related to the periodic classification (Phil. Trans. 179, 339–349). The author finds that a similar relation holds in the case of steel. Thus in general those elements the atomic volumes of which are less than that of iron, delay during cooling, the change of  $\beta$  iron (hard) into  $\alpha$  iron (soft), and that of hardening carbon into carbide. They therefore have a hardening action. (To this class belongs hydrogen, since electrolytic iron is very hard and brittle.) On the other hand, those elements the atomic volumes of which are greater than that of iron, tend to raise the change of the iron to the normal position which it occupies in pure iron. They hasten during cooling the combination of the carbon with the iron, and retard the inverse change during heating. They therefore communicate softness and malleability. In other words, elements of low atomic volume tend to maintain the iron in the form in which it has itself the lowest atomic volume, and conversely. The magnetic properties of iron are subject to the same influence, those elements the atomic volumes of which are less than that of iron increasing the permanent magnetism of the iron, whilst those the atomic volumes of which are greater, have no action on the permanent magnetism.—H. K. T.

#### PATENTS.

*Improvements in the Treatment of Refractory Gold and Silver Ores and in Apparatus therefor.* H. Hutchinson, London. Eng. Pat. 16,270, November 26, 1887. (Second Edition.) 8d.

Previously described (this Journal, 1889, 287).—A. W.

*Improvements in Coating or Plating Metal Plates or Articles of Large or Small Size.* W. P. Thompson, Liverpool. From A. A. L. Levy, Paris, France. Eng. Pat. 12,556, August 8, 1889. 6d.

This is a claim for the coating of any metal with another metal by simple immersion of the former in a solution of the chloride of the latter mixed with chloride of ammonium. The presence of the latter salt is the "essential foundation" of the process.—A. W.

*Improvements in Treating Liquid Acid Residues containing Iron to obtain Products therefrom.* M. N. d'Andria, Manchester. Eng. Pat. 13,575, August 28, 1889. 4d.

See under VII., page 860.

*An Improved Process for Recovering Valuable Substances from the Mother-Liquors Resulting from Treatment of Copper Ores.* E. de Cuyper, Perennes-lez-Binche, Belgium. Eng. Pat. 13,726, August 30, 1889. 8d.

See under VII., page 860.

*An Improvement in Foundry Sand.* J. Patriek, Frankfurt-on-Main, Germany. Eng. Pat. 14,408, September 12, 1889. 4d.

The patentee mixes with the sand, coal-tar, asphalt, bitumen, or a similar viscous body. Five parts of tar are liquefied by

heat, stirred into five parts of hot water and the mixture poured on to 100 parts of sand. It is maintained that by this means sand which is either too fine or too coarse for cores or moulds is rendered suitable, the former by having its porosity increased and the latter by an increase in its density and cohesion.—A. W.

*Improvements in Metallurgical Furnaces.* C. A. Piat, Paris, France. Eng. Pat. 18,985, November 26, 1889. 8d.

This is an arrangement in which a small cupola is placed over a crucible furnace in such a way that the waste gases from the latter can be utilised in assisting the heating of the former. Neither furnace is a fixture. The cupola is adapted to a hollow bearing, through which the air is supplied, in such a manner that it can be slightly raised from the crucible furnace and swung round out of the way, while the latter, which is held on one end of a counter-balanced rocking beam, is raised to discharge its contents. The cupola is so arranged that it can be used by itself simply as a cupola or in conjunction with the crucible furnace.—A. W.

*Improved Manufacture of Compositions or Alloys for Anti-Friction Purposes.* E. C. Miller, London. From The Magnolia Anti-Friction Metal Co., New York, U.S.A. Eng. Pat. 8655, June 4, 1890. 4d.

The ingredients are the following:—

Lead.....	80 lb.
Antimony.....	15 "
Tin.....	5 "
Bismuth.....	4 ounces.
Graphite.....	8 "

and according to the desired quality of the alloy—

Silver.....	4 ounces.
or Aluminium.....	4 "

The lead is melted and cleaned with sal-ammoniac, the antimony added, the graphite then stirred in and the tin and bismuth mixed in last. When desired silver or aluminium can be incorporated. The alloy is recommended for shaft bearings.—A. W.

## XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

*On the Electro-deposition of Platinum.* W. H. Wahl. J. Frank. Inst. 130, 1890, 62–75.

THE author points out the great demand that exists for a good and reliable process for producing a bright and adherent deposit of platinum, and mentions that, of all the methods that have been proposed, only three deserve special notice. These are: (1.) The Roseleur-Lanaux method, based on the electrolysis of a solution of the double phosphate of sodium and platinum. (2.) The process of the Bright Platinum Plating Company (London), a modification of that of Roseleur, involving the introduction into the bath of certain substances, such as sodium chloride and borax, to ensure a bright deposit of the metal; and (3.) Boettger's method, founded on the electrolysis of a solution of the double chloride of ammonium and platinum in sodium citrate. These processes give good results at the commencement, but the solutions deteriorate from a variety of causes, the chief among which is the insolubility of platinum as an anode, which necessitates occasional additions to the baths of fresh quantities of the platinum salt, whereby the electrolytes are continually altering in conductivity, and gradually becoming contaminated with secondary products. The character of the deposit of platinum is naturally impoverished by such alterations in the conditions.



All the solutions recommended for electro-plating, except Boettger's, are made by treating the platonic chloride with alkaline salts, the most favoured of which are the phosphates and oxalates. The result is a solution of the double phosphate or oxalate, as the case may be, together with the chloride of the alkali from the decomposition of the platonic chloride. As the solution becomes impoverished it is strengthened by fresh additions of platonic chloride, with the result that the alkaline chloride accumulates, until the bath is practically spoilt. Boettger maintains his bath by fresh additions of the original solutions, but here again the accumulation of foreign substances must follow. The author then describes his own experiments on the subject with a view to remedy these defects.

Platinum black deposited on carbon was used as an anode, in the hope that a large surface would facilitate the solution of the metal, but it was found to be impractical, as the solution had to be strongly acid, and the current of such a strength that only a black and non-adherent deposit was obtained. Again, it was quite useless with those solutions which give the best deposits, that is, the phosphates and oxalates. A solution of platonic hydrate in caustic potash was found to give a good deposit of metal, and the bath could be kept up to standard by additions of the platonic hydrate without any deterioration due to the accumulation of foreign salts. The author recommends the use of platonic hydrate in the preparation, and the maintenance of the strength of the ordinary solutions in use, and gives the following directions:—

#### *The Alkaline Platinate Solution.*

Platonic hydrate.....	2 oz.
Caustic potash .....	8 oz.
Distilled water .....	1 gall.

One-half of the caustic potash is dissolved in a quart of water and the platonic hydrate gradually added; when solution is effected, the remainder of the caustic dissolved in another quart of water, is stirred in, and the solution made up to a gallon. A current of about 2 volts is the best, and there should be only a slight, if any, evolution of hydrogen at the cathode, but a liberal one of oxygen at the anode. The solution may be worked at half the above strength. A little acetic acid improves the working of the bath when a heavy deposit is required. Articles of steel, nickel, tin, zinc, or German silver are preferably first thinly coated with copper in a hot cyanide bath.

The oxalate solution is prepared by dissolving 1 oz. of platonic hydrate in 4 oz. of oxalic acid, and diluting to one gallon. The best plan is to work with a saturated solution of the oxalate, keeping an undissolved excess always present. The addition of a small quantity of oxalic acid now and again is advantageous. The double oxalate may be prepared by saturating the alkaline oxalate with platonic hydrate, the strength of the bath being maintained by the presence of the single oxalate, as above. The deposits from these solutions are sensibly harder than that obtained with the alkaline bath, and will buff tolerably well.

#### *The Phosphate Solution.*

Phosphoric acid, syrupy (sp. gr. 1.7).....	8 oz.
Platonic hydrate .....	1-1½ oz.
Distilled water .....	1 gall.

The acid should be moderately dilute, and the solution of the hydrate effected at the boiling temperature, after which it is diluted to a gallon. The current in this case can be stronger than in the previous ones. The strength is maintained by additions of platonic hydrate. The double alkaline phosphates may be used, and are prepared by neutralising the above with the alkali, and then adding an excess of phosphoric acid. The deposit is described as brilliant and adherent, with the same steely appearance as with the oxalate, but to a less pronounced degree.

The author states that Professor Dudley has, quite independently, worked out, in a precisely similar manner, the problem of electro-plating with iridium. His work is not published, but he employed, in 1886, a bath made up of,

preferably, the double sulphate of iridium and ammonium, maintaining the strength by hydroxide of iridium suspended in canvas bags near or around the anodes.—A. W.

#### *The Electrolysis of Metallic Phosphates in Acid Solution.*

E. F. Smith. Amer. Chem. J. **12**, 329-336.

See under XXIII., page 898.

#### PATENTS.

##### *Improvements in Galvanic Batteries and in Electrolytic Baths.*

J. L. Roberts and H. L. Breyvoort, Brooklyn, U.S.A. Eng. Pat. 18,494, December 18, 1888. (Reprint.) 8d.

ALREADY appeared, this Journal, 1889, 623.—B. T.

##### *Improvements in Galvanic Batteries.*

A. Schanschieff, London. Eng. Pat. 7,005, April 26, 1889. 6d.

THE battery cell is made in the form of a drum (or box) by the rotation of which through a quarter of a revolution the electrodes can be either immersed in or withdrawn from the electrolyte.

One quarter of the drum is cut out by two *radial* planes at right angles; radial covers are then fitted on, to one of which the electrodes are fixed. When the electrodes are out of the liquid, they occupy one-third, and the liquid the remaining two thirds of the interior of the cell.—E. T.

##### *Improved Means for Supporting and Separating the Plates of Electric Accumulators.*

R. E. B. Crompton, London. Eng. Pat. 12,901, August 15, 1889. 6d.

A PAIR of notched double rails of suitable insulating material are used to support the plates in each cell, each double rail being made up of two single rails held apart at suitable intervals by short bars. Each rail is notched at regular intervals, and gaps are cut in the material between the notches; the single rails are then joined together by the before-mentioned bars in such a way that the notches of one are opposite the gaps of the other. All the positive plates will then rest in the notches of one rail, passing through the gaps of the attached rail and will rest in a similar manner in the notches of one of the rails opposite, while all the negative plates rest in the notches of the other two rails. A pair of supporting feet are provided for each pair of rails.—B. T.

##### *Electrolytic Treatment of Vegetable Matters and Cereals for the Production of Starch.*

E. Hermite, E. J. Paterson, and C. F. Cooper, London. Eng. Pat. 12,906, August 15, 1889. 4d.

See under XVI., page 878.

##### *Improvements in the Construction of Electric Accumulators.*

G. Downing, London. From J. F. Weyde and F. Clas, Cassioire, Hungary, and J. Eloner, Paris, France. Eng. Pat. 13,459, August 27, 1889. 1s. 3d.

THE active material is pressed into solid sticks or bars of any desired cross-section, and these bars are held firmly in plates of lead bent into a zigzag or corrugated tube form to make the electrodes. For large accumulators frames of wood, celluloid, porcelain, or other insulators may be coated with lead, and used to retain the active material. Various modes of suspension and connexion of the plates are described.—B. T.

##### *Improvements in Electric Glow Lamps.*

A. Berstein, London. Eng. Pat. 14,656, September 17, 1889. 6d.

CONDUCTING wires of springy nickel are used inside the lamp, and are so constructed as to very nearly approach one another at one part while they are held apart at one end by a glass distance-piece and at the other by the carbon rod. Should the carbon rod break, the springiness of the nickel conductors brings them into contact, so short-circuiting the lamp.—B. T.

*Improvements in or Relating to Secondary Batteries.*

P. J. R. Dujardin, Paris, France. Eng. Pat. 14,971, September 23, 1889. 6d.

In order to keep the plates of secondary cells at a uniform and unchangeable distance apart, even when the cells are moved about, the spaces between them are filled with "artificial white stone" broken into small pieces. This stone is prepared by compressing a paste of fine white sand and water, and burning in a kiln.

To regulate the potential difference in an incandescent circuit fed from accumulators, as the E.M.F. of the latter varies, a high resistance relay is placed across the mains, and according to the position of its tongue, causes one or other of two armatures to tremble like the armature of an electric bell, and so work a ratchet wheel, which causes resistance to be inserted into, or removed from, the circuit till the correct voltage is regained.—E. T.

*Improvements in Electrical Furnaces and their Manipulation.*

T. Parker, Wolverhampton. Eng. Pat. 17,060, October 29, 1889. 8d.

In this furnace the working electrodes, if of carbon, are held in metal sockets—kept cool if necessary by circulating water—at the required working distance. The arc is started by supplementary small electrodes inserted into the furnace temporarily. The electrodes may be kept in order by introducing fine or divided carbon on to their ends while the furnace is working. The potential difference between the ends of the carbons is regulated by a rheostat in the field magnet circuit of the dynamo, the carbons themselves not being moved.—E. T.

*An Improved Fluid for Primary Batteries.*

T. Coad, London. Eng. Pat. 1347, January 25, 1890. 4d.

"Nitric acid, mercury, carbonate of potash, carbonate of soda, bichromate of potash, sulphuric acid, common soda and water" are employed in varying proportions in this single fluid battery according to the nature of the work required.—B. T.

*Improvements in Electric Batteries Working with Two*

*Liquids or Electrolytes.* H. Weymerseh and R. McKenzie, London. Eng. Pat. 2302, February 12, 1890. 8d.

THE casing containing the cells of this two-fluid battery is provided with two channels, preferably in the bottom of the casing, one channel communicating with all the cells which are to contain one electrolyte, and the other channel with those which are to contain the other electrolyte; a gauge-glass and tap are provided at the outside end of each channel so that the cells can either be filled or emptied to any given level without taking the battery to pieces.

—B. T.

*An Improved Electrolytic Solution for Galvanic Batteries.*

L. Weigert, Berlin, Germany. Eng. Pat. 4949, March 29, 1890. 4d.

To prevent polarisation the inventor employs a solution of one or several chlorides such as zinc, copper, iron, &c. in combination either with a salt of mercury or with oxidising agents.—B. T.

*A New or Improved Storage Battery.*

A. J. Jarman, London. Eng. Pat. 6236, April 24, 1890. 8d.

PELLETS of an inactive partly soluble material are placed in position in a mould, and this mould is then filled with a suitable metallic alloy of lead and copper, or, aluminium and lead, or, aluminium and zinc, &c., to form the electrode, any desired form of terminal being provided for electrical connexion. Or long rods of the partly soluble material may be made in the form of screws, and a suitable alloy poured round them so as to form a solid block. Plates of

any desired thickness can then be cut off from this block, the cut being made at right angles to the length of the rods, the screw threads of which serve to retain the pellets in position. Several compounds, chiefly composed of lead salts, are described as suitable for the manufacture of the pellets and rods. The plates thus formed may be immersed in an electrolyte composed of an acidulated solution of alum, or of hyposulphite of soda with water and alcohol, or water alone, or acidulated sulphate of magnesium.—B. T.

*An Improved Method of Renewing the Carbon Filaments of Incandescence Electric Lamps.*

H. H. Lake, London. From La Société "L'Incandescence Electrique," Paris, France. Eng. Pat. 6581, April 29, 1890. 6d.

THE glass bulb of the lamp whose filament is to be repaired is opened at the top and the old filament removed, leaving only about 2 mm. of the old carbon upon the electrodes of platinum to serve as joining-pieces. A new filament is introduced and placed in such a position that its ends are in line and in contact with the carbon on the electrodes.

A suitable "hydrocarburet" is then introduced into the bulb so as to cover the joints, and a current of electricity passed through the joints decomposing the "hydrocarburet" and producing a deposit of carbon at the points of contact so as to form a strong joint.—B. T.

*An Improved Battery Element.*

J. N. Leysen, Amsterdam, Holland. Eng. Pat. 7383, May 12, 1890. 6d.

THIS is a modified form of Leclanché battery with the carbon and zinc placed in a loose sack filled with a mixture of retort carbon and manganese, sealed with resin and pitch, and the whole immersed in a solution of sal-ammoniac.

—B. T.

## XII.—FATS, OILS, AND SOAP MANUFACTURE.

*The Acidity of Vegetable Oils.*

Holde. Mitt. Konigl. Tech. Versuchs. 1890, 78—82.

See under XXIII., page 900.

### PATENTS.

*Ammoniated Soaps.*

C. R. Huxley, London. Eng. Pat. 11,817, July 25, 1889. 4d.

SOAP quite free from oleates is cut up in shavings and this is incorporated with gaseous ammonia by submitting the soap to the action of ammonia under a pressure of 15 lb. until every tissue of the soap is impregnated. The inventor claims that "its hygienic properties are such that it cleanses the pores of the skin which ordinary soap containing grease hermetically seals up."—K. M.

*A Manufacture of Linseed Mucns or Mucilage and the Preparation of Toilet and other Articles therefrom.*

A. Bosset, Paris, France. Eng. Pat. 13,535, August 27, 1889. 4d.

AQUEOUS extract of linseed is worked up in various ways into a variety of articles for toilet use. For example, the solution is mixed with rice starch and fecula, stirred well, passed through rollers and dried in the open air. It is pulverised, sifted, and scented as desired to form a toilet powder. From it are also prepared a shaving soap and a toilet liquid called "Lacto Neige."—A. J. K.

*Improvements in Apparatus for Purifying and Refining Oil.* E. Noppel, Philadelphia; B. Grosche and J. Bigler, New York, U.S.A. Eng. Pat. 5375, April 8, 1890. 8d.

In this apparatus the oil is heated by a steam coil and purified by settling and by repeatedly washing with water or other suitable liquid. For details of the apparatus the specification must be consulted.—K. M.

*Improvements in the Manufacture of Soap.* A. W. Rehnström, Malmö, Sweden. Eng. Pat. 7854, May 20, 1890. 4d.

Milk is used as a raw product for the manufacture of hard or soft soap.—K. M.

*Improved Composition of Soap.* G. Allen, London. Eng. Pat. 8077, May 23, 1890. 4d.

ONE part of paraffin wax is melted with one part ammonia and this compound is incorporated with 14 parts common soap.

This composition of soap is specially adapted for washing woollen goods and for preventing their shrinkage.—K. M.

### XIII.—PAINTS, PIGMENTS, VARNISHES, AND RESINS.

*On the Atomicity of the Right-handed Russian Turpentine.* A. Shehukarev. J. Russ. Chem. Soc. 22, 290—296.

As it has been recently suggested to divide all terpenes into two classes, diatomic terpenes or pinenes, combining directly with  $\text{HCl}$ ,  $\text{Br}$ , &c.—and tetraatomic, isoterpenes or limonenes taking  $2 \text{HCl}$ ,  $2 \text{Br}$ , &c., the author undertook to verify the diatomicity of the Russian right-handed terpenes, having some doubts on this matter; his doubts being corroborated by Tilden (J. Chem. Soc. 1888, 882). The action of  $\text{Br}$  upon the hydrocarbon in dry chloroform being repeated, the results proved that the right-handed terpene in weak solution at first takes up only  $\text{Br}_2$ , but in strong solution, and especially when bromine added in excess gives an unstable compound  $\text{C}_{10}\text{H}_{16}\text{Br}_4$ , which partly decomposes into  $\text{C}_{10}\text{H}_{14}\text{Br}_3$  and  $\text{HBr}$ .—N. W. T.

#### PATENT.

*Improved Process for making Paper, Linen, and similar Material Impervious to Water.* A. J. Boulton, London. From D. Macdonald and W. T. Tassie, Toronto, Canada. Eng. Pat. 7024, May 6, 1890. 4d.

THE material is soaked in oil, preferably hot boiled linseed oil, passed between wringing rollers and dried, then completely covered with printer's ink, or similar material evenly distributed over its surface with or without pressure. The object of the invention is to render the materials under treatment impervious to water, and to give them at the same time a highly finished smooth surface.—K. M.

### XIV.—TANNING, LEATHER, GLUE, AND SIZE.

*Unhairing of Skins in the Manufacture of Sole Leather.* W. Borchers. Zeits. f. angew. Chem. 1890, 230—233.

A NUMBER of erroneous statements may be found in books about this branch of leather manufacture. Lietzmann, e.g. (*Die Herstellung der Leder*, 42) states that in the manufacture of sole leather the skins have to be subjected to a so-called putrid fermentation, technically termed "sweating process," whilst the unhairing of those skins which are intended for upper leather is effected by the influence of solutions of alkalis or alkaline earths, especially lime water. This is wrong, for each method may be employed for either purpose, and moreover by a combination of them very good sole leather is obtained. F. Fischer (*Handbuch d. chem. Technologie*) gives a practically correct rule, viz., that the "sweating process" is to be recommended for thick tough skins, and the treatment with lime water for thinner ones. The chief objects in sole leather manufacture are to obtain as good a quality and as great a weight as possible. But in the process of manufacture it is difficult to ascertain the precise time when both these features have been attained. This juncture lies not in the tanning process but in the preceding process of unhairing the skins. Whether the unhairing is done by the "sweating" or by the lime water process, in both cases very often the great mistake is made to continue the treatment until the hair can be removed quite easily. In this respect the methods applied as they are in America are much to be preferred. The skins subjected to the "sweating process" are not left in the warm rooms until the conditions for unhairing are completed, but they are taken out sooner and then placed for about seven hours in a weak lime water solution, where they are kept in constant motion by machinery. This treatment gives very good results. The "sweating process" is doubtless a process of putrefying which begins on the surface and proceeds to the interior of the skins. Ammonia compounds are hereby formed which increase the action of the lime upon the skins. Therefore, if first the "sweating process" and afterwards the lime water treatment are applied, but both to a limited extent only, we restrict as much as possible the harmful influence of both methods, which consists in dissolving and removing substances from the skins which are capable of forming good leather. Long application of the "sweating" as well as of the lime water process, even if used in weak solutions, have a very bad influence upon the skins, and cause great losses of substance. In Buffalo, N.Y., another treatment came into use which gives favourable results as regards weight and texture of the leather. The skins are soaked for about seven hours in a strong solution of lime water, where they are kept in motion, then put into warm water for one night, and after that they are generally ready for unhairing. Should it, however, happen that after this process the hair is not easily removed, then the skins are treated once more in warm water. When it is considered what great losses constantly occur in leather manufacture by improper treatments in the unhairing of skins, especially by the common practice of applying lime water for as long a time as three or four days, the value of a knowledge of the above-mentioned facts in this branch of sole leather manufacture will be recognised.—H. S.

#### PATENT.

*Improvements in Machinery for Dressing or otherwise Preparing Hides, Skins, and Leather.* J. Hall, Leeds. Eng. Pat. 11,138, July 10, 1889. 11d.

THE improvements relate to the construction of machines for preparing leather. The claims are:—(1.) The application and use in leather-working machines of a cam on the crank pin for reciprocating the tool head by means of a rigid connecting rod in combination with a roller or face on a rigid connecting rod for imparting the required rocking motion to the tool-holder." (2.) The application and use in

leather-working machines of the various "stops" and means for actuating same from any desired point for the purpose of facilitating the lifting of one or other of the tools off the leather while the machine is in motion and thus being able to increase the working speed. (3.) "The construction of apparatus shown in the drawings" which accompany the specification.—B. II.

## XV.—AGRICULTURE, MANURES, Etc.

*Memoranda of the Origin, Plan, and Results of the Field and other Experiments, Conducted on the Farm and in the Laboratory of Sir J. B. Lawes, at Rothamsted. June 1890.*

*Experiments on Barley. Thirty-eighth Season.*—On the continuously unmanured plot the amount of dressed grain was 11.25 bushels per acre, the average yield over the first 19 years being 20.1 bushels, and over the second 19 years 13.25 bushels. The plot manured with superphosphate (3.5 cwt. per acre) gave 20 bushels per acre, which is nearly as much as the average yield over the whole 38 years (21.75 bushels). The produce with ammonium salts alone (200 lb. per acre per annum) and with sodium nitrate alone (275 lb.) amounted to 22.5 and 25.25 bushels respectively. The highest yield (42.1 bushels per acre) was on the plot manured with sodium nitrate, sodium silicate, and superphosphate; but the farmyard manure plot (14 tons per acre) gave nearly as much (41.75 bushels); the average yield over the whole period (38 years) is for the former plot 45, and for the latter 48.4, bushels per acre. When ammonium salts and sodium nitrate are supplemented by superphosphate, the average yields (38 years) are raised from 29.25 and 32.75 to 42.9 and 45.75 bushels respectively.

*Experiments on Wheat. Forty-sixth Season.*—The unmanured plot yielded 12.25 bushels per acre, not much less than the average over the first 38 years (13 bushels). The highest average yield (36.5 bushels) is that of the plot which received potassium sulphate (200 lb.), sodium and magnesium sulphates (each 100 lb.) superphosphate

(3.5 cwt.), and ammonium salts (600 lb. per acre), whilst the next highest (35.5 bushels) is the plot receiving the same minerals but sodium nitrate (275 lb.) instead of the ammonium salts. The farmyard manure (14 tons per acre) plot, which gave the highest yield this season (40.5 bushels), gave an average of 34 bushels over the first 38 years. A complete mineral manuring (as in the ammonia and nitrate plots already mentioned), but without nitrogen, gave 15.1 bushels this year, which is practically the same as the average yield (15 bushels) over the first 38 years. On the other hand nitrate alone (275 lb.) and ammonium salts alone (400 lb.) gave the respective average annual produce 22.9 and 20.25 bushels. The plots which are alternately manured with ammonium salts (400 lb.) and minerals, gave this season: ammonia plot 23.4 bushels, mineral plot 10.25 bushels.

*Experiments on Mangel Wurzel. Fourteenth Season.*—The highest amount of produce was obtained from the plot treated with farmyard manure (14 tons) and superphosphate (3.5 cwt.), and cross-dressed with sodium nitrate (550 lb. per acre), the yield of roots being 33 tons 19 cwt. per acre. Nearly as much produce was obtained on that portion of the same plot which was cross-dressed with rape-cake (2,000 lb.) and ammonium salts (400 lb.). Of the plots which received no farmyard manure or rape-cake, the greatest weight of roots was obtained by manuring with superphosphate (3.5 cwt.), potassium sulphate (500 lb.), sodium chloride (200 lb.), and magnesium sulphate (200 lb.) with a cross-dressing of sodium nitrate (550 lb.). The yield of this plot was 22 tons 12 cwt. per acre, but there had been a loss, probably of about 1 ton per acre, owing to a heavy rainfall in July. Where no organic or nitrogenous manure is applied, the yield of roots is very much less; thus, the portion of the last-mentioned plot which received no cross-dressing, yielded only 7 tons 9 cwt., and this is the highest yield of the purely mineral plots. On the other hand, a comparatively low yield (12 tons 7 cwt. per acre) was obtained with a cross-dressing of sodium nitrate (550 lb.) on the plot which was otherwise left unmanured. The unmanured plot gave 4 tons 12 cwt. of roots per acre.

*Experiments on Potatoes. Fourteenth Season.*—The following table shows the manuring of the different plots, and the yield of tubers per acre this season and the average yields over the first and second periods of five years:—

Plots.	Manures per Acre per Annum.	Produce per Acre.				
		Average.			1889.	
		1876-80.	1881-85.			
		Tons. Cwt.	Tons. Cwt.	Tons. Cwt.		
1	Unmanured in 1876, and each year since.....	2 6½	1 19½	0 16½		
2	Unmanured in 1882, and since. Previously farmyard manure (14 tons) .....	4 13½	4 7½	1 13		
3	Farmyard manure (14 tons) alone 1883 and since; previously 3½ cwt. of superphosphate also .....	5 6½	5 1½	1 19½		
4	Farmyard manure (14 tons) alone 1883 and since. In 1882, and previously, 3½ cwt. of superphosphate, and in 1881, and previously, 550 lb. of sodium nitrate .....	6 14½	5 0½	2 18½		
5	400 lb. of ammonium salts .....	2 10½	2 7½	1 4½		
6	550 lb. of sodium nitrate.....	3 4	2 8½	1 12		
7	400 lb. of ammonium salts, 3½ cwt. of superphosphate, 300 lb. of potassium sulphate, 100 lb. of sodium sulphate, 100 lb. of magnesium sulphate .....	6 17½	7 43½	3 9		
8	550 lb. of sodium nitrate, 3½ cwt. of superphosphate, 300 lb. of potassium sulphate, 100 lb. of sodium sulphate, 100 lb. of magnesium sulphate .....	7 6½	6 17½	3 11½		
9	3½ cwt. of superphosphate .....	3 13½	4 6½	1 18½		
10	3½ cwt. of superphosphate, 300 lb. of potassium sulphate, 100 lb. of sodium sulphate, and 100 lb. of magnesium sulphate .....	3 15½	4 6	2 5½		

**SERIES I.—REPORTS OF FIELD EXPERIMENTS, EXPERIMENTS ON VEGETATION, &c.** Published 1847—1890, inclusive.

The following lists give the titles of the papers already published, arranged in two series, and within each series

arranged in chronological order; and they show where each paper appeared.

1. Agricultural Chemistry (J. Roy. Agric. Soc. Eng. 8, 226), 1847.

2. Agricultural Chemistry, Turnip Culture (J. Roy. Agric. Soc. Eng. **8**, 491), 1847.
3. Experimental Investigation into the Amount of Water Given Off by Plants during their Growth, especially in relation to the Fixation and Source of their various Constituents (J. Hort. Soc. Lond. **5**, 38), 1850.
4. Report of some Experiments undertaken at the suggestion of Professor Lindley, to ascertain the Comparative Evaporating Properties of Evergreen and Deciduous Trees (J. Hort. Soc. Lond. **6**, 227), 1851.
5. Agricultural Chemistry, especially in relation to the Mineral Theory of Baron Liebig (J. Roy. Agric. Soc. Eng. **12**, 1), 1851.
6. On the Amounts of, and Methods of Estimating, Ammonia and Nitric Acid in Rain-water (Brit. Assoc. Report, 1854).
7. Report to the Right Hon. the Earl of Leicester on the Experiments, conducted by Mr. Keary, on the Growth of Wheat upon the same Land for Four successive Years at Holkham Park Farm (J. Roy. Agric. Soc. Eng. **16**, 207), 1855.
8. On some Points connected with Agricultural Chemistry; being a Reply to Baron Liebig's "Principles of Agricultural Chemistry" (J. Roy. Agric. Soc. Eng. **16**, 411), 1855.
9. On the Growth of Wheat by the Lois Weedon System, on the Rothamsted Soil; and on the Combined Nitrogen in Soils (J. Roy. Agric. Soc. Eng. **17**, 582), 1856.
10. On some Points in the Composition of Wheat Grain, its Products in the Mill, and Bread (J. Chem. Soc. **10**, 1), 1857.
11. On the Growth of Barley by Different Manures continuously on the same Land; and on the Position of the Crop in Rotation (J. Roy. Agric. Soc. Eng. **18**, 454), 1857.
12. Report of Experiments with Different Manures on Permanent Meadow Land, with Tabular Appendix (J. Roy. Agric. Soc. Eng. **19**, 532, and **20**, 228 and 398), 1858—59.
13. Report of Experiments on the Growth of Red Clover by different Manures (J. Roy. Agric. Soc. Eng. **21**, 178), 1860.
14. On the Sources of the Nitrogen of Vegetation; with special Reference to the Question whether Plants assimilate Free or Uncombined Nitrogen. Abstract (Proc. Roy. Soc. **10**, 544), 1860.
15. On the Application of Different Manures to Different Crops, and on their proper Distribution on the Farm, 1861.
16. On some Points in connection with the Exhaustion of Soils. Abstract (Brit. Assoc. Report, 1861).
17. On the Sources of the Nitrogen of Vegetation, with special Reference to the Question whether Plants assimilate Free or Uncombined Nitrogen (Phil. Trans. Part 2, 1861).
18. Report of Experiments made at Rodmersham, Kent, on the Growth of Wheat by different Descriptions of Manure for several Years in succession on the same Land (J. Roy. Agric. Soc. Eng. **23**, 31), 1862.
19. The Effects of different Manures on the Mixed Herbage of Grass Land (J. Roy. Agric. Soc. Eng. **24**, 131), 1863.
20. On the Sources of the Nitrogen of Vegetation, with special Reference to the Question whether Plants assimilate Free or Uncombined Nitrogen (J. Chem. Soc. **16**), 1863.
21. Liebig and the "Mineral Theory" (note, extracted from a Paper by Messrs. Lawes and Gilbert, J. Roy. Agric. Soc. Eng. **24** (2)), 1863.
22. Further Report of Experiments with different Manures on Permanent Meadow Land (J. Roy. Agric. Soc. Eng. **24** (2)), 1863.
23. Report of Experiments on the Growth of Wheat for Twenty Years in succession on the same Land (J. Roy. Agric. Soc. Eng. **25** (1 and 2)), 1864.
24. On the Selection of Artificial Manures for the Sugar-cane, 1864.
25. On the Accumulation of the Nitrogen of Manure in the Soil (Brit. Assoc. Report, 1866).
26. Preliminary Notice of Results on the Composition of Wheat grown for Twenty Years in succession on the same Land (Brit. Assoc. Report, 1867).
27. On the Home Produce, Imports, and Consumption of Wheat (J. Roy. Agric. Soc. Eng. **4** [2], Part 2), 1868.
28. Exhaustion of the Soil in relation to Landlords' Covenants, and the Valuation of Unexhausted Improvements (read before the London Farmers' Club, April 4, 1870).
29. Scientific Agriculture with a View to Profit (read before the Maidstone Farmers' Club, December 15, 1870).
30. Reports of Experiments on the Influence of various Manures on different Species of Plants (Proc. Roy. Hort. Soc. 1870).
31. Effects of the Drought of 1870 on some of the Experimental Crops at Rothamsted (J. Roy. Agric. Soc. Eng. **7** [2], Part 1), 1871.
32. Notes on Clover Sickness (J. Roy. Hort. Soc. **3**), 1871.
33. Report of Experiments on the Growth of Barley for Twenty Years in succession on the same Land (J. Roy. Agric. Soc. Eng. **9** [2], Parts 1 and 2), 1873.
34. Unexhausted Tillages and Manures, with reference to the Landlord and Tenant (Ireland) Act, 1870, 1874.
35. On the more frequent Growth of Barley on Heavy Land (read before the London Farmers' Club, February 1, 1875).
36. On the Valuation of Unexhausted Manures (J. Roy. Agric. Soc. Eng. **9** [2], Part 1), 1875.
37. Note on the Occurrence of "Fairy Rings" (J. Linn. Soc. Botany, **15**, 17), 1875.
38. On some Points in connection with Vegetation (Address delivered at South Kensington in the Chemical Section of the Science Conference), 1876.
39. On Rainfall, Evaporation, and Percolation (Proc. Inst. Civil Eng. **14**, Part 3), 1876.
40. Freedom in the Growth and Sale of the Crops of the Farm, considered in relation to the Interests of the Landowner and the Tenant Farmer (J. Soc. Arts, December 14, 1877).
41. Composition of Potatoes (J. Roy. Hort. Soc. **5**, Part 5; Proceedings, p. xxxvii.), 1878.
42. On Nitrification; Reports of Experiments made in the Rothamsted Laboratory. (Part I., J. Chem. Soc., January 1878; Part II., J. Chem. Soc. July 1879; Part III., J. Chem. Soc., December 1884.) See also Brit. Assoc. Report, 1881; J. Chem. Soc. October 1885, February 1887, and August 1888. 1878—88.
43. Is Higher Farming a Remedy for Lower Prices? (Lecture delivered before the East Berwickshire Agricultural Association, May 3, 1879. Published by G. Macaskie, "Warder" Office, Berwick).
44. On the Determination of Nitric Acid as Nitric Oxide, by means of its Action on Mercury; a Report of Experiments made in the Rothamsted Laboratory (J. Chem. Soc. July 1879).
45. On the Determination of Nitric Acid by Means of Indigo, with special Reference to Water Analysis; a Report of Experiments made in the Rothamsted Laboratory (J. Chem. Soc. September 1879.) See also—Chem. News, February 2 and 9, 1877.
46. Agricultural, Botanical, and Chemical Results of Experiments on the Mixed Herbage of Permanent Meadow, conducted for more than Twenty Years in succession on the same Land. Part I., the Agricultural Results. Abstract (Proc. Roy. Soc., No. 197, 1879).
47. On some Points in connection with Agricultural Chemistry. Abstract (Brit. Assoc. Report, 1879).
48. Our Climate and our Wheat Crops (J. Roy. Agric. Soc. Eng. **16** [2], Part 1), 1880.
49. On the Home Produce, Imports, Consumption, and Price of Wheat over Twenty-eight (or Twenty-seven) Harvest Years, 1852—53 to 1879—80, inclusive (Jour. of the Statistical Society, June 1880).
50. Agricultural, Botanical, and Chemical Results of Experiments on the Mixed Herbage of Permanent Meadow, conducted for more than Twenty Years in succession on the same Land. Part I., the Agricultural Results. Full Paper (Phil. Trans. 1880 [1]).
51. Sketch of the Progress of Agricultural Chemistry; Address to the Chemical Section of the British Association (Brit. Assoc. Report, 1880).
52. On the Determination of Nitric Acid as Nitric Oxide by means of its Reaction with Ferrous Salts. Reports of

Experiments made in the Rothamsted Laboratory. (Part I., J. Chem. Soc. July 1880. Part II., J. Chem. Soc. August 1882).

53. On the Determination of Carbon in Soils; a Report of Experiments made in the Rothamsted Laboratory (J. Chem. Soc. September 1880), 1880.

54. On the Home Produce, Imports, Consumption, and Price of Wheat over Twenty-seven (or Twenty-eight) Harvest-Years, 1852—53 to 1879—80 (J. Roy. Agric. Soc. Eng. **16** [2], Part 2), 1880.

55. Agricultural, Botanical, and Chemical Results of Experiments on the Mixed Herbage of Permanent Meadow, conducted for more than Twenty Years in succession on the same Land. Part II., The Botanical Results. Abstract (Proc. Roy. Soc. **30**, 556), 1880.

56. Letter on "Bread Reform" (J. Soc. Arts, January 21, 1881).

57. On the Amount and Composition of the Rain and Drainage-Waters collected at Rothamsted; Parts I., II., and III. (J. Roy. Agric. Soc. Eng. **17** [2], 241—279 and 311—350; **18** [2], 1—71). In the separate copies of the entire paper, Section 3 of Part III. is given as Part IV., and Appended Tables are also added, 1881—82.

58. Letters on "Fertility" (Agricultural Gazette, Feb. 21 and 28; March 7, 14, and 21; April 4, 11, 18, and 25; May 2 and 9, 1881).

59. Some Practical Aspects of recent Investigations on Nitrification (J. Soc. Arts, April 7, 1882).

60. Determinations of Nitrogen in the Soils of some of the Experimental Fields at Rothamsted, and the Bearing of the Results on the Question of the Sources of the Nitrogen of our Crops. (Read at the Meeting of the American Association for the Advancement of Science at Montreal, August 1882).

61. Agricultural, Botanical, and Chemical Results of Experiments on the Mixed Herbage of Permanent Meadow, conducted for more than Twenty Years in succession on the same Land. Part II., The Botanical Results. Full Paper. (Phil. Trans. 1882 [4]).

61A. On the Determination of Nitric Acid in Soils. (J. Chem. Soc. August 1882).

61B. On some of the Changes which Nitrogenous Matter undergoes within the Soil (Lecture delivered at South Kensington, April 16, 1883).

62. Contribution to the Chemistry of "Fairy Rings" (J. Chem. Soc. May 1883).

63. New Determinations of Ammonia, Chlorine, and Sulphuric Acid in the Rain-water collected at Rothamsted (J. Roy. Agric. Soc. Eng. **19** [2], Part 2), 1883.

64. The Nitrogen as Nitric Acid in the Soils and Subsoils of some of the Fields at Rothamsted (J. Roy. Agric. Soc. Eng. **19** [2], Part 2, 1883).

65. On the Composition of the Ash of Wheat-Grain and Wheat-Straw grown at Rothamsted in different Seasons and by different Manures (J. Chem. Soc. August 1884).

66. Report of Experiments on the Growth of Wheat for the Second Period of Twenty Years in succession on the same Land (J. Roy. Agric. Soc. Eng. **20** [2], Part 2, 1884).

67. On some Points in the Composition of Soils; with Results illustrating the Sources of the Fertility of Manitoba Prairie Soils (J. Chem. Soc. 1885, 380).

68. On Agricultural Investigation; being a Lecture delivered at the Michigan State Agricultural College, Lansing, Mich., October 14, 1884, and at Rutgers College, New Brunswick, N.J., October 27, 1884.

69. Note on some Conditions of the Development, and of the Activity, of Chlorophyll. Abstract (Brit. Assoc. Report, 1885).

70. On the Valuation of Unexhausted Manures (J. Roy. Agric. Soc. Eng. **21** [2], Part 2), 1885.

71. Results of Experiments at Rothamsted on the Growth of Barley for more than Thirty Years in succession on the same Land (Agricultural Students' Gazette, New Series, **3**, Part 1), 1886.

72. Remarques sur la relation qui existe entre les sommes de température et la production agricole (Arch. des sciences phys. et nat., Troisième période, **16**, No. 11, 15 Novembre, 1886).

73. The Home Produce, Imports, Consumption and Price of Wheat in the United Kingdom, Thirty-four Harvest years, 1852—53 to 1885—86 ("The Field," February 12, 1887).

74. A contribution to the Study of Well Waters (J. Chem. Soc. June 1887), 1887.

75. On the present position of the question of the Sources of the Nitrogen of Vegetation, with some new results, and preliminary notice of new lines of investigation. Preliminary Notice (Proc. Roy. Soc. **43**, 108), 1887.

76. Results of Experiments at Rothamsted on the Growth of Root-crops for many years in succession on the same Land (Agricultural Students' Gazette, New Series, **3** [5]), 1887.

77. On the Present Position of the Question of the Sources of Nitrogen of Vegetation, with some new Results, and Preliminary Notice of New Lines of Investigation. Full Paper. (Phil. Trans. **180** (1889), B., pp. 1—107).

78. Results of Experiments at Rothamsted on the Growth of Potatoes for twelve years in succession on the same Land (Agricultural Students' Gazette, New Series, **4**, Part 2, 1888).

79. The History of a Field newly laid down to Permanent Grass (J. Roy. Agric. Soc. Eng. **25** [2], Part 1, 1889).

80. The Amount of Nitric Acid in the Rain-Water at Rothamsted, with Notes on the Analysis of Rain-Water (J. Chem. Soc. August 1889).

81. Results of Experiments at Rothamsted on the Growth of Leguminous Crops for many years in succession on the same Land (Agricultural Students' Gazette, New Series, **4**, Parts V. and VI.), 1889—90.

82. New Experiments on the question of the Fixation of Free Nitrogen. Preliminary Notice (Proc. Roy. Soc. **47**, 85), 1890.

83. The Food of our Agricultural Crops (J. Roy. Agric. Soc. Eng. **1** [3], Part 1, 1890).

#### SERIES II.—REPORTS OF EXPERIMENTS ON THE FEEDING OF ANIMALS, SEWAGE UTILISATION, ENSILAGE, &c. Published 1849—1885, inclusive.

1. Agricultural Chemistry; Sheep Feeding and Manure, Part 1. (With Tabular Appendix in 1856.) (J. Roy. Agric. Soc. Eng. **10**, 276), 1849.

2. Report of Experiments on the Comparative Fattening Qualities of Different Breeds of Sheep; Hampshire and Sussex Downs (J. Roy. Agric. Soc. Eng. **12**, 414), 1851.

3. Report of Experiments on the Comparative Fattening Qualities of Different Breeds of Sheep; Cotswolds (J. Roy. Agric. Soc. Eng. **13**, 179), 1852.

4. On the Composition of Foods in relation to Respiration and the Feeding of Animals (Brit. Assoc. Report, 1852).

5. Agricultural Chemistry: Pig Feeding (J. Roy. Agric. Soc. Eng. **14**, 459), 1853.

6. On the Equivalency of Starch and Sugar in Food (Brit. Assoc. Report, 1854).

7. Experiments on the Comparative Fattening Qualities of Different Breeds of Sheep; Leicesters and Cross-breeds (J. Roy. Agric. Soc. Eng. **16**, 45), 1855.

8. On the Sewage of London (J. Soc. Arts, March 7, 1855).

9. Letter on the Utilisation of Town Sewage (from the Report ordered by the House of Commons to be printed, August 3, 1857. Appendix xii., 477).

10. Experimental Inquiry into the Composition of some of the Animals Fed and Slaughtered as Human Food. Abstract (Proc. Roy. Soc. **9**, 348), 1858.

11. Observations on the recently-introduced Manufactured Foods for Agricultural Stock (J. Roy. Agric. Soc. Eng. **19**, 199), 1858.

12. Experimental Inquiry into the Composition of some of the Animals Fed and Slaughtered as Human Food (Phil. Trans. Part 2, 1859).

13. On the Composition of Oxen, Sheep, and Pigs, and of their Increase while Fattening (J. Roy. Agric. Soc. Eng. **21**, 433), 1860.

14. On the Composition of the Animal Portion of our Food, and on its relations to bread. Abstract (J. Chem. Soc. **12**, 54), 1860.

15. Fifth Report of Experiments on the Feeding of Sheep (J. Roy. Agric. Soc. Eng. **22**, 189), 1861.
16. Report of Experiments on the Fattening of Oxen at Woburn Park Farm (J. Roy. Agric. Soc. Eng. **22**, 200), 1861.
17. Experiments on the Question whether the Use of Condiments increases the Assimilation of Food by Fattening Animals, or adds to the Profits of the Feeder (Edinburgh Veterinary Review and Annals of Comparative Pathology, July, 1862).
18. Supplementary Report of Experiments on the Feeding of Sheep (J. Roy. Agric. Soc. Eng. **23**, 191), 1862.
19. The Utilisation of Town Sewage (J. Roy. Agric. Soc. Eng. **24**, 65), 1863.
20. On the Chemistry of the Feeding of Animals for the Production of Meat and Manure (read before the Royal Dublin Society, March 31, 1864).
21. On the Sewage of Towns (Third Report and Appendices 1, 2, and 3, of the Royal Commission. Presented to Parliament), 1865.
22. Report (presented to Parliament) of Experiments undertaken by Order of the Board of Trade to Determine the Relative Values of Unmalted and Malted Barley as Food for Stock, 1866.
23. On the Composition. Value and Utilisation of Town Sewage (J. Chem. Soc. **4** [2], or **19** [1]), 1866.
24. Food in its Relations to the Various Exigencies of the Animal Body (Phil. Mag. July 1866), 1866.
25. On the Sources of the Fat of the Animal Body. (Abstract, Brit. Ass. Rep. 1866. Full paper, Phil. Mag. Dec. 1866).
26. Note—On Sewage Utilisation (Proc. Inst. Civil Eng. **14**, Part 3) 1876.
27. On some Points in connection with Animal Nutrition (Address delivered at South Kensington in the Biological Section of the Science Conferences), 1876.
28. On the Formation of Fat in the Animal Body (J. Anat. and Physiol. **11**, Part 4), 1877.
29. Supplement to former Paper entitled "Experimental Inquiry into the Composition of some of the Animals Fed and Slaughtered as Human Food"—*Composition of*

*the Ash of the Entire Animals, and of certain Separated Parts.* (Abstract, Proc. Roy. Soc. **35**. Full Paper, Phil. Trans. Part 3, 1883).

30. Experiments on Ensilage conducted at Rothamsted, Season 1884—5.—N. H. J. M.

*Record of Experiments in the Production of Sugar from Sorghum in 1889.* H. W. Wiley, U.S. Department of Agriculture. Bulletin **26**.

See under XVI., page 877.

*Agricultural Experiment Station of the State College of Kentucky, 1890.* Bulletins **26** and **27**.

*Corn (Maize) Experiments.*—Maize not being a British crop, the experiments may be briefly summarised. Increased yields on this "blue grass" soil followed the use of potash fertilisers and of potash and nitrogen combined, but phosphates or nitrogen without potash showed a loss instead of a profit. Sulphate and chloride of potassium produced identical results. Potash salts (160 lb. per acre) applied the year before to plots of maize greatly increased the yield, and this year the same plots showed almost as great an increase of yield, although no fresh dressings were applied. Analyses show that whereas the crop on the unmanured plots removed 17.4 lb. of  $K_2O$  per acre from the soil, that on the potash manured plots contained 43—52 lb. per acre; the amount applied in the potash manures was 80 lb. of  $K_2O$  per acre, so that less than half is recovered the first season. The same is true of phosphoric acid. In the case of nitrogen the maximum crop took up over threefold the amount applied as manure.

*Experiments with Commercial Fertilisers on Hemp.*—The soil was a much worn "blue grass" soil shown by experiments with other crops to respond to the use of potash fertilisers; oats were taken the previous year. The various manures were broadcasted just before sowing the hemp:—

Plot.	Fertiliser.	Lb. per Acre used.	Height of Plant.	Fibre per Acre.	Increase.	Quality.
1	None .....	..	Feet. 4½	Lb. 720	..	Poor; not strong; too short.
2	Sodium nitrate .....	160	} 7½	1,120	410	Good hand spinning.
	Soil .....	480				
3	Superphosphate .....	320	} 6	860	140	Fair.
	Soil .....	320				
5	Soil .....	480	} 6½	1,050	330	Fair hemp; good strength.
	Potassium chloride .....	160				
6	Sodium nitrate .....	160	} 7	1,210	520	Too short to make good quality.
	Soil .....	120				
	Superphosphate .....	320				
7	Sodium nitrate .....	160	} 8	1,500	780	Good strength and quality.
	Soil .....	160				
	Potassium chloride .....	320				
9	Superphosphate .....	320	} 5½	900	350	Only fit for shorts.
	Potassium chloride .....	160				
	Soil .....	160				
10	Superphosphate .....	20	} 7	1,360	810	Extra hand spinning hemp.
	Potassium chloride .....	160				
	Sodium nitrate .....	160				
11	Land plaster .....	160	4	550	..	



Potash and nitrogen are therefore required on this soil, and a good fertiliser for hemp should contain 12 per cent. of potash, 4 per cent. of nitrogen, and, say, 6 per cent. of soluble phosphoric acid. All the manures used showed a margin of profit, those on plots 7 and 10 nearly doubling the value of the crop. Potassium sulphate tried against an equal weight of chloride gave almost identical results, so the latter manure, being cheaper, is preferable. Ammonium sulphate tried against an equivalent quantity of sodium nitrate gave nearly an equal result—1,320 lb. fibre against 1,380 lb. Doubling the potash salt, whether sulphate or chloride, scarcely increased the yield. Doubling the sodium nitrate and the ammonium sulphate scarcely increased the yield or quality.—J. M. H. M.

*Experiments on the Gain and Loss of Nitrogen by Cultivated and Fallow Soils.* A. Pagnoul. Compt. Rend. 110, 910—912.

THESE experiments were made from March 1888 to March 1890. Six pots of impermeable earthenware, 34 cm. in height, and containing 22 kilos. of earth with a surface of 7.54 square decimetres were so constructed that the drainage water could be collected. To the earth was added 5 grms.

The drainage water gave the following results:—

	A.	a.	B.	b.	C.	c.
Ammoniacal nitrogen.....	0.025	0.017	0.024	0.020	0.021	0.01
Nitrogen in the form of nitrate.....	0.942	0.762	0.047	0.068	0.191	0.16

In March 1890 a sample of the earth was taken from each pot with a cylindrical borer and analysed. The results are tabulated as follows:—

	A.	a.	B.	b.	C.	c.
	Grms.	Grms.	Grms.	Grms.	Grms.	Grms.
(1) Nitrogen remaining in soil.....	24.640	23.730	26.180	27.720	29.260	32.310
(2) Nitrogen assimilated by crops.....	..	..	1.490	1.456	4.266	4.144
(3) Nitrogen carried off by drainage.....	0.967	0.779	0.071	0.088	0.212	0.184
Totals.....	25.607	24.539	27.741	29.264	33.738	36.638

Subtracting the nitrogen originally present (23.980 grms.) from these totals, then—

Nitrogen absorbed from the air during two years..	1.627	0.559	3.761	5.284	9.758	12.668
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Finally, if from these numbers be subtracted the amount of nitrogen assimilated by the crops and carried off by drainage (taking the means for each pair of pots) then the actual increase of nitrogen in the soil is—

	For each Pot, in Grms.	Per Hectare, in Kilos.
Fallow soil.....	0.220	29
Soil sown with grass .....	2.970	394
Soil sown with clover ....	6.820	904

To sum up, the loss of ammoniacal nitrogen through drainage, was almost *nil*; the loss of nitrogen as nitrate was very considerable for fallow land, very small for grass land, considerable for land sown with one of the *Leguminosæ* (clover).

The total gain in nitrogen was sensible for fallow land, considerable for grass land, and very considerable for the

of dried blood and 1 grm. of nitrogen as ammonium sulphate. The total amount of nitrogen in each pot was as follows:—

	Grms.
Contained in the 22 kilos. of earth .....	22.440
Contained in the dried blood .....	0.540
Contained in the ammonium sulphate .....	1.000
Total .....	23.980

The pots were lettered A, B, C, *a*, *b*, *c*: A and *a* were allowed to lie fallow, B and *b* planted with grass, C and *c* with clover.

The yields dried and analysed gave the following results:—

	1888.			1889.		
	Weight.	Nitrogen Per Cent.	Total Nitrogen.	Weight.	Nitrogen Per Cent.	Total Nitrogen.
B	41.0	3.08	1.263	10.7	2.13	0.227
<i>b</i>	42.0	3.08	1.294	7.7	2.10	0.162
C	74.5	3.92	2.990	36.3	3.71	1.346
<i>c</i>	72.5	3.92	2.843	37.5	3.47	1.301

clover land. On the fallow land the loss of nitrogen by drainage was greater in the second year than in the first, but it was less in the cultivated land. Notwithstanding this fact, and the continued gain of nitrogen by the soil, the second year's crops were smaller than the first year's. The author thinks that this singularity is due to the nature of the plants chosen, as oats sown this year are stronger on the clover land than on the grass land, and stronger on this than on the fallow land.—P. J. H.

*On the Role of Green Manures as Suppliers of Nitrogen.* A. Muntz. Compt. Rend. 110, 972—975.

GREEN plants are buried in the soil in many places as manure, and with good results. The plants generally chosen are those supposed to absorb nitrogen from the air, especially the *Leguminosæ*. Although it cannot be considered, as a rule, advantageous to sacrifice one crop for the sake of the next, yet in cases where the soil is deficient in humus, or where other manure cannot easily be obtained, this process may well be resorted to.

The action of green manures must be chiefly attributed to their nitrogen. Now, as plants generally take up their nitrogen from nitrates, the value of nitrogenous manures may be measured by their rate of nitrification.

Experiments were therefore tried with various samples of earth, differently manured, but containing each 1 gram. of nitrogen per kilo.

With a light soil containing 2 per cent. of calcium carbonate, the quantities of nitric acid per kilo. of earth produced in three months, were found to be—

	Megms. of Nitric Acid per Kilo.
1. Earth with green manure (lupin) .....	183
Earth with dried blood.....	161
Earth with ammonium sulphate.....	268

With a heavy soil containing but little calcium carbonate, the following results were obtained in the same time:—

	Megms. of Nitric Acid per Kilo.
1. Earth with green manure (lupin) .....	88.0
Earth with dried blood.....	3.6
Earth with ammonium sulphate.....	5.1

Thus, with a light soil green manure was superior to dried blood, and with a heavy soil much superior both to dried blood and ammonium sulphate.

The green manure, by its large volume, renders the heavy impermeable soils less compact, the air circulates more easily, and favours the development of the nitrification organisms. Culture experiments were carried out on the farm at Vincennes on a light soil, to which nitrogenous

manures were added, so that there were present 100 kilos. of nitrogen per hectare, other necessary elements being abundant.

A very exhausting plant, the giant maize, was sown for forage.

Eighteen days after having manured the soil, it was analysed, and the crops were cut and weighed at the end of September. The results obtained were as follows:—

	Nitric Acid in Megms. per Kilo. after 18 Days.	Weight of Yield in Kilos. per Hectare.
Portion with green manure (lucerne) .....	86.0	78,000
Portion with dried blood.....	72.2	71,500
Portion with ammonium sulphate....	121.4	66,000
Portion with no nitrogenous manure.	14.5	39,500
Portion with sodium nitrate .....	..	78,500

The author concludes that the efficacy of green manures is due to their rapid nitrification and to their favourable influence on the physical constitution of the soil.—P. J. H.

#### Copper Salts as Preventives of Potato Blight. A. Girard. Compt. Rend. 110, 1089—1092.

THE author has made a series of experiments to show the power possessed by copper salts of preventing the ravages of potato blight (*Phytophthora infestans*). The first experiments were made in August 1888, at Joinville-le-Pont and Clichy-sous-Bois, when the growth had begun to be attacked by blight, the action of the copper compound, in this instance, being solely curative. The dressing employed contained, per 100 litres, 1 kilo. of lime (weighed as CaO) and 3 kilos. of copper sulphate. Though the growth was far advanced, considerable benefit accrued to the crop from the application of this dressing, as the following results show:—

Variety of Potato.	Dressed Soil.			Undressed Soil.			Increase in the Proportion of Sound Tubers due to Dressing.
	Total Yield per Acre.	Diseased Tubers, per Acre.		Total Yield per Acre.	Diseased Tubers, per Acre.		
	Kilos.	Kilos.	Per Cent.	Kilos.	Kilos.	Per Cent.	Per Cent.
Eos .....	450.0	20.0	4.2	468	25.0	5.5	2.7
Kornblum .....	450.0	5.0	1.1	400	30.0	7.5	20.2
Aurélié .....	427.0	21.0	4.9	420	31.0	7.4	4.4
Gelbe rose .....	330.7	10.7	3.1	300	12.3	4.1	14.3
Jeuxey .....	414.5	25.0	6.0	365	48.0	13.1	22.9
Richter's .....	564.0	15.0	2.6	498	14.5	2.9	13.5
Red-skinned.....	460.0	33.0	7.0	423	51.0	12.0	17.0

The first three varieties were grown at Joinville, the others at Clichy. It follows from these results, (1), that a purely curative treatment does not ensure immunity from the disease; but that, (2), with certain varieties of the plant,

the proportion of sound tubers is increased to the extent of 20.2 and 22.9 per cent.

In 1889, at the same localities, a preventive treatment was tried, the results at Clichy-sous-Bois being as follows:—

Variety of Potato.	Dressed Soil.			Undressed Soil.			Increase in the Proportion of Sound Tubers due to Dressing.
	Total Yield per Acre.	Diseased Tubers, per Acre.		Total Yield per Acre.	Diseased Tubers, per Acre.		
	Kilos.	Kilos.	Per Cent.	Kilos.	Kilos.	Per Cent.	Per Cent.
Gelbe rose .....	328	..	..	308	8.0	2.6	9.3
Jenney .....	341	1	0.3	321	30.0	9.1	16.8
Richter's .....	439	..	..	421	1.0	0.2	4.3
Red-skinned .....	400	..	..	394	1.5	0.4	1.9

The author, in the original paper, shows that, after deducting the cost of the dressing, there was realised a pecuniary gain of 1.52 to 3 frs. per are (152 to 300 frs. per hectare) of surface treated.

By increasing the amount of copper salt to 3 per cent., the beneficial results are rendered still more certain.—E. B.

*The Decomposition of Rocks and the Formation of Arable Land.* A. Muntz. *Compt. Rend.* **110**, 1370—1372.

THE author regards it as probable that vegetation, and especially the lower classes of plant life, play a very important part in the disintegration of the bare rock. Indeed, he considers that the nitrifying organism itself plays indirectly a part. He suggests that the mosses and lichens, many of which cover the highest ground where vegetation is found to exist at all, cause, by the action of their roots, the numerous interstices, which afterwards serve for the more ready mechanical disintegration of the rock masses.

One of the most striking proofs of the fact that not only vegetable life but also the nitrifying organism may abound in rock is furnished by a mass obtained from the Oberland Alps, composed of a calcareous schist, and which was black and friable. This rock contained large quantities of the nitrifying organism.

The author has obtained earth from beneath the surface of glaciers which contained the nitrifying organism, the fact proving, he submits, that exposure to a low temperature for even long periods of time does not kill it, but merely prevents it from assuming the active state.—J. W. L.

improving the plant and selecting the best varieties, but this takes time. It is pointed out that "the beet sugar industry began 140 years ago, and for half of that time no one could make it pay." It encountered the same difficulties in selecting the best varieties; consequently there is good reason to hope that the sorghum sugar industry will ultimately be profitable.

Wiley speaks strongly as to the necessity of selecting favourable localities for the cultivation of this plant; present knowledge points to Central Southern Kansas as being the best adapted to this purpose. He says that: "Sorghum at present is so far inferior to the sugar beet and sugar cane as to require the help of the most favourable soil and climate in order to maintain its existence as a sugar-producing plant."—W. M.

*New Processes and Apparatus for Sugar Factories.* Dingl. *Polyt. J.* **276**, 567—572.

IN the estimation of sugar in beet juice, especially when the beets are unripe or diseased, the filtrate, after defecation with basic acetate of lead, often becomes turbid and dark, and cannot be polarised. Frolia has observed that the compound formed by the lead and chromogen is much more insoluble in an alkaline than in an acid or neutral solution, and that the addition of 1 to 2 drops of strong ammonia to such turbid solutions, with subsequent shaking and filtration, gives a perfectly clear filtrate, which can be easily polarised. In Belgium, where the beets are often paid for according to the sugar contained in them, analyses are consequently made in different laboratories to determine the amount of sugar, but it has been found that there is a great want of uniformity in the results obtained. The samples generally consist of quarters of the beet, and as several days often elapse between the different analyses, the samples lose weight considerably. The following formula has been proposed to make the correction for this loss of weight:—
$$PR = P'$$
 where P = the weight at the second analysis, P' = the weight at the first analysis, and R = amount of sugar found. The amount of sugar to be expected after the beets have dried may be calculated from the formula,  $R = \frac{ZP}{P}$

where Z = the original amount of sugar. These formulae would give correct results if the beet did not undergo alteration during drying, but Masson has shown that this is not the case, and it appears that the cut beets rapidly undergo chemical change. The cause of this change is not yet known, and calls for further investigation. An apparatus has been patented by Büttner and Meyer (D.R.P. 50,990, 1889), for the treatment of beet chips with milk of lime before pressing and drying them. It has been found that exhausted cane or beet chips cannot be satisfactorily pressed unless they are impregnated with a definite quantity of lime, the simple weighing of the lime and the chips does not suffice, on account of the variation in the amount of water contained in the chips. The inventors have found that if the milk of lime be in excess, the chips abstract by chemical action from milk of lime of a given concentration practically the same amount of lime in a given time. The chips are conducted mechanically into a vessel containing milk of lime of sp. gr. 1.0035, with violent agitation, and continuously removed from it. In connexion with the question, "Is there any reason to expect any serious competition from other sweetening agents (saccharine, &c.)?" the observation has been made at a large liqueur factory in Vienna that liqueurs sweetened with saccharine when exposed in uncorked bottles, &c. quickly lose their sweet taste. Stift has made experiments to test this observation by adding different quantities of saccharine to solutions of 35 volume per cent. of the purest rectified spirit in distilled water, and keeping the samples both in closed and open vessels. No difference was observed after eight days, but after 16 days the open samples were distinctly less sweet, and the saccharine finally could not be recognised by Remsen's delicate reaction. The question as to the compound into which the saccharine is split up was not investigated.—W. M.

## XVI.—SUGAR, STARCH, GUM, Etc.

*Derivatives of Diphenylhydrazine.* R. Stahel. *Annalen*, **258**, 242—257.

See under XXIII., page 902.

*The Quantitative Estimation of Furfuraldehyde and Pentaglucooses.* A. Günther and B. Tollens. *Ber.* **23**, 1751—1752.

See under XXIII., page 903.

*Record of Experiments in the Production of Sugar from Sorghum in 1889.* H. W. Wiley, U.S. Department of Agriculture. *Bulletin* **26**.

THIS report gives an interesting account of the work carried on at 15 different sugar works situated in Iowa, New Jersey, Virginia, Louisiana, Maryland, and Kansas, but the results have been very discouraging. In seven factories the record is total failure. A great part of this failure must be set down to the careless and incompetent manner in which most of the factories were constructed, and also to inattention to absolute conditions of success which had been previously determined by the work of the Department.

In spite of all the commercial failure attending last year's work many of those engaged in this struggling industry are convinced that success will ultimately crown their efforts, when sufficient capital backed by thoroughly capable management really takes the matter in hand. It would appear that the sorghum sugar industry has never had a fair chance from a merely mechanical point of view.

On the other hand, the plant as at present grown is extremely variable in quality, and unreliable as to the quantity of sugar it contains. Much good work has been done under the direction of the Department in the way of

*Invert Sugar in Cane Juices.* H. Schweitzer. Chem. Zeit. 14, 1031—1032 and 1061—1062.

THE author remarks that it is only very recently that any chemical control has been exercised in the Louisiana sugar factories, and now gives the result of some observations he has made during his work last winter at the Ashton plantation, St. Charles Parish, La. The factory was one of the better class of Louisiana sugar houses. The different operations were—(1) crushing of the canes, (2) clarification of the juice with sulphurous acid, (3) further clarification with lime, (4) further clarification by heating and addition of small quantity of lime, (5) concentration in open pans, (6) concentration in vacuum pans. He analysed the liquors from the different operations, and also after keeping them for known periods, and found that delay in the crushing of the cut canes or in the treatment of the liquors was the chief source of increase of invert sugar. Were a factory so arranged that the canes and liquor could be worked without loss of time, the increase of invert sugar due to the processes would be practically nil, except during the evaporation in the open pan, and to obviate this he recommends the suppression of open pan evaporators, and their replacement by vacuum apparatus. In regard to the generally accepted opinion among sugar growers that frosty weather injures the sugar juice in the cane, his observations lead him to conclude that frost by itself has an insignificant effect on the production of invert sugar, but if the frosty weather be succeeded by very warm days the juice is injured.—W. M.

#### PATENTS.

*Improvements in Evaporators or Apparatus for the Treatment of Cane Juice.* S. Pitt, Sutton. From L. W. Tracy, New York, U.S.A. Eng. Pat. 11,136, July 10, 1889. 8d.

THIS invention relates to that class of apparatus in which cane juice is successively passed through a series of evaporators, and in transit is evaporated in a vacuum to a desired density. The pans are worked in double or triple effect, the steam driven off from the first pan passing through the coils of the second pan and so on. The invention consists in the special mode of construction of the evaporators and their connexions which can be fully understood only by reference to the drawings which accompany the specification. The evaporators are oblong in shape and in horizontal section are long and narrow; the interior of each pan contains a series of horizontal tubes through which the steam circulates and over which the liquor is allowed to fall in a fine rain-like spray.—A. J. K.

*Electrolytic Treatment of Vegetable Matters and Cereals for the Production of Starch.* E. Hermite, E. J. Paterson, and C. F. Cooper, London. Eng. Pat. 12,906, August 15, 1889. 4d.

IN the manufacture of starch from potatoes, rice and other vegetable substances, the pulpy liquor resulting from the disintegration and washing of the raw material contains in addition to the starch a considerable quantity of organic impurities. To remove these the liquor is treated by electrolysis in presence of a chloride, preferably magnesium chloride.—A. J. K.

*An Improved Process of Treating Bran and other Cereal Substances for obtaining Sugar, Starch, Food for Animals, Gum and Paper.* E. Steiger, E. Schulze, and C. Auer-Schollenberger, Zurich, Switzerland. Eng. Pat. 12,920, August 15, 1889. 6d.

THIS invention is based upon the discovery that in the bran and husk obtained in the treatment of cereals there exists a carbohydrate which the authors designate "metaraban." From "metaraban" by suitable treatment with reagents can be obtained a saccharine body consisting chiefly of arabinose, a glutinous body resembling gum arabic. The invention comprises a process for obtaining either of these two substances, and at the same time rendering the fibrous

residue suitable for the manufacture of paper. It is first necessary, however, to free the bran from the protein and other alimentary substances which it contains. For this purpose the bran is boiled for about three hours with a 1 per cent. solution of ammonia or common salt. The liquor is expressed from the solid residue which is then dried and ground. The liquor is neutralised by phosphoric acid, evaporated, dried and ground, forming the fodder meal. The dried ground bran cake from which the food substances have been extracted is boiled with dilute acid, preferably sulphuric of 1 or 2 per cent. By this means the "metaraban" is converted into sugar and dissolved out, the liquor is neutralised by carbonate of lime, decolourised by bone-black, and concentrated by evaporation. The sugar then crystallises out and consists largely of arabinose. The cellular membrane remaining is toughened by boiling with weak alkaline lye, and is then suitable for the manufacture of comparatively strong paper. If a more dilute sulphuric acid, say one-tenth to one-fifth per cent. be used in above process, no sugar will be formed, but a glutinous substance which is soluble in water and resembles gum arabic. A similar glutinous substance is obtained if a weak alkaline lye be substituted for the dilute sulphuric acid. In either case the solution is expressed from the solid residue, suitably neutralised, clarified with hydrated oxide of lead and then evaporated. A solid substance is then obtained, which has the properties of gum arabic, and is very adhesive.—A. J. K.

*An Improved Process and Apparatus for the Concentration of Saccharine and other Liquids and obtaining Crystals therefrom.* R. Raeymaeckers, Tirlemont, Belgium. Eng. Pat. 13,555, August 27, 1889. 8d.

THE improved apparatus consists in a modification of the ordinary vacuum pan. The lower half of the pan is provided internally with false sides, consisting of finely perforated plates. In a series of pans the space between the false and true sides of one pan is connected with the interior of the next pan and so on.

By the improved process the pans are worked in series. Good liquor is run into No. 1; after boiling some time the syrup and fine crystals are drawn off into No. 2 pan, leaving the larger crystals behind in No. 1. These are now boiled with more of the original liquor and the syrup again drawn off, and this goes on until the syrup from No. 1 is as good as original liquor. The contents of No. 1 are now discharged, and the crystals separated by centrifugal machine. By applying similar methods to the other pans of the series a graduated series of crystals and syrups is obtained.

—A. J. K.

*Improvements in the Production of Invert Sugar.* A. Wolf and A. Kollrepp, Berlin, Germany. Eng. Pat. 16,540, October 19, 1889. 6d.

THIS invention is based on the following observations:—(1.) The rapidity with which the inversion of saccharose takes place is, for all temperatures up to 95° C. and for all degrees of concentration of the solution of cane sugar up to about 80 per cent. by weight, dependent solely on the relation of the quantity of acid employed to the quantity of water present. Thus a solution of cane sugar of 80 per cent. strength contains only about one-sixteenth of the quantity of water contained in a 20 per cent. solution for the same quantity of sugar, hence the quantity of acid required for the complete inversion of an 80 per cent. solution, other things being equal, is only one-sixteenth of that necessary for a 20 per cent. solution. (2.) For each kind of acid there is a certain very weak degree of concentration at which, on being heated for from half an hour to an hour at about 80° to 90° C., it completely inverts cane sugar in a quantity equal to about four times the quantity of the diluted acid, and without decomposing or colouring the invert sugar. Tables are given showing the time taken for inversion with various acids, temperatures, and concentrations.—A. J. K.

## XVII.—BREWING, WINES, SPIRITS, Etc.

*The Alcoholic Fermentation of Invert Sugar.* U. Gayon and E. Dabourg. *Compt. Rend.* **110**, 865—868.

The authors have determined the fermentative action of various yeasts on invert sugar by noting the optical activity

of its fermenting solutions at regular intervals. If the relation existing between the rotatory powers and the amounts of sugar fermented be represented graphically, a parabolic curve will be obtained, of which the form is constant for the same species of yeast placed under the same conditions; but varies greatly for different species. In the following table, the results obtained by the cultivation of various yeasts at 25° C. are given.

Nature of Yeast.	Original Levo-Rotation in Saccharimeter Degrees.	Maximum Levo-Rotation in Saccharimeter Degrees.	Maximum Increase in Rotation.
1. R. Yeast, non-inverting .....	100	103	3
2. M. Brewing yeast, inverting .....	100	105	5
3. P. <i>Sacch. pastorianus</i> , inverting .....	100	107	7
4. G. Top yeast, inverting .....	100	108	8
5. H. Yeast, inverting .....	100	110	10
6. A. Do. ....	100	112	12
7. B. Brewing yeast, inverting .....	100	122	22
8. Z. Yeast, inverting .....	100	125	25
9. " <i>Mycoglossure Duclaux</i> ," non-inverting .....	100	129	29
10. <i>Mucor alternans</i> , non-inverting .....	100	165	65

The curves obtained for these yeasts are all more or less flattened in the same direction, and the rotations are always levo. The form of the curves is little modified by temperature, concentration, acidity, and other circumstances. There are other yeasts, however, which, contrary to the above, ferment the levulose faster than the dextrose. Four yeasts possessing this property are known; one of the most active is a variety of *Saccharomyces eriguus*. It has inverting power, and cultivated in an invert sugar solution at 25° C., yielded the following results:—

Duration of the Experiment.	Rotation in Sacchari- meter Divisions.	Total Un- fermented reducing Sugar per Litre.	Unfermented Dextrose per Litre.	Unfermented Levulose per Litre.
Hours. 0	-190	Grms. 102.0	Grms. 51.0	Grms. 51.0
24	-154	97.1	51.0	46.1
34	- 53	79.4	46.7	32.7
48	+ 38	61.3	43.0	18.3
58	+ 61	38.1	28.9	9.2
72	+ 57	26.3	21.2	5.1
82	+ 20	10.9	8.4	2.5
96	+ 0	0.0	0.0	0.0

The action of this yeast is greatly influenced by temperature, concentration, and acidity of the sugar solution used, as the following table will show:—

Strength of Sugar Solution.	Initial Acidity.	Temperature of the Fermentation.	Maximum Dextro- rotation.
Per Cent.		Degrees.	
10	0.0	$\begin{cases} 10 \\ 25 \\ 40 \end{cases}$	$\begin{cases} 81 \\ 41 \\ 8 \end{cases}$
10	2.4	25	23
25	0.0	$\begin{cases} 10 \\ 25 \\ 40 \end{cases}$	$\begin{cases} 123 \\ 102 \\ 73 \end{cases}$
25	2.4	25	104

In concentrated solutions at low temperatures, the levulose almost entirely disappears before the dextrose is touched. The other three yeasts mentioned have the same general properties as the above, but they have no inverting action on cane sugar. One of them is a variety of *S. apiculatus*. These researches explain certain anomalies that have been encountered in the rotatory powers of fermenting liquids, and in the analysis of raw sugars and molasses.—H. T. P.

*Alcoholic Fermentation and the Transformation of Alcohol into Aldehyde induced by Means of Champignon du Muguet (Fungus of the Lily of the Valley).* G. Linossier and G. Roux. *Compt. Rend.* **110**, 868—870.

VERY contradictory assertions have been made by different observers concerning the action of *Champignon du Muguet* on sugar solutions. In order to settle the question the authors have sown various sterilised media with a pure culture of the ferment. The maximum amounts of alcohol obtained ranged from 2.7—5.5 per cent. by volume, according to the nature of the medium employed. There were produced besides, glycerol, succinic acid, butyric acid, and very considerable quantities of aldehyde and acetic acid. The ferment does not assimilate lactose. Placed in a solution of sucrose it develops, at the expense of the sugar, without fermenting or inverting it, and in a solution of invert sugar the dextrose is destroyed most rapidly. Aldehyde and acetic acid are always produced during the development of the fungus, and in fact the oxidising properties of the ferment may be easily demonstrated by sowing some in a weak solution of alcohol containing the necessary mineral substances. The alcohol rapidly disappears, being replaced by aldehyde and acetic acid. Air is absolutely necessary for the growth of the fungus. It seems probable that the organism only effects the conversion of alcohol into aldehyde, the further oxidation into acetic acid being due to the direct action of the air. This is borne out by the fact that the ferment does not develop in a weak solution of aldehyde. Although the production of alcohol during the growth of the fungus in sugar solutions seems to warrant its classification with the saccharomyces, the authors are of opinion that it far more closely resembles certain species of *muor* in its mode of action. The maximum amount of alcohol produced is less than that formed during the growth of the less energetic varieties of saccharomyces; and, moreover, this maximum is only attained after 3—5 months. The ratio of alcohol produced, to sugar destroyed, is about 0.38, whilst for yeast it is

nearer 0.50. Further, the ratio of sugar destroyed to the crop of ferment produced is about 16—19, instead of 34 for yeast placed under identical conditions. Finally, the alcohol produced during the growth of the fungus is soon converted into aldehyde; so that it is at the same time an alcoholic ferment and an oxidising agent.—H. T. P.

*The Bouquet of Fermented Liquors.* G. Jacquemin. Compt. Rend. **110**, 1140—1142.

THE author has experimented on the various fermentations produced by different kinds of yeast in tartarised barley worts and grape juices. He has found that in common with the opinion expressed by Pasteur, the flavour and quality of the wines produced are, to a great extent, dependent on the special yeast used for fermentation. The same grape juice or tartarised barley wort submitted to the treatment of different kinds of yeast have produced wines of distinct quality and bouquet. So that from barley wort under the influence of yeasts characteristic of the grapes of Beaune, Chablis, and Riquewyhr products were formed, which possessed the well-known bouquets of these growths. Further, tartarised barley wort treated with the yeast extracted from the lees of a tun of cider gave, after fermentation, a product having the exact flavour of cider.—K. M.

*The Preparation of Wine Ferments.* A. Rommier. Compt. Rend. **110**, 1341—1343.

CAREFULLY picked grapes are crushed and placed in a flask, several experiments being started at the same time, so that the one containing least bacteria and mycoderma may finally be chosen. When fermentation has thoroughly commenced, the contents of the flask are gently shaken, and drops of the liquid are sown in flasks containing sterilised and filtered grape juice. Other flasks are inoculated from these, the operation being repeated several times at intervals of 2—4 or more days. In this manner a pure cultivation of *Saccharomyces ellipsoideus* is obtained, and in order to increase its quantity it is now sown in large bulks of grape juice. The author employs flasks plugged with sterilised cotton wool and fitted with side-tubes, which are allowed to dip into water. The yeast is employed in practice by adding 1.5 per cent. of one of the later cultivations to the grape juice immediately after pressing. In order to preserve the ferment it is separated by decantation from the liquid after complete fermentation, and placed in glass bulbs which are then sealed before the blow-pipe. Two such bulbs of yeast, obtained from grapes producing a superior class of wine, were used to start the fermentation in the juice of a common variety of grape. The fermentation was complete in eight days. The wine contained 9.5 per cent. of alcohol and had a most agreeable bouquet, which the wine from these grapes did not ordinarily possess.—H. T. P.

#### PATENTS.

*Improvements relating to Apparatus for the Pasteurisation of Beer, Wine, and other Liquids.* W. Kuhn, Clermont-Ferrand, France. Eng. Pat. 15,261, October 23, 1888; amended May 31, 1890. 8d.

THE patentee now withdraws the claims relating to the second and third methods and apparatus described in the original specification (this Journal, 1889, 719).—A. L. S.

*Improvements in the Method of Malting and Drying Grain, and in Machinery or Apparatus therefor.* J. Sleeman, Taunton. Eng. Pat. 10,891, July 5, 1889. 8d.

A SPECIAL air-tight chamber is constructed of brick, iron, or other suitable material, and it is so arranged and fitted up inside as to serve the purposes of (1) a cistern in which the grain can be steeped; (2) a germinating room; (3) a kiln for drying the malt or grain. Hence grain introduced into such a chamber can be converted into finished malt without being removed from the chamber until all the necessary operations are completed. Inside the chamber is

a cylindrical or polygonal case formed of perforated metal or woven wire work which can be made to revolve on a horizontal axis. The journals on which this axis revolves can be so lowered as to submerge the grain which is contained in the case in the water which the chamber contains during the first operation. When the steeping is finished, the chamber is supplied with fresh air at a suitable temperature for germination. And finally the grain can be dried by the introduction of dry hot air or other suitable means. Drawings are given.—A. J. K.

*Improvements in the Method of Malting and Drying Grain and in Machinery or Apparatus therefor.* J. Sleeman, Taunton. Eng. Pat. 11,844, July 25, 1889. 8d.

THIS invention consists in a special arrangement of tubes for introducing air into the germinating cell described above (Eng. Pat. 10,891), and can best be understood by reference to the drawings.—A. J. K.

*Improvements in the Manufacture of Yeast.* L. Lederer, London. Eng. Pat. 12,187, July 31, 1889. 4d.

AN infusion of hops is prepared by boiling with water a quantity equal to about  $1\frac{1}{2}$  per cent. on the total grain to be mashed. About 10 per cent. of the grain (coarsely crushed barley, malt, and rye) are steeped in the hop infusion at 145° F. and allowed to saccharify. The mash is led into wooden vessels and allowed to acidify for 30—42 hours. It is then cooled to 70° F. and about 3—6 per cent. distillery yeast added. In the meantime the remainder of the grain has been mashed, and the mixture, as above prepared, after fermenting 8—10 hours, is added to the wort. The yeast as it rises is skimmed off and led on "to a double sieve, driven from a shaft with a constant supply of cold water to force the yeast through a fine gauze into a reservoir." After standing in the reservoir an hour or more the clear water is drawn off, fresh water added, and the yeast left until pressing time. After pressing the yeast is broken up, water and spirit added if it is too dry, and packed.—A. L. S.

*Improvements in Filtering Apparatus for Alcoholic Beverages.* H. Mundorf, Pöhlfigheim, Germany. Eng. Pat. 13,258, August 22, 1889. 8d.

THE ledge at the bottom of an ordinary filtering tub, which carries the cake of paper, asbestos, or other filtering material, is provided with a notch all round, but not quite close to the inner wall of the tub. A hoop of metal is wedged into this notch, the hoop being of such breadth that its upper half, the edge of which is sharp, stands free. The pressure of the liquid being filtered causes this sharp edge to cut right into the filtering mass, and thus entirely prevents the leakage of unfiltered liquid between the edge of the filtering cake and the side of the tub. Any liquid finding its way round the edge of the cake is compelled to ascend again and thus undergo filtration. To increase the pressure on the filtering cake, the tub is fitted with an air-tight lid. The filtering mass is supported from below by a fine wire gauze net. Under this is a coarser net which rests on a light wooden grating supported by woodwork. By this means a large filtering area is obtained.—H. T. P.

*A New or Improved Process for the Purification of Mineral Oils, and for Producing Alcohol.* W. P. Thompson, Liverpool. From P. Marix, Paris, France, and N. Notkin, Moscow, Russia. Eng. Pat. 13,653, August 29, 1889. 6d.

See under H1., page 852.

*Improvements in and Relating to the Treatment of Alcoholic Liquors.* J. Hutchison, Shawlands. Eng. Pat. 14,406, September 12, 1889. 4d.

ALCOHOLIC liquors are impregnated with a suitable, tasteless gas, in order to render them effervescent. In practice, still whisky, brandy, rum, gin, wine, or other alcoholic

liquid, is placed in a strong vessel, and oxygen, ozone, or other tasteless gas is forced in. The impregnated liquid is filled into strong bottles, syphons, or other suitable vessels. Liquid syrups, or glucose may be added to the spirit before bottling, in order to produce a more crystalline effervescence resembling that of champagne. Flavouring essences may also be added to the alcoholic liquid if desired.—H. T. P.

*Improvements relating to the Manufacture of Spirit from Grain and Cereals, and to the Treatment of the By-products derived therefrom for the Production of Cattle Food, Charcoal, and Manure.* A. Manbré, sen., London. Eng. Pat. 14,719, September 18, 1889. 6d.

THE grain to be employed is finely ground and cleansed from husk. The meal is mixed with two-thirds of the total quantity of water intended to be used, and 2 to 5 lb. of malt flour are added for every 100 lb. of meal taken. The mash is raised to 180° F., being stirred all the time, and is then run into a copper or gun-metal vessel, in which the remaining third of water plus 2—3 lb. of sulphuric acid for every 100 lb. of grain used, have been previously heated to 180° F. The vessel is then closed and the temperature is raised to from 300° to 320° F., at which temperature the conversion of the starch is complete. The sulphuric acid is then neutralised by phosphate of lime. The sulpho-phosphate of lime formed greatly aids the subsequent fermentation and leads to the production of spirit free from fusel oil. The neutralised liquid is filtered and fermented, and the spirit distilled off in the usual way. Three by-products are obtained:—1. The husk, derived from the grinding of the grain; 2. The precipitate filtered off from the neutralised solution; 3. The wash left after distillation. For the production of cattle-food the by-products are mixed, either together or separately, with any suitable nutrient meal or other substance, the mixture being subsequently converted into cakes by powerful pressure. For the production of charcoal, the by-products are compressed, either separately or together; the cakes are then ground and calcined in the usual way. For the preparation of manure, the compressed by-products are ground and mixed with other suitable manure products, such as nitrates, phosphates, &c.—H. T. P.

*Improvements relating to the Manufacture of Refined Malt Extract from Barley Malt, and from Barley Malt and Amylaceous Substances, and the Treatment of the Spent Grains for the Production of Cattle Food.* A. Manbré, London. Eng. Pat. 14,928, September 21, 1889. 6d.

BEST matured barley is cleansed from all dust, seeds, broken kernels and small grain, allowed to germinate in the usual way until the arospire has attained to about one half the length of the grain, and then desiccated as usual. The desiccated barley malt is ground to a fine meal, the husks are removed and the malt meal is saccharified at a temperature not exceeding 175° F., until the desired proportion of maltose and dextrine is produced. The saccharine wort is now subjected in a strong vessel to a temperature of 275°—300° F. under pressure in order to destroy any germs of disease. After filtering and draining the wort is boiled with hops and finally evaporated to form a solid extract of malt. The process may be varied by using with the barley malt flour, suitable proportions of other amylaceous substances. The husk from the barley and the spent grains are worked up with other suitable materials, such as oatmeal, barley meal, rye meal, maize meal, bran, linseed oil, &c. into cattle food.—A. J. K.

*Improvements relating to the Production and Manufacture of Methylated Spirit and the Treatment of the By-Products derived therefrom.* A. Manbré, sen., London. Eng. Pat. 15,064, September 24, 1889. 6d.

By this method methylated spirit may be directly prepared from starchy and woody substances. In carrying out the process, any grain or mixture of grains, or other starchy substances, is reduced to a fine powder and mixed with a

suitable proportion of resinous wood, either ground or in the shape of sawdust. The mixture is diluted with water, and from 5—10 lb. of sulphuric acid are added for every 100 lb. of material taken. The liquid mass is then run into a converter and heated for one hour to 300° F. The sulphuric acid is now neutralised by means of chalk or other suitable material, and the neutralised liquid is decanted from the precipitate and fermented and distilled in the usual way. The spirit obtained is ready for use as methylated spirit. Another process is to treat the starchy meal and the wood flour separately with sulphuric acid, the saccharine solutions being subsequently mixed and fermented. Or, the solutions may be separately fermented, and the two kinds of spirit obtained mixed in suitable proportions to form methylated spirit. For the production of charcoal and manure the by-products are subjected to powerful pressure, and then finely ground. The meal is calcined if charcoal be required, or mixed with other manurial substances, such as nitrates, phosphates, &c. if manure be sought for.—H. T. P.

*A New or Improved Process and Apparatus for the Distillation and Rectification of Alcohols.* W. T. Whiteman, London. From F. Haeck, Brussels, Belgium. Eng. Pat. 6587, April 29, 1889. 8d.

THE must is first filtered through a series of conical filters constructed of a cloth called by the French "Molleton" and lined with paper pulp. If the must contain more than 30 per cent. of alcohol it must be reduced to this strength by dilution before filtration.

The distilling apparatus used consists of a boiler heated by naked steam, a distillation column placed over the boiler and surrounded by a water jacket which may be kept at any desired temperature, and a refrigerator.

The filtered must is led into the boiler and its temperature raised to 22°—24° C.; the distillation column is also kept at this temperature.

The aldehyde evaporates from the liquid in the boiler and passes through the column to the refrigerator where it is condensed and runs into a receiver. Any other substances which may have been carried off from the liquid in the boiler with the aldehyde and have been condensed in the column do not flow back into the boiler but into a special receiver.

When all the aldehyde has passed over, the temperature of the boiler and column is raised to 53°—56° C. The ordinary ethers present in the must now evaporate and traversing the column without condensation, pass into the refrigerator and thence into a receiver; in the same way as before, any higher boiling bodies which may have been carried into the column with the ethers and there condensed pass into a special receiver.

As propyl, butyl, and amyl alcohols (the disagreeable end products) are but slightly soluble in 30 per cent. alcohol and are lighter than alcohol of this strength, they float on the surface of the liquid in the boiler. The vapours which, as above described, have been distilled off at temperatures of 22° and 54° C., having thus to pass through this layer of alcohols floating on the surface of the liquid in the boiler, naturally carry off part of these with them into the column where, as has been mentioned, these latter are condensed and flow from the bottom of the column into a special receiver. This receiver is so arranged that the lighter portion of the liquid flowing into it (propyl, amyl, and butyl alcohols) is mechanically separated from the heavier portion (solution of ethyl alcohol in water), the latter being led back into the still. When no more distils over at 53°—56° C., the temperature of the boiler and column is raised to 73°—76° C., at which temperature the acetic ether evaporates and passing the column is condensed in the refrigerator. In the same way as before any bodies of higher boiling point which may have passed into the column are led into the special separating receiver. When no more ether passes over, the temperature of the boiler is raised to 85° C., and that of the column is reduced to 50° C. and the distillation continued.

When no more end products are separated from the receiver above described, the distillation is stopped and the



must run into another boiler furnished with a distillation column, the same as the first boiler but larger, and a refrigerator. The column is kept at a temperature of  $78^{\circ}\text{C}$ . by the water jacket, and the must in the boiler raised to  $85^{\circ}$ – $88^{\circ}\text{C}$ . Pure ethyl alcohol passes through the column and is condensed in the refrigerator. The water, &c. condensed in the column passes back into the boiler. The alcohol from the refrigerator is at first 97 per cent.; when the strength falls to 95 per cent. the receiver is changed, the water in the jacket of the column run away and the contents of the boiler boiled; a brandy of 42–43 per cent. and of great aromatic richness is condensed by the refrigerator.

The alcohols thus obtained contain poisonous gases. These are expelled by heating the alcohol to  $32^{\circ}\text{C}$ . in vessels provided with a condenser for 2–3 hours, during which time these gases will have been estimated.

There are many little details in the apparatus and processes which are not described here and for which reference must be made to the complete specification.—A. L. S.

*Improvements in Stills of the Coffey Type.* A. Chapman, Liverpool. Eng. Pat. 8147, May 24, 1890. 8d.

In Coffey stills the water condensed in the rectifier forms a layer about one inch in depth on each of the perforated diaphragms, and the degree of rectification obtained depends on their number. In Coffey stills as ordinarily made, these diaphragms are fixtures; and consequently the strength of the spirit cannot be altered without injuring its quality.

By this invention, one or more of the diaphragms in the rectifier can be thrown out of action, so far as having a layer of liquid on the top is concerned, without interfering with the flow of the liquid, or with the action of the respective water-seals which prevent the alcohol and water vapour passing up the down pipes. Several ways of effecting this in practice are suggested; but the most convenient is to construct that portion of the down pipe which projects (about one inch) above the diaphragm, in the form of an annular valve or ring, fitting over the opening in the diaphragm and sufficiently weighted to keep in its place; this valve can be lifted clear of the opening in the diaphragm by means of a lever actuated from the outside of the column. The down pipes themselves have an out-turned flange at top by means of which they are riveted to the under-side of the diaphragm. As long as the valve rests on the diaphragm the water-level remains normal (about one inch); but as soon as the valve is raised the water escapes underneath it and passes into the down pipe, thus practically putting the diaphragm out of action and leading to the production of weaker spirit.—H. T. P.

## XVIII.—CHEMISTRY OF FOODS, SANITARY CHEMISTRY, AND DISINFECTANTS.

### (A).—CHEMISTRY OF FOOD.

#### PATENTS.

*An Improved Process of Treating Bran and other Cereal Substances for obtaining Sugar, Starch, Food for Animals, Gum, and Paper.* E. Steiger, E. Schulze, and C. Auer-Schollenberger, Zurich, Switzerland. Eng. Pat. 12,920, August 15, 1889. 6d.

See under XVI., page 878.

*Improvements in the Manufacture of Bread.* J. H. Besnard, Paris, France. Eng. Pat. 651, January 14, 1890. 4d.

LEAVEN of wheat or barley flour is added to dough of half hard flour, suitably salted. After the dough has risen

somewhat it is divided into lumps, put into perforated moulds, which are then closed and baked in an oven at  $110^{\circ}$ . The rising continues and the bread is thus baked under pressure.

This process produces a bread which is more consistent, less brittle, and more preservable than bread which is baked in the usual ways.—A. G. B.

### (B).—SANITARY CHEMISTRY.

#### PATENTS.

*Improvements in the Means Employed for Purifying Sewage and other Waters, and for Deodorising and Utilising the Solids and Excreta.* B. Jagger, H. B. Jagger, and A. Turley, Leeds. Eng. Pat. 5753, April 4, 1889. 6d.

ASH-RIT, town, or manufacturers' refuse, or waste products of any kind, which contain animal or vegetable matter, is carbonised in any suitable kiln or furnace. It is then broken or ground according to the purpose to which it is applied. Mixed with sewage, &c. either with or without some defecating agent, it is used to precipitate the impurities therein, the residue deposited forming a valuable manure. It may also be used either alone or in conjunction with a precipitation process as a purifying filter, through which impure or partially purified sewage may be passed. Where the pail system is employed the substance may be employed with advantage for the deodorisation of excreta.—C. C. H.

*Improvements in the Clarification or Purification of Sewage and other Foul Waters of Analogous Nature.* C. H. Harvey, London. Eng. Pat. 6994, April 26, 1889. 6d.

AN acid solution of a ferric salt is prepared by dissolving burnt pyrites, &c. or some such cheap oxide of iron in hydrochloric acid, the specific gravity of the solution being about 1.275. Soda waste (black-ash waste) or gas lime is well oxidised by exposure to the atmosphere for three or four weeks and ground to a powder. To every million gallons of sewage 1,000 lb. of the powdered waste is added and mixed therewith, followed by the addition of 62.5 gallons of the iron salt solution. Sewage so treated is rapidly and effectually clarified.—C. C. H.

*Improvements in Apparatus for Purifying Water and other Liquids.* H. Desrumaux, Brussels, Belgium. Eng. Pat. 7006, April 26, 1889. 8d.

THE specification describes and illustrates an automatic apparatus for the purposes above mentioned, and for details the specification must be referred to.—C. C. H.

*A New Apparatus for Removing, Purifying, and Freeing Noxious Gases and Vapours from Public and Private Sewers, Tunnels, Mines, Factories, Buildings, and all Places containing Impure Gases.* G. Smith, Hammer-smith. Eng. Pat. 7924, May 13, 1889. 8d.

THE new apparatus consists of two gasometers placed side by side and connected to the sewer by means of suction pipes and valves. These are alternately raised and lowered automatically by means of an engine and intermediate mechanism, which also at the proper times opens and shuts the suction and delivery valves. The foul air is delivered into a main communicating with a furnace through which it is forced and thus completely purified before discharge into the atmosphere.—C. C. H.

*Improvements in and Connected with the Purification and Filtration of Sewage and other Foul Liquids.* C. H. Beloe and F. Candy, London. Eng. Pat. 8088, May 15, 1889. 11d.

THIS specification describes a combination of sewage precipitation tanks and filters, the effluent from the tanks being

passed through a pair of filters worked side by side. The filtering bed consists of the material called "polarite" and the arrangement of the filters is such that when the bed of one has become foul, the filtered liquid from the other of the pair is conducted to the under side of the foul filter bed, and passing upward through it removes the scum and impurities originally deposited, which are carried back again to one of the empty precipitating tanks for deposition. Several modifications of the different elements of the combination are shown.—C. C. H.

*Improvements in the Treatment of Sewage, Fecal Matter, Foul Waters, or the Like, for the Purification or Deodorising thereof, and the Obtaining of Products therefrom.* B. Nicholson and T. Palmer, London. Eng. Pat. 9777, June 13, 1889. 6d.

THE sewage is treated with a mixture of slaked lime, calcium sulphate, clay, or aluminium sulphate, to which has also been added in the proportion of one-sixth of the combined weights of the lime and calcium sulphate, such a material as powdered coke, oxide of iron, spongy iron, the ashes of wood or peat, and sulphur. The sludge is separated by decantation and incinerated in a furnace; the gases given off are condensed; the incinerated sludge mixed with a fresh quantity of reagents may be again used as a precipitant. The effluent can be rendered innocuous by the addition of the aqueous solution of the gases condensed by the use of iodide of mercury, sanitas, carbolic acid or other suitable disinfectant. The burnt residue may finally be mixed with salts of iron or chromium and by fusion rendered into a pigment of any shade or colour.—C. C. H.

*Improvements in the Treatment of Sewage for the Purification thereof and the Production of Valuable Products therefrom.* C. G. Moor, Oxford. Eng. Pat. 11,798, July 24, 1889. 8d.

SLUDGE resulting from the treatment of sewage is incinerated as hereafter described and subsequently used in conjunction with sulphate of magnesium for the defecation of a further quantity of sewage. The sludge may be partially dried by exposure to air on a bed of porous material (preferably carbonised or incinerated sludge), or by means of filter presses. When dried down to contain about 25 per cent. of moisture it is burned under a forced draught in a furnace provided with a charging door, revolving grate, and draw-off door, but otherwise resembling an ordinary cupola or closed limekiln. The ammonia given off under destructive distillation in the furnace is condensed and made into sulphate of ammonia in the usual way; this is finally mixed with the carbonised ash from the sludge and forms a valuable manure.—C. C. H.

*Improved Materials for Purifying Sewage and Polluted Waters from Works or Manufactories.* T. Jones, Sedgley, and J. Jones, Wolverhampton. Eng. Pat. 14,500, September 14, 1889. 4d.

FIRECLAY shales or marls, and lime, are mixed with the sewage or polluted water as it comes from the sewers or works. The whole is then run into settling tanks. About 3 ewt. of fireclay shale or marl and 3 ewt. of lime are used to 36,000 galls. of the liquid to be purified.—A. G. B.

*An Improvement in the Means and Proportions of Materials Used for the Deodorisation, Evaporation, and, to a certain extent, Decomposition of Human Excrement, and its Transformation into a nearly Inodorous and Valuable Fertiliser.* C. W. Doughty, Augusta, Georgia, U.S.A. Eng. Pat. 2238, February 11, 1890. 4d.

CALCAREOUS marl dried at 400° F. and finely ground is used in privy boxes for the reception of the excreta and urine. The contents of the boxes are disinfected on removal by the addition of a few pounds of a mixture containing calcareous marl 391 parts, sulphate of iron 8 parts, chloride

of lime, 1 part. The compost so obtained is dried at a temperature of 250° F. and may either be used as a manure or re-used, and so fortified further, in the privy boxes. The patentee states the calcareous marl described as prepared has the power of combining with and fixing putrescible matter and preventing decomposition.—C. C. H.

## (C)—DISINFECTANTS.

### PATENTS.

*Improvements in Bleaching, Disinfecting, Purifying and Preserving Substances, and Apparatus therefor.* G. Brownen, London, and J. Gamgee, Wimbledon. Eng. Pat. 9763, June 13, 1889. 8d.

THE articles to be bleached or disinfected are moistened and placed upon perforated or "gridiron" shelves in a closed receptacle, the shelves being so arranged that the current of gas subsequently admitted passes backwards and forwards between the shelves before leaving the apparatus. At the end of and beneath the lower shelf under a suitable cover there is a lamp in which is burned a carbon bisulphide solution of sulphur. The products of combustion, which consist of the dioxides of sulphur and carbon, pass into the interior of the apparatus and so accomplish the desired effect. In some instances it may be desirable to cool the gas before it enters the bleaching chamber; this may conveniently be effected by means of a Liebig condenser. An asbestos wick is used in the lamp.—C. C. H.

*A Method and Apparatus Employed therein for Destroying the Germs of Infectious Maladies.* A. Wassmuth, Barmen, Germany. Eng. Pat. 14,562, September 16, 1889. 8d.

THIS method consists in scattering the medicine, which is to be inhaled by the patient, in the form of fine spray throughout the room; e.g., a solution is used containing 5 per cent. of "natrium chloroborosum," which is a salt composed of 9 per cent. of chlorine, 17 per cent. of sodium, 27 per cent. of boracic acid, and 47 per cent. of water.

—A. G. B.

## XIX.—PAPER, PASTEBOARD, Etc.

*The Behaviour of Wood and Cellulose at High Temperatures and Pressures in Presence of Caustic Soda.* H. Tauss. Dingl. Polyt. J. 276, 411—428.

THE author has shown in the first part of his experiments on cellulose and wood (see Dingl. Polyt. J. 273, 276—285; this Journal, 1889, 913—914), that woody substance in considerable quantity is dissolved by distilled water. Little is yet known about the relations of wood and cellulose to caustic soda, although caustic soda has been used in the manufacture of cellulose for a considerable time past. The objects of the author's experiments were to show to what extent cellulose and wood were affected and dissolved by caustic soda at different temperatures. The experiments were partly made with solutions of a concentration as is usually employed in practice, partly with more concentrated and more diluted solutions. The time of boiling was always three hours. The heating was effected at the ordinary pressure in glass vessels; for higher pressures Muecke's digester was used, but as copper is affected by alkaline solutions, iron parts were used instead of copper ones in the digester. The solutions were allowed to stand, decanted, the subsiding part filtered, well washed, dried with the filter, and weighed. The filtrates were diluted to a known volume, generally one litre, and were tested:—

1. For substances which reduce Fehling's solution (at ordinary and at higher temperatures).

2. For substances which are precipitated by an equal volume of alcohol.

3. For substances which are precipitated by acids.

4. For substances which are extracted by ether.

5. For substances which are extracted by ether after the filtrate has been acidified.

A quantitative determination of the precipitates obtained by acids and alcohol was made when possible. Those parts of the filtrate of the woody substance reducing Fehling's solution are certain kinds of sugar or similar products, those precipitated by alcohol, wood gum (according to Koch, Thomson, Weehler, and Tollens), those precipitated by acids, ulmic and resinous acids, &c.

The ether extracts gave colourations with phloroglucinol and hydrochloric acid; vanillin was eventually obtained.

The filtrates obtained by boiling cellulose with caustic soda may also contain substances reducing Fehling's solution, and, according to Koch, a compound with the formula  $4C_6H_{10}O_5NaOH$  is also precipitated by alcohol. These filtrates were also precipitated by acids and extracted with ether. The substances used for the experiments were pure Swedish filter-paper and fine chips of soft pine wood and hard beech wood. The substances were taken in an air-dried condition and the calculations made accordingly.

*I.—Boiling of Cellulose and Wood with Caustic Soda Solution of 1.09 sp. gr. (8 per cent. NaOH) at the Ordinary Pressure.*

Ten grms. of cellulose boiled three times, each time with 500 cc. of caustic soda solution, gave:—

—	1.	2.	3.	Total.	Per Cent.
Insoluble .....	8.301	7.814	7.721	..	..
Soluble .....	1.699	0.487	0.093	2.199	21.99

After the first boiling the solution was of a yellowish colour and gave no precipitate with alcohol or acid. The ether extract of the acidified liquid gave, after evaporation of the ether, a very small yellowish residue which showed no colouration with phloroglucinol and hydrochloric acid. The solutions of the other boilings gave similar reactions.

Ten grms. of soft wood, treated as before, gave:—

—	1.	2.	3.	Total.	Per Cent.
Insoluble .....	6.6105	5.926	5.480	..	..
Soluble .....	3.3895	0.6845	0.446	4.9195	49.19

Two more boilings of the same substance gave a still greater loss, viz.:—

—	4.	5.
Insoluble .....	5.157	4.863
Soluble .....	0.323	0.294

After the first boiling the solution was brown coloured; alcohol or acid precipitated only very minute quantities. The ether extract of the acidified liquid gave, after the evaporation of the ether, a yellow residue which showed a red colouration with phloroglucinol and hydrochloric acid. The solutions of the other boilings gave similar reactions.

Two grms. of hard wood, treated as before, gave:—

—	1.	2.	3.	Total.	Per Cent.
Insoluble .....	5.829	4.810	4.532	..	..
Soluble .....	4.171	1.019	0.279	5.469	54.69

After the first boiling the solution was brown coloured; alcohol precipitate (from 50 cc.) 0.137 grm. = 27.4 per cent., and acid (from 50 cc.) 0.030 grm. = 6 per cent. of the wood. The ether extract of the acidified liquid gave, after the evaporation of the ether, a residue which showed a red colouration with phloroglucinol and hydrochloric acid. The solutions of the other boilings gave similar reactions.

*II.—Boiling of Cellulose and Wood with Caustic Soda Solution of 1.09 sp. gr. (8 per cent. NaOH) at 5 Atmospheres Pressure.*

Ten grms. of cellulose boiled three times with 500 cc. of caustic soda solution gave:—

—	1.	2.	3.	Total.	Per Cent.
Insoluble .....	5.548	4.624	4.198	..	..
Soluble .....	4.452	0.924	0.426	5.802	58.02

After the first boiling the solution was brown coloured; alcohol precipitated (from 50 cc.) 0.008 grm. = 1.6 per cent., and acid (from 50 cc.) 0.005 grm. = 1 per cent. of the cellulose. The ether extract of the acidified liquid gave, after the evaporation of the ether, no colouration with phloroglucinol and hydrochloric acid. The solutions of the other boilings gave similar reactions.

Ten grms. of soft wood, treated as before, gave:—

—	1.	2.	3.	Total.	Per Cent.
Insoluble .....	4.309	3.253	2.415	..	..
Soluble .....	5.691	1.056	0.838	7.585	75.85

After the first boiling the solution was deep brown; alcohol precipitated (from 50 cc.) 0.010 grm. = 2 per cent., and acid (from 50 cc.) 0.080 grm. = 16 per cent. of the wood. The ether extract of the acidified liquid, after the evaporation of the ether, was of a yellowish colour, and showed a red colouration with phloroglucinol and hydrochloric acid. The solutions of the other boilings behaved similarly.

Ten grms. of hard wood, treated as before, gave:—

—	1.	2.	3.	Total.	Per Cent.
Insoluble .....	3.564	3.277	3.020	..	..
Soluble .....	6.436	0.287	0.257	6.980	69.80

After the first boiling the solution was deep brown; alcohol precipitated (from 50 cc.) 0.178 grm. = 35.6 per cent., and acid (from 50 cc.) 0.076 grm. = 15.2 per cent. of the wood. The ether extract of the acidified liquid, after the evaporation of the ether, was of a yellowish colour, and gave a red colouration with phloroglucinol and hydrochloric acid. The solutions of the other boilings gave similar reactions.

*III.—Boiling of Cellulose and Wood with Caustic Soda Solution of 1.09 sp. gr. (8 per cent. NaOH) at 10 Atmospheres Pressure.*

Ten grms. of cellulose, boiled three times with 500 cc. of caustic soda solution, gave:—

—	1.	2.	3.	Total.	Per Cent.
Insoluble .....	4.971	4.320	4.102	..	..
Soluble .....	5.029	0.651	0.218	5.899	58.99

After the first boiling the solution was brown; alcohol precipitated (from 50 cc.) 0.0515 grm. = 10.30 per cent., and acid (from 50 cc.) 0.0365 grm. = 7.3 per cent. of the

cellulose. The ether extract of the acidified liquid, after the evaporation of the ether, was of a brown colour, and gave no colouration with phloroglucinol and hydrochloric acid. The solutions of the other boilings gave similar reactions.

Ten grms. of soft wood, treated as before, gave:—

—	1.	2.	3.	Total.	Per Cent.
Insoluble .....	2.5675	2.111	1.820	..	..
Soluble .....	7.4325	0.4565	0.291	8.180	81.80

After the first boiling the solution was dark brown; alcohol precipitated (from 50 cc.) 0.0087 gm. = 1.7 per cent., and acid (from 50 cc.) 0.127 gm. = 25.40 per cent. of the wood. The ether extract of the acidified liquid, after the evaporation of the ether, was of a brown colour, and gave no colouration with phloroglucinol and hydrochloric acid. The solutions of the other boilings gave similar reactions.

Ten grms. of hard wood, treated as before, gave:—

—	1.	2.	3.	Total.	Per Cent.
Insoluble .....	2.934	2.492	2.061	..	..
Soluble .....	7.066	0.532	0.341	7.939	79.39

After the first boiling the solution was dark brown; alcohol precipitated (from 50 cc.) 0.0026 gm. = 0.5 per cent., and acid (from 50 cc.) 0.092 gm. = 18.4 per cent. of the wood. The ether extract of the acidified liquid gave a brown residue, which showed no colouration with phloroglucinol and hydrochloric acid. The solutions of the other boilings gave similar reactions.

#### IV.—Boiling of Cellulose and Wood with Caustic Soda Solution of 1.162 sp. gr. (14 per cent. NaOH) at the Ordinary Pressure.

Ten grms. of cellulose, boiled with 500 cc. of caustic soda solution, gave:—

Insoluble.....	7.812
Soluble .....	2.188 = 21.88 per cent.

Solution, yellow; alcohol precipitated (from 50 cc.) 0.039 gm. = 7.8 per cent. of the cellulose, only a minute quantity was precipitated by acid. The ether extract of the acidified liquid gave a yellowish residue, which showed no colouration with phloroglucinol and hydrochloric acid.

Ten grms. of soft wood, treated as before, gave:—

Insoluble.....	6.455
Soluble .....	3.545 = 35.45 per cent.

Solution, brown; alcohol precipitated (from 50 cc.) 0.024 gm. = 4.8 per cent., and acid (from 50 cc.) 0.010 gm. = 2 per cent. of the wood. The ether extract of the acidified liquid gave a brown residue, which showed no colouration with phloroglucinol and hydrochloric acid.

Ten grms. of hard wood, treated as before, gave:—

Insoluble.....	5.357
Soluble .....	4.643 = 46.43 per cent.

Solution, brown; alcohol precipitated (from 50 cc.) 0.054 gm. = 10.8 per cent., and acid (from 50 cc.) 0.028 gm. = 5.6 per cent. of the wood. The ether extract of the acidified liquid gave a yellow residue, which showed no colouration with phloroglucinol and hydrochloric acid.

#### V.—Boiling of Cellulose and Wood with Caustic Soda Solution of 1.162 sp. gr. (14 per cent. NaOH) at 5 Atmospheres Pressure.

Ten grms. of cellulose, boiled with 500 cc. of caustic soda solution, gave:—

Insoluble.....	2.267
Soluble .....	7.733 = 77.33 per cent.

Solution, brown; alcohol precipitated (from 50 cc.) 0.135 gm. = 27 per cent., and acid (from 50 cc.) 0.059 gm. = 11.80 per cent. of the cellulose. The ether extract of the acidified liquid gave yellow-brown residue, which showed no colouration with phloroglucinol and hydrochloric acid.

Ten grms. of soft wood, treated as before, gave:—

Insoluble.....	0.287
Soluble .....	9.713 = 97.13 per cent.

Solution, dark brown; alcohol precipitated (from 50 cc.) 0.134 gm. = 26.8 per cent., and acid (from 50 cc.) 0.124 gm. = 24.8 per cent. of the wood. The ether extract of the acidified liquid gave a brown residue, which showed a red-violet colouration with phloroglucinol and hydrochloric acid.

Ten grms. of hard wood, treated as before, gave:—

Insoluble.....	0.852
Soluble.....	9.148 = 91.48 per cent.

Solution, dark brown; alcohol precipitated (from 50 cc.) 0.079 gm. = 15.8 per cent., and acid (from 50 cc.) 0.168 gm. = 33.60 per cent. of the wood. The ether extract of the acidified liquid gave a yellow residue, which showed no colouration with phloroglucinol and hydrochloric acid.

#### VI.—Boiling of Cellulose and Wood with Caustic Soda Solution of 1.013 sp. gr. (3 per cent. NaOH) at the Ordinary Pressure.

Ten grms. of cellulose, boiled with 500 cc. of caustic soda solution, gave:—

Insoluble.....	8.793
Soluble .....	1.207 = 12.07 per cent.

Solution, yellow; gave with alcohol or acid no precipitate. The ether extract of the acidified liquid gave no residue.

Ten grms. of soft wood, treated as before, gave:—

Insoluble.....	7.163
Soluble .....	2.837 = 28.37 per cent.

Solution, brown; alcohol precipitated nothing, and acid (from 50 cc.) 0.0065 gm. = 1.31 per cent. of the wood. The ether extract of the acidified liquid gave a yellow residue, which showed a red colouration with phloroglucinol and hydrochloric acid.

Ten grms. of hard wood, treated as before, gave:—

Insoluble.....	6.975
Soluble.....	3.025 = 30.25 per cent.

Solution, brown; alcohol precipitated (from 50 cc.) 0.065 gm. = 13 per cent., and acid (from 50 cc.) 0.027 gm. = 5.4 per cent. of the wood. The ether extract of the acidified liquid gave a brown residue, which showed no colouration with phloroglucinol and hydrochloric acid.

#### VII.—Boiling of Cellulose and Wood with Caustic Soda Solution of 1.043 sp. gr. (3 per cent. NaOH) at 5 Atmospheres Pressure.

Ten grms. of cellulose, boiled with 500 cc. of caustic soda solution, gave:—

Insoluble.....	8.464
Soluble .....	1.536 = 15.36 per cent.

Solution, yellowish; alcohol precipitated nothing, acid almost nothing. The ether extract of the acidified liquid gave a trace of a yellow residue, which showed no colouration with phloroglucinol and hydrochloric acid.

Ten grms. of soft wood, treated as before, gave:—

Insoluble.....	4.904
Soluble.....	5.096 = 50.96 per cent.

Solution, brown; alcohol precipitated nothing, acid (from 50 cc.) 0.0797 gm. = 15.94 per cent. of the wood. The ether extract of the acidified liquid gave a yellow-brown residue, which showed a red colouration with phloroglucinol and hydrochloric acid.

Ten grms. of hard wood, treated as before, gave:—

Insoluble.....	4.434
Soluble .....	5.566 = 55.66 per cent.

Solution, deep brown; alcohol precipitated (from 50 cc.) 0.011 gm. = 2.2 per cent., and acid (from 50 cc.) 0.047 gm. = 9.4 per cent. of the wood. The ether extract of the acidified liquid gave a yellow-brown residue, which showed a red colouration with phloroglucinol and hydrochloric acid.

*VIII.—Boiling of Cellulose and Wood with Caustic Soda Solution of 1.043 sp. gr (3 per cent. NaOH) at 10 Atmospheres Pressure.*

Ten grms. of cellulose, boiled with 500 cc. of caustic soda solution, gave:—

Insoluble.....	7.972
Soluble .....	2.028 = 20.28 per cent.

Solution, yellow; alcohol precipitated (from 50 cc.) 0.043 gm. = 8.6 per cent., and acid (from 50 cc.) 0.021 gm. = 4.2 per cent. of the cellulose. The ether extract of the acidified liquid gave a yellow residue, which showed no colouration with phloroglucinol and hydrochloric acid.

Ten grms. of soft wood, treated as before, gave:—

Insoluble.....	2.969
Soluble.....	7.031 = 70.31 per cent.

Solution, yellow-brown; alcohol precipitated a minute quantity, and acid (from 50 cc.) 0.080 gm. = 16 per cent. of the wood. The ether extract of the acidified liquid gave a yellow residue, which showed no colouration with phloroglucinol and hydrochloric acid.

Ten grms. of hard wood, treated as before, gave:—

Insoluble.....	3.441
Soluble .....	6.559 = 65.59 per cent.

Solution, brown; alcohol precipitated a minute quantity, and acid (from 50 cc.) 0.070 gm. = 14 per cent. of the wood. The ether extract of the acidified liquid gave a brown residue, which showed no colouration with phloroglucinol and hydrochloric acid.

All the solutions were treated with Fehling's solution, but none of them gave a reaction. The direct extracts with ether (without acidifying the liquid) were likewise without any result.

In all experiments higher pressure and greater concentration of the alkaline solution employed, increased the solvent power of the latter.

If it be assumed that soft wood (pine wood) contain 54 per cent. of cellulose and 46 per cent. of other matters, these 46 per cent. of other substances will be dissolved after three hours' boiling by an alkaline solution of 1.043 sp. gr. (3 per cent. NaOH) at a pressure of five to six atmospheres, by an alkaline solution of 1.090 sp. gr. (8 per cent. NaOH) at a pressure of four to five atmospheres, and by an alkaline solution of 1.162 sp. gr. (14 per cent. NaOH) at a pressure of two to three atmospheres. But, if the boiling be repeated, ordinary pressure will be sufficient if an alkaline solution of 1.090 sp. gr. (8 per cent. NaOH) be used. The same rules may be applied to hard wood (beech wood), which however contains 46 per cent. of cellulose and 54 per cent. of other matters.

But a much higher pressure is often applied for technical purposes. This is done to facilitate the mechanical loosening of the fibres, but then it cannot be avoided that in the dissolving of the other substances much cellulose is lost, and in many cases only 30 or 35 per cent. of cellulose are obtained.—H. S.

*On the Use of the Edge-Runner in Paper Mills.*

E. Muth. Dingl. Polyt. J. 276, 506—513.

In paper manufacture the edge-runner not only serves to render the working of the material easier, but it also improves the article. One of the chief objects of this

machine is to disintegrate "broke"-paper. The several stones used in the construction of the edge-runner are mostly of fine-grained granite and sandstone, the bottom stones being granite and the revolving stones sandstone. Iron plates are sometimes used instead of the bottom stones, but without any special advantage. The sandstones used must be as homogeneous as possible. The two flat sides of the sandstones are generally roughly hewn, but the author thinks it would be better to have them of a smoother surface in order to prevent any loosening of the sand particles of which the stone consists, and the mixture of them with the material. The substance used for lubricating the shaft often falls in drops, and spoils the material being worked, and therefore lubricating substances of a non-liquid nature have been recommended, but this is only a partial remedy, for as soon as the machine gets warm, such substances melt. A good remedy seems to be to use a box containing hemp saturated with tallow. Mistakes are often made in fixing the edge-runner at unsuitable and dark places in the mill. It should be placed next to the beaters and always be kept as clean as possible, as well as situated in a light spot where the state of the material can be easily ascertained, and the exact time when it has to be taken out to be transferred to the beaters. In working "broke"-paper with the edge-runner, such kinds of paper as are glazed and sized in a high degree require especially careful treatment. Formerly these kinds of paper could be disintegrated only after having been boiled, and could only be employed for an inferior quality of that, but now boiling is no longer necessary, and hence they can be used in making the same kind of paper as that of which they were waste. But to produce the higher quality, the various impurities of "broke"-paper should be removed as far as possible previous to their treatment with the edge-runner. Unsized "broke"-paper can be disintegrated without any difficulty with the edge-runner by moistening it with a little water. The best way is to bring the material dry into the machine and to regulate the water supply from a small tap connected with the machine. Medium sized paper must be moistened before being brought into the edge-runner, whereas paper glazed and sized in a high degree must be soaked for some time in water of 40° to 50° C. Entire sheets should be torn before being placed in the warm water, because they would offer too much resistance to the stones of the edge-runner.

The proper quantity of water is of great importance for the more or less complete disintegration of "broke"-papers. But according to the sizing, the fibres are different in their tenacity and the moistening of the material previous to its being brought into the edge-runner, is not sufficient for paper sized in a high degree. But this has to be effected by a careful regulation of the water supply whilst the paper is in the edge-runner. As long as the material is not moist enough, the supply of fresh material must be slow to avoid stoppage. Should, however, a stoppage occur, it is best to empty the whole machine, and to divide the compressed parts mechanically. Attempts to lift the stones without removing the material in most cases fail, and the risk is run of spoiling the material. Too much water is just as bad as too little in working with the edge-runner. The proper amount is added if little water be given forth from a small quantity of the material squeezed in the hand.

The disintegration is more effected by the continuous motion and friction of the material itself than by the direct action of the stones. To use rough surfaced stones for the machine is therefore without value, as the unevenness is soon filled by the disintegrated material. This friction of the material itself explains the importance of the proper amount of the water supply, too much or too little water preventing the desired friction. It is of advantage not to empty the edge-runner entirely when new material is put in, but to leave a thin layer at the bottom in order to prevent the direct contact of the bottom stones with the revolving stones. Experience only can show when the material is ready to be taken out from the edge-runner and removed to the beater, it not being possible to apply any rule for this. If the edge-runner be emptied the material should be placed in a wooden box, which is afterwards covered with a wet piece of felt, to prevent the material from getting dry and

spoiled by impurities. It is best to continue working the material; it is always a disadvantage to let it stand. The chief use of the edge-runner is for the disintegration of "broke"-paper, but it may also be employed for wood, straw, &c. As the disintegration of these substances is preceded by boiling, the work which the edge-runner has to do in these cases is much easier than for "broke"-paper. No other machine at present exists for this treatment of "broke"-paper than the edge-runner. (See also this Journal, 1890, 320).—H. S.

*Sulphite-Pulp Boiling.* Chem. Trade J. 1890, 7, 174—176.

AN anonymous writer in the *Papier Zeitung* of September 7th, 1890, refers to the experiments of Mr. Thilmann, Kaukauna, U.S.A., on the treatment of various kinds of wood in the bisulphite-pulp manufacture (this Journal, 1890, 540), and gives a report of laboratory experiments made in the same direction with sulphite lyes, of pure aqueous solution of sulphurous acid, and of aqueous solutions of the sulphites of calcium, barium, magnesium, sodium and potassium upon wood and other raw fibrous materials. With the exception of the "*Pinus maritima*," and the sugar- and Spanish-cane (*Saccharum officinarum* and *Calamus rotundus*), the other woods and fibrous plants were all grown on German soil. The tables given show that most of the experiments were carried out at a temperature above 100° C. In each test the wood and lye were placed in a glass tube hermetically sealed, and then heated to the required temperature in an oven. The experiments made below 100° C. were made in small glass-stoppered tubes, and heated in a water-bath. From the results obtained the author arrives at the following conclusions:—

In the boilings carried on below 100° C., all the wood samples remained hard and tough. The other fibres, those of flax, straw, &c., were only half soft. In those experiments conducted above 100° C., the woods treated with pure aqueous sulphurous acid were brown, and all tests besides *Abies excelsa*, *Populus*, and sugar cane, *Saccharum officinarum*, remained hard or were brittle. But the woods treated with metallic sulphite lyes were mostly white. With calcium sulphite lye nearly all the woods were soft, white, and sufficiently boiled. Excepting *Abies excelsa* the whitest was the *populus*, whilst *Larix decidua* (larch, a strong resinous wood) remained tough. *Pinus maritima* always remained hard, and yielded a strongly aromatic odour and a sticky or viscous lye. Oak wood is, as is well known, treated with difficulty by the bisulphite methods. With magnesium, potassium, and sodium sulphite lyes, the wood remained almost always soft, or tolerably so, whilst with weak barium sulphite lye the woods were tough and hard. With this reagent, the *populus* was easily boiled, yielding soft white fibre.

#### PATENTS.

*An Improved Process of Treating Bran and other Cereal Substances for obtaining Sugar, Starch, Food for Animals, Gum, and Paper.* E. Steiger, E. Schulze, and C. Auer-Schollenberger, Zurich, Switzerland. Eng. Pat. 12,920, August 15, 1889. 6d.

See under XVI., page 878.

*Improvements in the Method of Preparing Colours for making Printing Inks.* R. Ashton, Manchester. Eng. Pat. 14,388, September 12, 1889. 6d.

See under VI., page 857.

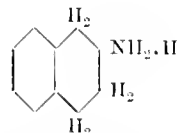
*Improved Process for making Paper, Linen, and Similar Material Impervious to Water.* A. J. Boulton, London. From D. MacDonald and W. T. Tassie, Toronto, Canada. Eng. Pat. 7024, May 6, 1890. 4d.

See under XIII., page 870.

## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

*The Relation between the Physiological Properties and the Constitution of Hydro-bases.* E. Bamberger and W. Fiehn. Ber. 22, 777—778.

Among the properties of the *ac-β*-Tetrahydronaphthylamine—



the most striking is the strong *mydriasis* produced by doses which produce absolutely no other or attendant symptoms. Stern has thoroughly studied this action ("*Ueber die Wirkung der Hydronaphthylamine auf den thierischen Organismus*," *Inaug. Dissert.* Breslau, 1888, and *Virchow's Archiv.* Bd. 115, 14.)

Indisputably the most interesting action of the *ac-β*-Tetrahydronaphthylamine is the considerable rise of temperature brought about in animals, this rise amounting to 4.5°.

Almost the whole group of the hydronaphthalene bases has been studied in the direction referred to.

The properties described as shown by the tetrahydro-*β*-naphthylamine, are shared by all the alicyclic hydro-bases of the *β*-series. These peculiar properties are apparently a condition of—

1. *The presence of additive hydrogen atoms in the substituted nucleus.*

2. *The β-position of the basic atomic group.*

Consequently the circle of the mydriatic and otherwise active substances is narrower than that to which the hydro-bases of aliphatic character belong. The latter comprises all the alicyclic bases; the former only those of the *β*-series.

The following table illustrates the fact referred to:—

Mydriatic and otherwise Active.	Non-Mydriatic or otherwise Active.
<i>ac.</i> Tetrahydro- <i>β</i> -naphthylamine.	<i>ac.</i> Tetrahydro- <i>α</i> -naphthylamine.
<i>ac.</i> Tetrahydro- <i>β</i> -naphthylamine.	<i>ar.</i> Tetrahydro-ethyl- <i>β</i> -naphthylamine.
<i>ac.</i> Tetrahydrodimethyl- <i>β</i> -naphthylamine.	<i>ar.</i> Tetrahydrodimethyl- <i>β</i> -naphthylamine.
	<i>ac.</i> Tetrahydro- <i>α'</i> - <i>α'</i> (1, 5) amidonaphthol.
	<i>ac.</i> Tetrahydro- <i>α'</i> - <i>α'</i> (1, 5) naphthylenediamine.
	<i>ar.</i> <i>α'</i> - <i>β'</i> (1, 2) tetrahydronaphthylenediamine.

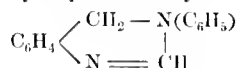
Thus the "carrier" representing the physiological function is that atomic group in the *β*-position—



It is even suggested that in order to the qualitative discrimination of the members of these groups that it would be most economical as regards expenditure of substance, as well as most certain as to result, to perform an experiment upon some small animal.

*Report on New Drugs and Fine Chemicals.* Merck's Bull. 1890 [5], 33—48; 1890 [6], 49—72; and 1890 [7], 73—88.

*Orexin* (phenyldihydroquinazoline hydrochloride)—



F. Penzoldt, of Erlangen University, has reported minutely as to physiological and therapeutical experiments made with this substance (Ther. Monatsh. 1890, 59).

Orexin taken internally is but very moderately toxic, in fact a dose to be decidedly toxic in the case of a man would amount to 25 grms. at least. Half a gm. produces in the healthy human subject a quickly-formed and strong appetite; indeed, to satisfy it, as a rule, the food has to be increased to twice its usual amount. Experiments showed that on the direct application of orexin to the mucous membranes of the conjunctiva and the stomach and to the subcutaneous cellular tissue, no escharotic topical action ensued.

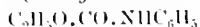
**Laudanine.**—This is one of the rarer natural opium bases, and has only recently been prepared on a commercial scale by Merck. The yield is so small that a quantity of mother-liquors capable of yielding a pound of *Cryptopine* yields only 5 oz. of *Laudanine*. *Laudanine* dissolves with tolerable ease in boiling alcohol, recrystallising on cooling in transparent granules. It melts at about 166° C. Pure concentrated sulphuric acid dissolves it at ordinary temperatures, giving a very faint pink tint; the same acid containing some ferric oxide yields but a slightly deeper tint. When these solutions are heated up to the point of evaporation of the acid, they assume a violet colour. The colour is, however, most intense in the ferruginous solution. *Laudanine* is a strong base forming well crystallised salts. It is soluble in soda or potash solutions, and thus resembles morphine. It differs, however, from morphine in that, whilst the morphine-soda compound can be crystallised only from a highly-concentrated and preferably alcoholic solution, the *laudanine*-soda compound may be precipitated from its aqueous solution even by an excess of the soda solution. It then appears in white resplendent needles. By indifferent solvents, such as chloroform or amyl alcohol, not a trace of the alkaloid can be removed from solutions of *laudanine* in the fixed alkalis. Hence *laudanine* possesses a phenolic character; a character made evident, moreover, by the well-known ferric chloride reaction. The experiment has been tried of methylating *laudanine* and so transforming it into an allied base, just as morphine may be changed into cocaine. Hesse pointed to the probability of the natural opium alkaloid, *laudanone* being the methyl-ether of *laudanine*. Actual experiment did not yield the result expected, for instead of the analogue of cocaine being formed from *laudanine*, a *laudanine* methyl-ether was obtained, not identical with Hesse's "*laudanone*." A close study of *laudanine* and its derivatives, chemically as well as physiologically, is commended as a subject of rich promise.

**Mercuric Benzoate**,  $(C_6H_5CO_2)_2Hg + aq.$ , is formed in small, white, inodorous and insipid crystals, but slightly soluble in cold, though more readily so in hot water and in alcohol. It is a powerful antiseptic, and is successfully employed in syphilitic and similar diseases.

**Methyl Guanidine**  $NH_2C(NH_2) \cdot NH(CH_3)$ , a colourless and very deliquescent substance of alkaline reaction, soluble in water but insoluble in alcohol. Methyl guanidine kills rabbits quickly, with symptoms of dyspnoea and convulsions.

**Mururé Oil** is obtained from the Brazilian plant *Bichetea officinalis* (*Urticaceæ*) and termed "*Mururé*." In Brazil this oil is extensively used in grave cases of syphilis, the ulcerations, it is said, being very rapidly healed by it. Moreover in rheumatic pains exceedingly favourable results were obtained with *Mururé* oil. The oil is also highly spoken of as an efficacious *emmenagogue*.

**Phenyl Prethane** (Phenyl carbaminic acid)—



occurs in white crystals, insoluble in water, easily soluble in dilute and also in strong alcohol. Melting point = 49° C. Giacomini (Deutsch. med. Zeit. 1889, 71) recommends it as a reliable antipyretic, which also exercises a favourable influence on the general tone of the system. In both acute and chronic *Arthritic rheumatism* it is useful, but then somewhat larger doses are required; the analgetic effect is, however, not fully reliable.

**Sodium Silico-fluoride** ("*Salufer*"),  $Na_2F_6 : SiF_6$ , first introduced by W. Thomson, is highly spoken of by Roberts (Deutsch. med. Zeit. 1889) who states that solutions as weak as 2:1000 are efficacious for surgical antiseptic purposes. It is also commended by various authorities as a styptic and astringent in dentistry.

**Tarine.**—This is the alkaloid of the leaves of *Tarus baccata* (yew). It appears in whitish laminae, with difficulty soluble in water, readily soluble in alcohol, ether, and carbon bisulphide. Its melting point is about 110° C.

This alkaloid has been so far only investigated physiologically. It is a powerful poison, the lethal effect being asphyxia with spasms.

"**Thermine**" is the tetrahydro- $\beta$ -naphthylamine of Bamberger and preceding abstract (Ber. 22, 777). It is a colourless, limpid, slightly viscous liquid, of peculiar odour. It has very strong basic power, even fixing carbonic acid firmly, so that a drop exposed to the air soon becomes a crystalline solid, in consequence of its transformation into the carbonate of the base.

As shown by Filelme, the physiological effects of *Thermine* embrace the two very strongly marked characteristics of *Mydriasis* and *elevation of animal temperature*. The latter effect has been observed to the extent of 4½° C. The name of *Thermine* was given on account of this peculiar action.

**Thermine Hydrochloride** (Tetrahydro- $\beta$ -naphthylamine hydrochloride),  $C_{10}H_{11}NH_2 \cdot HCl$ , forms white, well-defined crystals, readily soluble in water, alcohol and amyl alcohol. Melting point = 237° C. W. de Jong has demonstrated that the *Thermine* salts cannot replace cocaine in ophthalmology since their mydriatic action, whilst not much exceeding that of cocaine in intensity, is accompanied by marked pain.

**Thioresorcinol** forms a yellowish-grey, insipid powder, insoluble in water, and but sparingly soluble in alcohol and ether.

Thioresorcinol has been proposed as a succedaneum for iodoform, but it appears from more recent trials that it is not entirely innocuous, and requires caution in its application.

**Rubidium and Caesium Compounds.**—S. Botkin (Inaugural Dissert. St. Petersburg, 1888) has investigated the physiological action of the caesium and rubidium halogen salts, both on canine and human subjects. His experiments show that the chlorides of these metals injected into the circulatory system, augment the blood pressure (chiefly by vascular contraction), whilst they also retard the heart-movement to some extent. The effect, as regards heart-action, is but of subordinate character with the rubidium salts, and it is more insignificant still with the caesium compounds. Generally, the salts of both these metals appear to be closely related to those of the alkalis as to the character of their physiologic action.

In this regard, too, rubidium bromide has lately been the subject of study. Laufenauer (Therapeutische Monatsh. 1889, 348) first pointed out the remarkable relation of the atomic weight and electro-positivity of metals to the anti-epileptic power of their bromides, this power being generally greater in proportion as the afore-named properties are higher. Accordingly, the bromides of rubidium and of caesium ought to exert stronger anti-epileptic action than the bromides of potassium or of sodium. This conclusion has been verified by Laufenauer and Karlowsky (*Pesther med. chir. Presse*, 1889, No. 27). But rubidium bromide, with which these investigators experimented, being difficult of preparation, Laufenauer recommends the following for medicinal purposes:—

**Double Rubidium and Ammonium Bromide**,  $RbBr + 3 NH_4Br$ .—A white or slightly yellowish crystalline powder, readily soluble in water. Laufenauer and Karlowsky found that in nearly all the clinical varieties of epilepsy and allied ailments, its therapeutic action corresponds in the main with that of potassium bromide. In fully one-third the number of cases treated, however, the rubidium-ammonium bromide produced an indisputably greater sedative effect than any of



the allied bromine preparations. It was also useful as a hypnotic in other cases. Leufenauer is now investigating the therapeutic action of caesium bromide.

**Caesium-Rubidium-Ammonium Bromide (the Triple Salt).**—This is the latest anti-epileptic.

**Caesium and Ammonium Bromide (Double Salt),  $CsBr + 3NH_4Br$ .**—A white crystalline powder, readily soluble in water.

**Caesium Carbonate,  $Cs_2CO_3$ .**—A sand-like white substance, melting at a red heat, very hygroscopic, very soluble in water and also soluble in alcohol.

**Caesium Hydroxide,  $CsOH$ .**—A greyish white mass, melting below a red heat, rather deliquescent, and behaving towards water or alcohol as potassium hydroxide does.

**Caesium Sulphate,  $Cs_2SO_4$ .**—Anhydrous, colourless prisms, permanent in the atmosphere; far more readily soluble in water than the corresponding potassium salt; insoluble in alcohol.

**Cocaine Hydrochloride.**—The great importance of the freedom from amorphous coca bases is borne ample testimony to by the researches of Liebermann and Liebreich. Liebermann (Ber. 21, 2344; also this Journal, 1889, 305, 566—567) discovered among the amorphous accessory bases of coca leaves a very toxic substance, Isatropyl-cocaine. G. Falkson (Inaug. Dissert. 1889, Berlin) describes in detail the physiological action of this deadly alkaloid. The yield of pure crystallised cocaine from the crude commercial cocaines is much smaller than is generally thought. Moreover the complete separation from the amorphous bases is complicated, difficult, and involves considerable loss of material.

**Amylene Hydrate (pure Medicinal) versus Sulphonal.**—A comparison has been experimentally made between these two hypnotics with the result that the hypnotic effect of amylene hydrate is secured promptly after the lapse of half an hour, and lasts for 7—9 hours, the patient finding himself rested and refreshed on awaking in the morning. This gives a preference over sulphonal, the effect of which usually supervenes only after several hours, and frequently continues all through the following day in a disagreeable manner.

**Apo-atropine** is a colourless crystalline substance, but little soluble in water, readily soluble in alcohol, chloroform, and in benzene. Melting point,  $60^{\circ}$ — $62^{\circ}$  C. It is identical with the atropyl-tropine of Pesci and of Ladenburg.

**Benzanilide,  $C_6H_5NH(CO.C_6H_5)$**  (this Journal, 1889, 476).—The results of further experiments in proof of its usefulness as an antipyretic are given. It is a white, crystalline powder, almost insoluble in water, soluble in 58 parts of cold and seven parts of hot alcohol, and but little soluble in ether.

**Butyl-chloral Hydrate,  $C_4H_9Cl_3O + aq$**  (this Journal, 1889, 476), is found in white crystalline laminae of a peculiar odour, with difficulty soluble in cold, more easily in hot water; very readily soluble in alcohol. Melting point  $= 78^{\circ}$  C. Sp. gr. = 1.69.

**Caffeine Phthalate** has been exceedingly well adopted for hypodermic use on account of its ready and perfect solubility in a small quantity of water (1:5), the alkaloid itself only possessing one-fifteenth of that solubility.

**Chloral-Ammonium,  $CCl_3CH(NH_2).OH$**  (this Journal, 1889, 413), is a white crystalline powder of taste and odour resembling chloral hydrate, but little soluble in cold water, more readily so in alcohol. Melting point  $= 64^{\circ}$  C.

**Cubic Acid,  $C_{25}H_{30}O_7$**  (Schulze), is found in officinal cubebs associated with cubebene, an ethereal oil, and with cubebin, an indifferent solid. The acid is a white, wax-like substance, soon turning brown in the air. It is readily soluble in alcohol and ether. Its therapeutic value is variously estimated. According to Bernatzik (Husemann and Hilger's "Pflanzenstoffe," 492) the acid represents the anti-blennorrhagic principle of cubebs.

**Kephir and Koumiss.**—The kephir fungus or "seeds," which the nomadic tribes of Russia use in preparing the original kephir, have now been brought into commerce, and are obtainable through the drug trade. According to Kern they consist of two fungi of totally different character, the regular brewer's yeast (*saccharomyces cerevisiae*, Meyen) and a new bacillus which, from its tendency to emit spores at both ends, he terms *Dispora caucasica*. Associated with these there appear to be some other cells, principally of *Oidium lactis*, and it has not yet been ascertained whether these are essential or accidental. The prime origin of this composite ferment has not yet been ascertained. It is certain, however, that neither common yeast nor atmospheric spores can replace with equal effect, milk fermented by either never yielding a product endowed with the dietetic and therapeutic virtues of true kephir. The appearance of good and dry kephir fungi is that of light brown or yellowish, irregular aggregations of hard roundish or oval granules from about the size of a rye-grain to that of a pea, or even larger. The odour is similar to that of fermented pure cream, or of good fresh Swiss cheese. A whitish incrustation sometimes visible in the recesses of the grains of fungi is harmless, but if greenish, mould and therefore some deterioration, is indicated.

The preparation of true kephir is next described in detail, and then the dietetic virtues and action are chemically and physiologically discussed.

**Phenylacetic Acid,  $C_6H_5.CH_2CO_2H$  (Alpha-toluic Acid) and  $\beta$ -phenylpropionic Acid,  $C_6H_5.CH_2.CH_2.CO_2H$  (Hydrocinnamic, Homotoluic, or Benzylacetic Acid).**—Both these acids have been made the subjects of comparative bacteriological research by Klein and Lingard, and both are capable, in half per cent. solutions, of destroying completely the infectious capacity of tubercular virus. Williams (Brompton Hospital) found that both phenylacetic and  $\beta$ -phenylpropionic acid given internally benefit the appetite and digestion of phthisical patients, and were well borne even in large doses.

Allivia (*Gazetta degli Ospitali*, 1888, No. 86) administered it in large doses in cases of typhus with favourable results.

**Indian Geranium Oil.** F. W. Semmler. Ber. 23, 1098—1103.

THE author has investigated several ethereal oils, more particularly geraniol, possessing the empirical formula  $C_{10}H_{18}O$ , with the object of throwing some light on the constitution of the terpenes obtainable from them, and on the manner in which certain terpenes are formed in plants. Two Indian geranium oils were examined. They were olive green in colour and possessed an agreeable pear-like odour. Their specific gravities at  $16^{\circ}$  C. were 0.8868 and 0.8871 respectively. Chemically the oils were identical, and both exerted a slight levo-rotatory action on polarised light. Distilled under a pressure of 17 mm., 92 per cent. of the oil passed over between the temperatures of  $120.5^{\circ}$ — $122.5^{\circ}$  C. The whole of this fraction was found to consist of geraniol. Geraniol is a colourless liquid of the specific gravity 0.89 at  $15^{\circ}$  C. and possesses a pleasant odour. Hitherto geraniol has been looked upon as a benzene derivative isomeric with borneol. But its high boiling point and low specific gravity as compared with borneol and similar bodies, point to the possibility of geraniol really being an open chain compound. To settle the point, the refractive index was determined and found to be at  $20^{\circ}$  C. :—

	$n_D$ .	$n_D$ .
Li .....	1.4741	
Na .....	1.4766	

Therefore  $n_D = 1.4745$ .

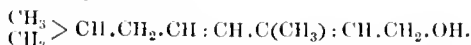
The molecular refraction obtained by means of the formula  $\frac{(n_D^2 - 1)P}{(n_D^2 + 1)d}$  will therefore be 48.71. Using Brühl's constants, it appears that geraniol contains at least two ethylene groups, the calculated molecular refraction being 48.66. This is confirmed by the fact that geraniol readily takes up 4 atoms of bromine or iodine. A body of the formula  $C_{10}H_{18}O$

containing two ethylene residues, cannot possibly be a benzene derivative; and as geraniol is undoubtedly an alcohol, it must belong to the series of unsaturated alcohols,  $C_{17}H_{28}O$ . On oxidation with potassium permanganate, a molecule of geraniol yields almost quantitatively a molecule of iso-valerianic acid. By the action of phosphorus pentoxide on geraniol, a terpene and a poly-terpene, boiling respectively at 60–65° C. and 205°–215° C. under a pressure of 17 mm., are obtained, the latter being splendidly dihydroic. These bodies have not yet been thoroughly examined, but they are undoubtedly benzene derivatives. Under these circumstances, two formulæ are possible for geraniol.

1. Optically active—



2. Optically inactive—

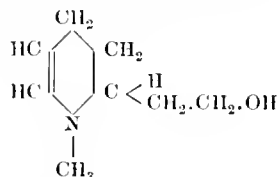


The ease with which the ether,  $(C_{10}H_{17})_2O$ , and the sulphide,  $(C_{10}H_{17})_2S$ , are formed, inclines to the latter formula. The author is continuing his researches.—H. T. P.

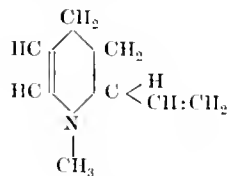
#### The Relation of Cocaine to Atropine. A. Einhorn.

Ber. 23, 1338–1344.

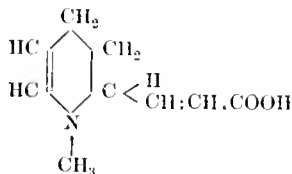
БЕЧКА has ascribed to tropine, without sufficient evidence the following constitutional formula, in which, however, he expressly states, it is uncertain whether the hydrogen atoms are attached to the  $\alpha$ ,  $\beta$ , or  $\gamma$  carbon-atoms—



If this formula be correct, tropine must bear a close relationship to ecgonine—the basic decomposition product of cocaine. This relationship is more clearly exhibited in the formulae of their respective derivatives—tropidine and anhydro-ecgonine—



Tropidine.



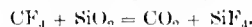
Anhydro-ecgonine.

the latter being the carboxylic acid of the former. The correctness of this view has been proved by experiment. The author has previously shown (this Journal, 1889, 634) that when anhydro-ecgonine is heated for eight hours at 280° C. with strong hydrochloric acid, a number of hydrogenised pyridine bases are produced. To one of these, yielding a gold salt melting at 212° C., the formula  $C_7H_{13}N$  was attributed. It now appears that the analytical results then obtained for the gold salt were incorrect, owing to the presence of a slight impurity. The base is, however, easily obtained in a state of purity by conversion into the difficultly soluble picrate. Its correct formula is  $C_8H_{13}N$ , which is the

same as that of tropidine. The complete identity of the two bodies was proved by comparing their picrates, and gold and platinum salts. A complete description of the crystalline form of these compounds follows.—H. T. P.

#### On the Preparation and Properties of Carbon Tetrafluoride. H. Moissan. Compt. Rend. 120, 951–954.

VARIOUS methods of preparing this gas are described. It is best obtained by passing the vapour of carbon tetrachloride over silver fluoride heated in a metallic tube to a temperature between 195° and 220°. The gas obtained by operating in glass has approximately the same density, but is a mixture of silicon tetrafluoride, carbonic acid, carbon tetrafluoride, and a carbon fluoride of higher density. Silicon tetrafluoride has a density of 3.09 compared to air, the theoretical number being 3.03. It is liquefiable under a pressure of four atmospheres in Cailliet's apparatus at +20° C., and under the ordinary pressure at –15° C. It is but slightly soluble in water, very soluble in ordinary ether, and especially in anhydrous alcohol. It is insoluble in monohydrated sulphuric acid, aqueous potash, and baryta solutions. In contact with a heated glass surface, the following reaction takes place gradually:—



When carbon tetrafluoride is heated with sodium, sodium fluoride is formed, and the carbon is deposited. It is absorbed by alcoholic potash and gradually converted into potassium fluoride and potassium carbonate.—P. J. H.

#### On Amethylcamphophenolsulphone and its Tetra-nitro Derivative: a Yellow Colouring Matter. P. Cazeneuve. Compt. Rend. 110, 961–964.

See under IV., page 853.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

#### Manganous Sulphate in Photo-Lithography. W. Weissenberger. Phot. Corr. 27, 159–161.

PAPER coated with gelatin is sensitised on the following solution:—Water, 1,000 parts; potassium dichromate, 40 parts; manganous sulphate, 5 parts. Paper thus prepared is more sensitive than paper prepared with dichromate alone, and the quantity of dichromate reduced in a given time by light of a given intensity is considerably greater. At the same time the degree of alteration is proportional to the intensity of the light. The employment of manganous sulphate renders it unnecessary to use the concentrated solutions of dichromate which have lately been recommended.—C. H. B.

#### Recovery of Silver from Residues by means of Hydroxylamine. A. Lamer. Phot. Corr. 27, 209–212.

OLD fixing baths and other waste liquids, concentrated if possible by exposure to air and sunlight in shallow dishes, are made strongly alkaline with crude caustic alkali, heated and mixed with successive small quantities of a solution of commercial hydroxylamine chloride ("reducing salt" of the Badische Anilin und Soda-fabrik), previously made alkaline. Precipitation of the silver takes place rapidly, and after a time a small quantity of the liquid is withdrawn and filtered. One part of the filtrate is mixed with caustic alkali and heated in order to see whether excess of alkali is present, and the other part is mixed with more hydroxylamine and heated, and if further reduction takes place, more hydroxyl-

amine is added to the bulk of the liquid, and the process is continued. An excess of caustic alkali is essential to complete reduction. The precipitated silver is washed with hot water.—C. H. B.

*Ammonium Iodide.* W. Weissenberger. *Phot. Corr.* **27**, 273—274.

AMMONIUM iodide, which is largely used in the wet collodion process, is a very unstable compound, and the author has devised a method of preparing it as required from substances which themselves are quite stable. 10 grms. of potassium iodide are dissolved in 20 cc. of water, and mixed with 4 grms. of ammonium sulphate dissolved in 30 cc. of water. Absolute alcohol is now added until the volume of the liquid is 218 cc.; potassium sulphate is precipitated and the liquid contains 4 per cent. of ammonium iodide.—C. H. B.

*Iodine in the Ferrous Oxalate and Pyrogallol Developers.* A. Lainer. *Phot. Corr.* **27**, 306—307.

SMALL quantities of an alcoholic solution of iodine added to the ferrous oxalate developer reduce the contrasts in the resulting negative without affecting the amount of detail. Iodine solution added to a mixed pyrogallol developer has little effect unless added in much larger quantity than in the case of the ferrous oxalate; it then produces a very similar result. A very dilute solution of potassium iodide has a similar effect.

If iodine solution is added to the soda solution before the latter is mixed with the pyrogallol solution, very strong restraining action is observed, and if the quantity of iodine is considerable, the development of an image may be prevented altogether. This result is doubtless due to the formation of sodium iodide. The action of iodides is quite different from that of bromides, since they change the silver bromide in the film more or less completely into iodide. —C. H. B.

*Orthochromatic Photography.* L. Vida. *Phot. Corr.* **27**, 351—352.

A MIXTURE of indophenol blue and malachite green sensitises gelatin plates for yellow, orange, and red, but somewhat reduces the sensitiveness to blue, and hence may be used in place of the costly and unstable azaline (a mixture of cyanine and quinoline red). 0.1 gm. indophenol blue (naphthalene blue) is dissolved in 500 cc. of alcohol. 1 gm. of malachite green is dissolved in 200 cc. of water, heated to about 70°, and mixed with a hot solution of 10 grms. of potassium dichromate in 100 cc. of water, heated for half an hour and filtered. The precipitate is washed, dissolved in a small quantity of hot alcohol, and diluted to 250 cc. with alcohol containing 6—8 grms. of quinine sulphate.

Gelatin plates are immersed for 2 minutes in a mixture of 4 cc. of the indophenol blue solution, 4 cc. of the prepared malachite green solution, and 600 cc. of water. They are then allowed to drain, and are dried in the dark.—C. H. B.

*A Standard Method of Development.* C. H. Bothamley. *J. Photo. Soc.* **14**, 134—142.

In ordinary photographic practice the method of development must be adapted to the nature of the subject which is being dealt with, and the character of the result which is desired, but in astronomical photography, the testing of plates, and for other scientific purposes, a standard method of development is desirable. A standard method must fulfil the following conditions—(1), it must develop the maximum amount of detail which the plate is capable of giving, without producing fog, *i.e.*, without reducing metallic silver in those parts of the film which have not been acted on by light; (2), it must be simple in execution; (3), it must resemble as closely as possible the methods used in ordinary practice; (4), it must involve the use of only such chemicals as can be purchased in a state of purity, and are not hygroscopic or liable to undergo other alteration when

stored with ordinary care; (5), the solutions must be easily prepared and not liable to alteration.

The only developing agent in general use which satisfies these conditions is pyrogallol used with ammonia as an accelerator and ammonium bromide as a restrainer, or with sodium carbonate as accelerator and potassium bromide as restrainer. The use of sulphites is not admissible because their composition is very variable and their precise influence is not yet understood.

A stock solution of ammonia is made by diluting strong ammonia solution with 15 times its bulk of water, is carefully titrated with a solution of oxalic acid containing 37.06 grms. per litre (1 cc. = 0.01 gm.  $\text{NH}_3$ ) using aurin as an indicator, and is used for making up the final ammonia solution. Pure sodium carbonate is made by carefully heating the pure bicarbonate at 150°.

The author made a large number of experiments with plates of different kinds with a view to determine the influence of variations in the composition of the developer, time of development, continual or intermittent agitation of the liquid, and exposure to or exclusion of air. He found that the maximum amount of detail which can be developed on a plate which has received a given exposure is independent of variations in the composition of the developer provided that the time of action is sufficiently prolonged. The time required varies considerably in different cases and the densities of the deposits corresponding to different intensities of light are not constant. Similar results have previously been obtained by Mr. Lionel Clark. Two standard methods are recommended.

*Standard Pyro-Ammonia.*—Five parts of pyrogallol, 5 parts of ammonium bromide, 2.5 parts of real ammonia ( $\text{NH}_3$ ) in 1,000 parts by measure of the solution. Develop for 10 minutes in an open or closed vessel at 15° C. or 60° F. with occasional shaking. The developer is made up in two solutions. A. Ammonia ( $\text{NH}_3$ ) 5 parts, ammonium bromide 10 parts, water up to 1,000 parts. B. Pyrogallol 10 parts, water up to 1,000 parts. These are mixed in equal volumes immediately before use; solution A. will keep indefinitely in properly closed vessels, solution B. must be made as required.

*Standard Pyro-Soda.*—Five parts of pyrogallol, 10 parts of anhydrous sodium carbonate, 2.5 parts of potassium bromide in 1,000 parts by measure of the solution. Develop for half an hour in a closed vessel at 15° C. or 60° F. In this case also two solutions are prepared and are mixed in equal volumes immediately before use. A. Sodium carbonate 20 parts, potassium bromide 5 parts, water up to 1,000 parts. B. Pyrogallol 10 parts, water up to 1,000 parts.

The proposed standards agree very closely in composition with developers used in ordinary practice. They will develop the maximum possible amount of detail on all the different makes of plates of best repute without giving chemical fog, and any plates which will give chemical fog with these developers during the time specified may be regarded as below the highest standard of quality.—C. H. B.

*Backing for Plates.* W. E. Debenham. *J. Photo. Soc.* **14**, 203—206.

CAREFUL measurement of the relative efficiency of various substances used for backing plates in order to prevent halation, shows that bitumen, which has frequently been recommended, is very far from satisfactory. This is true also of mixtures of bitumen and lamp-black, and the result seems to be due to imperfect optical contact with the glass after the backing has dried. Carbon tissue moistened with glycerol and squeezed in contact with the glass is also unsatisfactory.

Gum and burnt sienna; gum or gelatin and burnt sugar, with or without the addition of lamp-black or Chinese ink and also gum and lamp-black make very efficient backing. The mixtures containing burnt sienna or burnt sugar show very little halation even with prolonged exposures, and the sugar confers considerable elasticity on the backing. The addition of lamp-black or Chinese ink is not necessary if a sufficiently thick coating of the mixture is laid on the plate.—C. H. B.

*Gelatinobromide Emulsions.*

BURTON (Amer. Am. Phot. 1890, 107) describes the following method of preparing an emulsion with a fine grain: 10 grms. of finely powdered dry silver nitrate are added to a solution of 7 grms. ammonium bromide, 0.5 gm. of potassium iodide and 2 grms. of soft gelatin in 124 cc. of water, previously heated to 50° C., and the mixture is violently shaken for some time. 8 grms. of hard gelatin previously swelled in water are then added, thoroughly melted, and the emulsion allowed to solidify, after which it is washed in the usual manner.

Bell (Phot. News, 34, 259) describes a novel method of preparing a highly sensitive emulsion: 60 parts of silver nitrate dissolved in 220 parts of water is mixed with a solution of 5 parts of ammonium iodide in 440 parts of water until the precipitated silver iodide ceases to dissolve. 20 parts of ammonium chloride dissolved in 220 parts of water slightly acidified with nitric acid are then added, and the silver chloride is washed three times by decantation, mixed with broken glass, and 16 parts of Nelson's No. 2 gelatin are added and melted at 32–38° F. The mixture is then thoroughly shaken and a solution of 43 parts of potassium bromide and 27 parts of strong ammonia solution in 220 parts of water is then added all at once and the vessel is corked and heated at 32° for 15 or 20 minutes. From time to time a drop of the emulsion is examined by transmitted light, and as soon as it is greyish-blue or green in colour, 120 parts of Heinrich's gelatin, previously softened in water and melted at a temperature higher than 32°, is added and the mixture is well shaken and allowed to cool slowly. After standing 6 hours it is remelted at 100° and again allowed to cool. After a second 6 hours it is remelted and is then mixed with some boiled acid emulsion, or if it is to be used alone, is allowed to set in a dish and is treated in the usual way.—C. H. B.

*Acid Fixing Bath.* F. Beach. Amer. Am. Phot. 1890, 185. 120 parts of crystallised sodium sulphite is dissolved in 1,000 parts of water, 90 parts of powdered chrome alum and 15 parts (by vol.) of sulphuric acid are added, and the liquid is mixed with a solution of 1,000 parts of sodium thiosulphate in 3,000 parts of water. This bath has the advantage that it exerts a very powerful astringent action on the film and thus prevents frilling.—C. H. B.

*Combined Toning and Fixing Bath.* Liesegang. Phot. Arch. 1890, 118.

1,000 parts of sodium thiosulphate, 400 parts of alum, and 10 parts of lead nitrate are dissolved in 2,000 parts of water. After 2 days, 2,000 parts of water are added, the liquid filtered, mixed with a solution of 800 parts of ammonium thiocyanate in 6,000 parts of water, and 50 to 100 parts of a 1 per cent. gold chloride solution are added. Acidification with tartaric acid accelerates the toning but impairs the whites of the prints. Sodium acetate, phosphate, and benzoate, reduce the action of the toning bath, but give somewhat warmer tones.

If potassium chloroplatinite is used instead of gold chloride a platinum toning and fixing bath is obtained.

—C. H. B.

*Ferric Chloride for Copper Etching.* W. Weissenberger, Phot. Wochenblatt, 16, 210.

Ferric chloride containing only 2 per cent. of free hydrochloric acid attacks even a coarse grain somewhat energetically. For fine work neutral ferric chloride is therefore indispensable and it can be prepared in the following manner. 1,000 parts of ferric chloride are dissolved in 300 parts of water. 5 parts of this solution are diluted with 500 parts of water and precipitated with potassium hydroxide. The well-washed precipitate is added to the original solution, and after one or two days the cleared liquid is decanted from the undissolved precipitate.—C. H. B.

## PATENTS.

*Improvements in or relating to Films or Supports for Photographic Negatives or Prints.* J. S. Fairfax, London. From F. Crane, New Jersey, U.S.A. Eng. Pat. 10,393, June 26, 1889. 6d.

A METHOD of preparing films or supports of pyroxylin which can afterwards be coated with gelatinobromide emulsion in any suitable manner. The film may be made from a comparatively limpid solution which is flowed over a smooth surface or carrier, the desired thickness being given to the film by successive coats of the solution, which can readily be done provided that the solutions are practically saturated. It may also be made from a heavy viscous solution which is spread over the carrier by means of a knife or straight-edge, the thickness of the film being regulated by the spreader. The latter method is the quicker and cheaper of the two.

A good formula for the thin solution is: 40 galls. of methyl alcohol (wood spirit); 20 galls. of amyl, propyl, or butyl acetate, or mixtures thereof; 40 galls. of amyl alcohol; 50 lb. of soluble pyroxylin. For a viscous solution the following may be used: 55 galls. of methyl alcohol (wood spirit); 20 galls. of amyl alcohol (fusel oil); 25 galls. of amyl acetate; 50 lb. of gum camphor; and 100 lb. of soluble pyroxylin. The proportions of the constituents may be varied, butyl alcohol or its isomers may be used in place of amyl alcohol, and butyl acetate or other ethereal salts of butyl or amyl which will dissolve pyroxylin may be used in place of amyl acetate.

When the film is dry it is stripped from the carrier or temporary support and cut into pieces or into long strips for use on rollers.—C. H. B.

*Improvements in Apparatus for Coating Photographic Dry Plates and other Plane Surfaces with an Emulsion or Viscous Liquid.* J. H. Smith, Zurich, Switzerland. Eng. Pat. 11,553, July 19, 1889. 11d.

THE claims made are for a trough divided by means of a removable partition into two compartments communicating at the bottom, one serving to receive a regular stream of emulsion, whilst the other is provided with an overflow lip consisting of a flat horizontal surface or of two flat surfaces meeting at an obtuse angle; a moveable curved piece for adjusting the surface down which the liquid flows, in order to correct for the different thicknesses of the surface to be coated; rollers supported in a trough containing water, and covered with some good absorbing material so that they apply cold water to the backs of the coated plates or surfaces; rollers driven by a double worm so that increased speed can be given to the plates or other surfaces after they have been coated.—C. H. B.

*Improvements in the Treatment of Paper or other Fabrics to render them Suitable for Photographic Purposes.* J. Williams, Willesden. Eng. Pat. 12,309, August 2, 1889. 4d.

A STRONG solution of pure cuprammonium hydroxide is prepared, 1.5 to 2.5 per cent. by weight of metallic copper being used according to the brilliancy of surface required. Paper or any other fabric which is to be treated is floated on this solution, care being taken that only one surface comes in contact with the liquid. The surface is thus converted into a structureless film of cellulose, which at this stage exists in combination with the cupric oxide, ammonia, and water. The paper or other fabric, with the minimum exposure to air required to make the film set, is dried over one or more hot rollers. In order to remove the copper from the film, it is passed through successive baths of weak acid, which dissolves the copper without affecting the glazed surface.

In this way a permanent glaze resembling that of albuminised paper is obtained on paper or other fabric, and it withstands the action of water, steam, weak acids, alkalis, and ordinary solvents.

Other suitable solvents may be used in place of cuprammonium hydroxide.—C. H. B.

## XXII.—EXPLOSIVES, MATCHES, Etc.

## PATENTS.

*Improvements in the Construction of Electric Fuses and Detonators, and in the Method of and Appliances employed for Manufacturing the same.* P. Ward and E. M. Gregory, London, Eng. Pat. 11,501, July 18, 1889. 8d.

THIS patent relates to a tool by which a small plug of insulating material may be placed round the ends of the limbs of the "armature" of Eng. Pat. 3981 of 1884, so as to hold them apart at a fixed and uniform distance. The ends of the limbs project through this plug a fixed distance, and the end of the plug itself is furnished with a small cavity into which the priming material may be placed so as to surround the exposed ends of the limbs. The insulating plug with its priming is then protected by dipping into paraffin or other wax, and then into Roman or other hard cement, and may finally, if desired, be covered with a coating of fusible alloy or lead.

For detonators, after the protective coating of wax, the detonating composition is placed on the end of the fuse, and then covered with final protective coatings.—E. T.

*Process and Apparatus for the Manufacture of Explosives in the Form of Wires or Rods and for forming the same into Cartridges.* F. A. Abel, London; J. Dewar, Cambridge; and W. Anderson, London. Eng. Pat. 11,667, July 22, 1889. 11d.

THIS "invention relates to a process and apparatus for the manufacture of explosives in the form of wires or rods which are produced by mixing together certain ingredients so as to form a plastic explosive compound such as blasting gelatin or similar compounds, and then converting the compound into a wire or rod-like condition. The ingredients of the explosive are in the first place thoroughly mixed, for which purpose an ordinary kneading machine such as that used for dough may be employed; and the mixture is by means of pugging apparatus forced out by helical blades through a die into the form of a column which can be cut into suitable lengths as in a brick-making machine, these lengths being afterwards operated on by a machine, converting the material into flexible wires or rods which are wound on suitable reels."

"From the reels a number of the wires or rods are unwound and dried by passing them through a steam-jacketed tube or otherwise, and are then by means of a suitable machine formed into bundles and introduced into cartridge cases, to constitute their explosive charges."—W. M.

*Improvements Relating to the Waterproofing of Blocks, Charges, or Cartridges of Explosive Material and to Apparatus therefor.* C. Lamm, Stockholm, Sweden. Eng. Pat. 12,442, August 6, 1889. 8d.

THIS specification describes a machine for dipping blocks charges or cartridges of explosive material into a bath filled with a suitable waterproofing material kept in a liquid state. The cartridges, &c. pass down a channel on to an endless belt provided with hooks which take the cartridges and convey them to corresponding spring clamps fixed to the surface of a wheel which catch the cartridges. The lower part of the wheel is immersed in the waterproofing bath, and as the wheel rotates the cartridges are dipped into the bath and then delivered on to another endless belt which conveys them away. There are special contrivances for ensuring a uniform coating being given to the cartridges. Detailed descriptions and drawings are given of the machine.

—W. M.

*Improvements in the Manufacture of certain Fireworks for Display Purposes.* H. Stephenson, London, Eng. Pat. 14,804, September 19, 1889. 8d.

THESE improvements relate to fireworks which display a bright silver or gold star when burnt. Hitherto fireworks of this character could only be kept for two or three weeks as the iron borings employed in their manufacture were affected by the atmospheric influences and thus chemical action was set up between them and the other chemicals. The main feature of this invention is the preparatory treatment of the iron borings whereby they receive a coating of zinc and thus, after mixing with the other chemicals, the composition can be safely stored for lengthened periods without undergoing any alteration or deterioration.—W. M.

*Improvements in Making and Applying Explosives.* C. O. Lundholm and J. Sayers, Stevenston. Eng. Pat. 13,486, August 27, 1889. 6d.

THE inventors make a "sub-sensitive" explosive by gelatinising one or more nitro-derivatives of cellulose, oxy-cellulose, or hydro-cellulose, with nitro derivatives of the aromatic group of substances. The cellulose derivative is suspended in water and the aromatic nitro-compound (also diffused through water sufficiently warm to keep it liquid) is poured in with suitable agitation, or a solid nitro derivative such as dinitro-benzene may be liquefied by heat and simply poured into the liquid containing the suspended nitro-cellulose.

Explosive compounds made in this way may be used alone for filling shells or in conjunction with more sensitive explosives, such as blasting gelatin; in the latter case the less sensitive explosive is arranged outermost in the shell and the blasting gelatin in the centre.—W. M.

*An Improved Priming and Detonating Composition.* P. Ward and E. M. Gregory, London, Eng. Pat. 8481, May 31, 1890. 4d.

THIS composition consists of 2 lb. of carbon in the form of coke, 1 lb. of amorphous phosphorus, and "75 lb. of potassium chlorate." In the claim the amount is stated to be 0.75 lb. The phosphorus and chlorate are ground separately under benzene, carbon chloride or amyl acetate, and then, together with coke, still under the liquid.

Paraffin oil is added, and after the grinding fluid has evaporated this causes the composition to cake and also prevents oxidation and reduces its sensitiveness to friction or percussion without detracting from its explosive violence.

—W. M.

## XXIII.—ANALYTICAL CHEMISTRY.

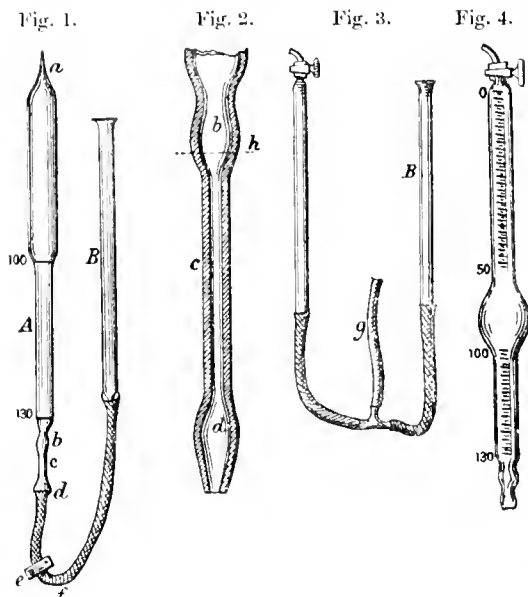
## APPARATUS, &amp;c.

*On the Preparation of Gas Reduction Tubes ready for Use.* G. Lunge. Zeits. f. angew. Chem. 1890, 227—229.

THE author shows how the reduction tube mentioned in his article on the "gas volumeter" (this Journal, 1890, 547—549)—which may also be used for ordinary reduction apparatus—can be manufactured and kept ready-made for sale, so as to save the buyer the trouble of adjusting it himself by making barometer and thermometer observations at the beginning of his work. This will be desirable when using the "gas volumeter" for many technical purposes, e.g., the determination of carbon dioxide in cement manufacture, of nitrogen in ammonium sulphate, of chlorine by bleachers and dyers, in urine tests made by physicians, and for various other practical purposes. It would not be sufficiently safe, if the apparatus had to be sent to a distance, to seal up the tube at one end and to close it at the other with a stop-cock after the proper adjustment

(compare this Journal, 1890, 547—548) has been made. Absolute safety is only possible if the tube be sealed up on both ends.

For this purpose glass tubes must be used as shown in Fig. 1. *a* is a comparatively thick tube of capillary



THE PREPARATION OF REDUCTION TUBES.

bore which can, however, be sealed up with perfect ease. The part at *c* is a similar capillary tube with thick walls about 5 cm. long and terminating in the bulbs *b* and *d*, as shown in the figure. Fig. 2 shows the parts *b*, *c*, and *d* in natural size. The tube *A* is connected (as shown in Fig. 1) with the levelling tube *B*, and mercury is introduced into the latter. If the gases are to be measured moist, a small drop of water is brought into tube *A*, but if they are to be measured dry, a small drop of concentrated sulphuric acid is introduced instead of water.

By barometric and thermometric observations (the thermometer used has to be suspended closely to tube *A*) and by the use of the formula  $\frac{273(B-f)}{(273+t)760}$  for moist gases,

and  $\frac{273B}{(273+t)760}$  for dry gases, or by the use of tables

calculated therefrom, we find the volume which 100 cc. of perfectly dry air, measured at 0° C. and 760 mm., would occupy under the existing conditions. This may be, e.g., 112.3 cc. Now *B* is moved so as to have the level of the mercury in *A* at 112.3 cc., then the part *a* is covered with a piece of asbestos cloth, to prevent *A* from getting hot, and finally the capillary end of *a* is sealed up. The work can be proceeded with, if after some minutes the level of the mercury has undergone no change, but if a change has taken place, *a* has to be opened again, and the operation repeated more carefully. After that the india-rubber tube is closed tightly with a small vice (an ordinary burette clip not being sufficient), the mercury below *e* is poured off, the tube cut through at *f*, and lastly the capillary tube *c* is closed by sealing it up. This can easily be effected by holding *A* almost horizontally. The apparatus is now ready, and the author recommends that the parts *a* and *c* be covered with small india-rubber tubes for protection.

Arrived at its destination and ready for use, the "gas volumeter" is first put together, but without the reduction tube just described, as is shown in Fig. 3. The india-rubber tube *g* which is to be connected with the reduction tube is fixed vertically, and then mercury is poured into the levelling tube *B* (Fig. 3), until *g* is entirely filled. After that the reduction tube is notched with a file at its lower part at *h* (Fig. 2), this end put into the mercury of *g*, *g* being very

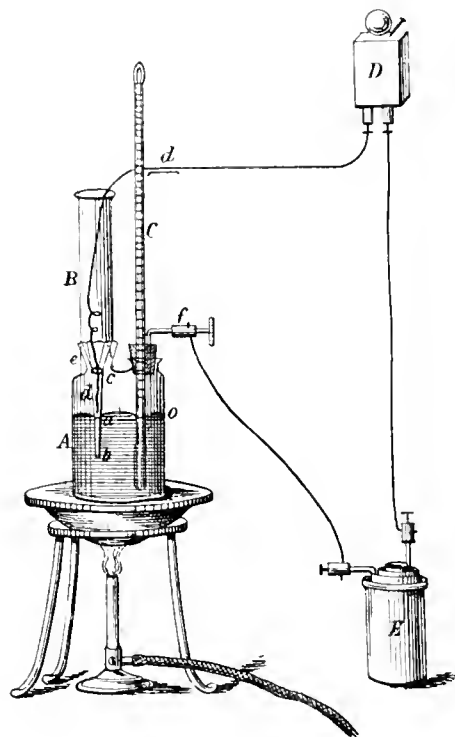
carefully placed over the bulb *b*, to avoid air bubbles, and lastly *c* is broken at *h* whilst inside the tube *g*. The point *h* has to be taken at a comparatively wide part of *c*, in order to enable a quick motion of the mercury when the apparatus is in use.

Another shape of the tube *A* is shown in Fig. 4, which is very convenient for large as well as for small volumes of gases. Of course the ungraduated part (between 50 and 100 cc.) has to be avoided, but this can easily be done in most cases.—H. S.

#### Apparatus for the Determination of Melting Points.

A. C. Christomanos, Ber. 23, 1093—1096.

*A* is a cylindrical glass vessel 12 × 6 cm., which is heated on a sand-bath or in an air-bath. A cork is inserted in one of the necks and carries a thermometer *C* and a stout platinum wire *f*, while the other neck is conical and ribbed to allow the tube *B* to rest firmly within it. The vessel *A* is



THE DETERMINATION OF MELTING POINTS.

filled with mercury to such a height that the lower end of the tube *B* dips into it about 2 cm. The substance the melting point of which is to be determined is fused in a test tube and a portion sucked up into *B* and allowed to solidify in its contracted part, forming a layer 0.5—1.5 cm. in thickness. The tube *B* being now restored to position, a platinum wire *d* is inserted so as just to touch the upper surface of the layer, and mercury is poured on to a depth of 1.5—2 cm. The two platinum wires are connected with the cell and bell shown in the figure, and the whole is ready for use. The temperature of the vessel *A* is slowly raised until the substance melts, allowing the mercury in the tube *B* to flow down and complete the circuit, ringing the bell and calling the operator's attention.

At temperatures above 250° C. a draught cupboard should be used; the applicability of the apparatus is limited to those substances that do not attack mercury appreciably. It can be used for a considerable range of temperature, the author having determined the melting points of ice and of saltpetre (338° C.) by its means.—B. B.

*Alteration of the Coefficient of Compressibility of Glass at different Temperatures.* E. H. Amagat. *Compt. Rend.* **110**, 1246—1249.

See under VIII., page 862.

# INORGANIC CHEMISTRY.—QUALITATIVE.

*On the Concentration of Solar Rays for Chemical Reactions.* J. W. Brühl. *Ber.* **23**, 1462.

MANY chemical processes are produced or accelerated by sunlight, and the author has therefore tried to increase the effect of sunlight by the concentration of rays by means of a concave mirror. As was expected, the experiments showed very favourable results, *e.g.*, in preparing zinc ethyl. The process is sometimes very tardy in its beginning, therefore the retort containing zinc and ethyl iodide was brought into the focus of a concave mirror of about 30 cm. diameter. The reaction then very readily began, and soon became so active that it was necessary to cool the retort. In about 15 minutes the process was finished and a large quantity of zinc ethyl was obtained by subsequent distillation in the oil-bath. The author thinks that this concentration of solar rays by means of a concave mirror will be of value also for other experiments, especially with halogen compounds. A glass lens may be used instead of a metallic concave mirror, but the action will be less effective, because of the considerable athermancy of glass.—H. S.

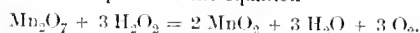
*The Action of Hydrogen Dioxide on the Oxides of Manganese.* A. Gorgeu. *Compt. Rend.* **110**, 857—859.

ANHYDROUS crystallised manganese dioxide decomposes hydrogen dioxide with comparative slowness. In presence of a slight excess of baryta the manganese dioxide suffers no change in composition; if, however, the hydrogen dioxide be acid, the dioxide of manganese is reduced more or less according to the amount of acid present. In two experiments, manganese dioxide, containing 98.5 per cent. of  $MnO_2$ , after treatment with three equivalents of hydrogen dioxide, acidified in one case with 0.05 per cent., and in the other with 0.20 per cent. of sulphuric acid, contained only 97 and 94.4 per cent. of  $MnO_2$  respectively. Hydrated dioxide of manganese acts far more energetically on hydrogen dioxide, the dioxide of manganese itself being in all cases considerably reduced. A sample containing 97.5 per cent. of  $MnO_2$  (reckoned on the anhydrous oxide), after decomposing three equivalents of hydrogen dioxide, contained only 70 per cent. of  $MnO_2$ . It appears, however, that reduction stops when the oxide arrives at the composition expressed by the formula  $Mn_2O_4$ ; in fact, this oxide is wholly unalterable by the action of hydrogen dioxide. It may be prepared by agitating moist manganous oxide in contact with air. Under these circumstances it might be thought that it were impossible, by means of hydrogen dioxide, to oxidise hydrated manganous oxide further than is expressed by the same formula  $Mn_2O_4$ , but this is not the case. In several instances hydrated manganous oxide treated with an excess of hydrogen dioxide was converted into an oxide containing appreciably more oxygen than the compound  $Mn_2O_3$ . The decomposing influence of manganous oxide on hydrogen dioxide is no doubt due to its strong basicity. The rate of decomposition is further accelerated by the higher oxide of manganese soon formed from the manganous oxide, and the high degree of oxidation attained may perhaps be ascribed to the large quantity of nascent oxygen thus produced. Anhydrous manganous oxide and precipitated manganous carbonate are not affected by hydrogen dioxide. The author believes to have proved that  $Mn_2O_4$  is slowly converted in contact with air or aerated water into  $Mn_2O_3$ .—H. T. P.

*The Action of Hydrogen Dioxide on Oxygen Compounds of Manganese. Part II.—Action on Permanganic Acid and Permanganates.* A. Gorgeu. *Compt. Rend.* **110**, 958—961. (See preceding abstract.)

In a solution containing either sulphuric acid or some other body, such as ammonium chloride, capable of dissolving manganese protoxide, hydrogen dioxide reduces all peroxides and acids of manganese to the state of manganese protoxide.

Hydrogen dioxide acting on a solution of pure permanganic acid decolourises it, oxygen being evolved and a peroxide of variable composition formed. Under certain circumstances the reaction corresponds to the equation—



If hydrogen dioxide be added to neutral or alkaline solutions of potassium and sodium permanganates the manganates and manganites are formed, the manganite being only slowly further reduced, while it exercises a rapid reducing action on the hydrogen dioxide itself. The barium salt, under similar conditions, gives the insoluble manganate and manganite. Silver permanganate yields a brown crystalline precipitate of silver manganite. In all the above reactions with neutral solutions, the hydrogen dioxide was poured into the permanganate solution, and the quantity of hydrogen dioxide decomposed by 1 equivalent of permanganic acid varied from 3 to 11 equivalents. If the permanganate be poured into the hydrogen dioxide, one equivalent seems to decompose an unlimited quantity of the dioxide. In 10 minutes one equivalent of permanganate decomposed from 150 to 300 equivalents of  $H_2O_2$ .—P. J. H.

*A New Characteristic Reaction of Hydrogen Peroxide.* G. Denigès. *Compt. Rend.* **110**, 1007.

A SOLUTION of ammonium molybdate in water (1:10) added to its own volume of concentrated sulphuric acid (1 cc. of each for example) gives, with traces of hydrogen peroxide, a yellow colouration, the intensity of which may be equal to that of a solution of an alkaline chromate or bichromate. Addition of sulphuric acid decreases the intensity of the colour in proportion to the quantity added, whilst addition of water causes a greater proportional decrease. One-tenth of a mgrm. of hydrogen peroxide may be thus detected. The ammonium molybdate may be replaced by the sodium salt, but without any advantage.

—J. W. L.

*Lead Phosphite and Lead Pyrophosphite; Qualitative Distinction between Pyrophosphites and Phosphites.* L. Amat. *Compt. Rend.* **110**, 901—904.

NEUTRAL lead phosphite was found to be extremely insoluble. If lead nitrate be added to lead phosphite in acid solution, an insoluble crystalline precipitate of *lead nitrophosphite*,  $PbHPO_3 + Pb(NO_3)_2$ , is thrown down.

By dissolving neutral lead phosphite in a hot concentrated solution of phosphorous acid, *acid lead phosphite*,  $PbH_2(PO_3)_2$ , is formed in well defined crystals, which decompose, in dilute solution, into phosphorous acid and the neutral phosphite of lead. If the acid phosphite be heated to 140° in a dry vacuum, one molecule of water is driven off, and *lead pyrophosphite*,  $PbH_2P_2O_5$ , is produced. In the presence of water this pyrophosphite becomes hydrated, but the acid phosphite formed gives rise immediately to the insoluble neutral phosphite and phosphorous acid.

When a solution of lead nitrate is added to a solution of sodium pyrophosphite, a crystalline precipitate of lead nitrophosphite is formed, and the solution becomes acid. This reaction takes place immediately in a *concentrated* solution even in the cold, but only after hours, or sometimes days if the solution be *cold* and *dilute*. On boiling, however, precipitation takes place immediately even in a dilute solution.

The reactions above described afford a ready method of distinguishing qualitatively pyrophosphites from phosphites.

Lead nitrate is added to the cold dilute solution; when phosphites are present, an immediate precipitate is formed, which is filtered off. The filtrate is then boiled; if pyrophosphites be present a precipitate will then be formed.

—P. J. H.

*New Mode of obtaining Crystalline Metallic Oxychlorides; the Oxychlorides of Copper.* G. Rousseau. *Compt. Rend.* **110**, 1261—1264.

See under VII., page 859.



*On the Action of Potassium Sulphate on Strontium Carbonate.* F. Kuklin. J. Russ. Chem. Soc. **22**, 322—326.

THE usual analytical method of separation of barium from strontium and calcium by boiling the solution of these metals with the mixture of potassium sulphate and potassium carbonate often fails and makes necessary a second boiling. The author, who undertook to find out the best condition under which the reaction is practically reliable, finds that strontium carbonate ceases to change into strontium sulphate when the proportion of sulphate to carbonate in the mixture is less than 55 : 1.

On the other hand the precipitation of strontium sulphate by the mixture is prevented when the proportion is not higher than 5 : 1.—N. W. T.

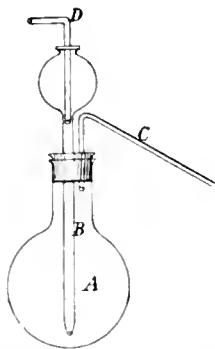
### INORGANIC CHEMISTRY.— QUANTITATIVE.

*Some Observations on the Relations of the Ferric and Aluminium Hydroxides, and of some Iron and Aluminium Salts to each other.* E. A. Schneider. Ber. **23**, 1349—1354.

See under VII., pages 857—858.

*Determination of Nitric Acid in Drinking Water by Schulze-Tiemann's Method.* L. Spiegel. Ber. **23**, 1361—1363.

SEVERAL years ago the author showed (Zeits. für Hygiene, 1887, 11, 163) that exact determinations according to Schulze-Tiemann's method can only be obtained if towards the end of the process the gas in the flask be expelled by a current of carbonic acid. The tests there described showed that an error of 3 per cent., or more, of the actual quantity of nitric acid present is the consequence of working according to Tiemann. In the new edition of his "Wasseruntersuchung," Tiemann does not, however, accept the proposals made in this direction, remarking (but without stating in figures his results of testing) that he has obtained just as good results by producing for a second time a partial vacuum in the flask. His first objection to the application of carbonic acid is that he considers it impossible to obtain this gas sufficiently free from air. This objection according to Spiegel is untenable, as he obtains a gas of quite sufficient purity by using small pieces of marble, which are placed some time in warm water before being put into Kipp's apparatus. The second objection to carbonic acid is, that it renders the apparatus more complicated in use, because of the necessity of a new tube being introduced. But this can easily be avoided by the following construction of the apparatus,



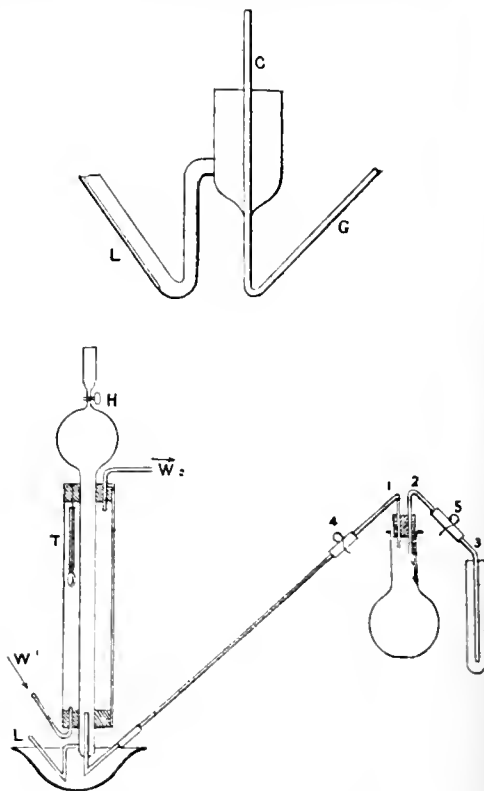
THE DETERMINATION OF NITRIC ACID.

which the author considers even more simple than that of Tiemann, because it dispenses with the partial vacuum. B, which is a tube terminating 2 cm. above the bottom of A, has at its upper end a bulb having a capacity of about 50 cc. The tube D is ground into the lower end of this bulb, the tube D being in connexion with the carbonic acid apparatus. The solution to be tested having been introduced

into the flask, carbonic acid is introduced through D, and the flask heated till the liquid boils gently; and this is continued until the gas going off through the tube C is free from air, which can be tested with a test tube containing caustic potash. The measuring tube is now placed over the delivery end of C, then 20 cc. of freshly-prepared and well-boiled ferrous chloride solution are introduced into the bulb forming the upper part of B, the solution flowing into the flask A when D is raised. In the same way, 40 cc. of concentrated and well-boiled hydrochloric acid are added. As soon as the liquid in the flask begins to boil again, the current of carbonic acid is stopped, and only towards the end of the reaction this gas is introduced again into the flask. The author mentions as a matter of course that the treatment with carbonic acid does not allow any unboiled reagents to be used in the test, and he is of opinion that in using these simple precautions this method gives more reliable results than any other.—H. S.

*An Improved Apparatus for the Estimation of Nitrates by Schulze-Tiemann's Method.* F. Scheiding. Chem. Zeit. **14**, 635—637.

THE nitric oxide produced by the decomposition of the nitrate is measured in a gas burette, consisting of a tube of about 100—180 cc. capacity, graduated in 0.2 cc. and enlarged at the top to a bulb fitted with a tap-funnel of about 20 cc. capacity. At the bottom the measuring tube is cut off straight, and is joined by a short piece of rubber tubing to the arrangement shown enlarged in the drawing.



THE ESTIMATION OF NITROGEN.

G is connected to the tube leading from the gas evolution flask, whilst L is joined by a long piece of rubber tube to the bottom of a flask of about 500 cc. capacity which serves to contain the caustic soda solution. A space of at least 10 cc. must exist between the 180 cc. mark of the burette and the top of the tube G. The straight portion of the burette is surrounded by a water jacket, in which hangs a thermometer, T. The burette is filled with a soda solution

of 1.25 sp. gr., and it is used until its density falls to 1.10—1.15. In carrying out an analysis, the nitrate to be analysed is washed with as little water as possible into the decomposition flask of about 200—250 cc. capacity. The soda solution is allowed to rise up to the tap 4, which is then closed. The air in the flask is now expelled through the tube 2, by briskly boiling the nitrate solution. The tap 5 is then closed, and tap 4 momentarily opened in order to expel the trace of air in tube 1 into the burette. The flame being removed, 20—25 cc. of a cold saturated solution of ferrous chloride are placed in the test tube and allowed to enter the flask by cautiously opening tap 5; 8—10 cc. of strong hydrochloric acid and two portions of water are passed into the flask in the same way. No trace of iron solution must remain in the tube 2. Whilst the contents of the flask are being gently warmed, the burette is filled by raising the soda-reservoir and opening the tap H to let out the air. The reservoir is then placed on the table, and a dish of cold water is put underneath the burette to cool the gas tube. As soon as the expansion of the rubber tubing indicates the existence of pressure in the flask, the tap 4 is opened and the gas driven over into the burette by evaporating the solution in the flask almost to dryness. Tap 4 is then closed and No. 5 opened. During the course of the distillation, the cooling water is adjusted to the same temperature as that indicated by a thermometer hung close to the bulb of the burette. When the temperature has remained constant for about 10 minutes, the gas volume may be read off. In accurate analyses it is necessary to equalise the densities of the soda solution in the burette and flask by sucking a quantity of fresh soda solution into the burette through the tap H. The results are calculated by means of the following formula:—

$$\text{Per cent. N} = \frac{V(b-f) \cdot 273 \times 0.6281}{760(273+t)} \times \frac{100}{g};$$

Where—

V = Volume as read off.

b = Height of barometer in mm. reduced to 0° C.

f = Tension of vapour from soda solution.

g = Weight of substance taken in mgms.

0.6281 = Weight of 0.5 cc. of nitrogen at 0° C. and 760 mm. in mgms.

$$\text{Now } \frac{273 \times 0.6281 \times 100}{760} = 22.562;$$

and if  $\frac{22.562}{273+t} = K$ , then

$$\text{Per cent. N} = \frac{V(b-f)K}{g};$$

Or  $\log. \text{ per cent. N} = \log. V + \log. (b-f) + \log. K - \log. g$ .

The values of  $K = \frac{22.562}{273+t}$  at different temperatures  $t^\circ$ , are given in the following table:—

$t^\circ \text{ C.}$	K.	$t^\circ \text{ C.}$	K.	$t^\circ \text{ C.}$	K.
11.0	0.07944	16.5	0.07793	22.0	0.07648
11.5	0.07930	17.0	0.07780	22.5	0.07635
12.0	0.07916	17.5	0.07767*	23.0	0.07622
12.5	0.07903*	18.0	0.07753	23.5	0.07609
13.0	0.07889*	18.5	0.07740	24.0	0.07597*
13.5	0.07875	19.0	0.07727*	24.5	0.07584*
14.0	0.07861	19.5	0.07714*	25.0	0.07571
14.5	0.07848*	20.0	0.07700	25.5	0.07558
15.0	0.07834	20.5	0.07687	26.0	0.07546*
15.5	0.07820	21.0	0.07674	26.5	0.07533
16.0	0.07807*	21.5	0.07661		

\* These numbers have been increased by one in the last place of decimals.

—H. T. P.

*On the Reduction of Nitric Acid to Ammonia, and on the Estimation of Nitric Acid.* E. Boyer. *Compt. Rend.* 110, 954—956.

The reduction of nitric acid by means of zinc and hydrochloric acid generally gives rise to oxides of nitrogen and nitrogen, as well as to ammonia, and hence all attempts to estimate nitric acid by this reaction have hitherto failed. The author has determined the conditions under which ammonia only is produced, and describes a method for estimating nitric acid under these conditions.

The solution to be estimated must not contain more than 3.17 per cent. of  $\text{N}_2\text{O}_5$  (corresponding to 5 per cent. of pure  $\text{NaNO}_2$ ). Five grms. of granulated zinc, in pieces the size of a pea, are introduced into the bottom of a test tube 30 centimetres long, 2.2 centimetres in diameter, and provided with a lip, and a quantity not exceeding 10 cc. of the nitrate solution is then poured from a pipette into the bottom of the tube, and 5 cc. of hydrochloric acid, of density 1.19, are poured down the sides to wash down any traces of nitrate. The tube is then agitated by a circular motion till the evolution of hydrogen ceases; a fresh quantity equal to 5 cc. of hydrochloric acid is then added, which completes the reduction of the nitric acid. After 10 minutes this reduction is complete, even though hydrogen continue to come off. No loss takes place by projection or evaporation, owing to the length of the tube. The contents of the tube and wash-water are introduced into a flask, the zinc precipitated by potash, 2 grms. of caustic magnesia added, and the liquid distilled with an ordinary Schloesing condenser, the ammonia being driven off into a known quantity of standard acid. Magnesia is used instead of an excess of potash or soda, as both of these bases give rise to double salts with ammonia, which only decompose after a considerable time.

To ensure success, the precautions described are absolutely necessary; if the quantities of solution taken be altered, or the density of the acid be less than 1.19, oxides of nitrogen will be formed, and show their presence by the yellow colouration of the liquid. The liquid should remain perfectly colourless.—P. J. H.

*A New Method of Estimating the Free and Combined Carbon in Iron and Steel.* O. Pettersson and A. Smith. *Ber.* 23, 1401—1402.

From 0.4 to 0.8 gm. of the metal in the form of a single rolled or hammered piece is dissolved in fused potassium bisulphate, by which operation—requiring from 5 to 12 minutes—the iron is converted into ferric sulphate, and the combined carbon oxidised into carbonic anhydride which is evolved together with sulphurous anhydride. These gases are led into and absorbed by a mixed solution of sodium and barium hydrates, which solution is then treated first with potassium permanganate to oxidise the sulphite to sulphate, and then with nitric acid to liberate the carbonic anhydride, which is then estimated by the method described in the following abstract. This gives the combined carbon. The graphite, which remains unchanged in the melt, is obtained by dissolving the sulphates in warm hydrochloric acid, and filtering through asbestos in a small platinum filter. The whole is dried, gently ignited, and weighed, then placed in a glass tube and ignited in a stream of air laden with nitrous fumes, and again weighed. The difference between the two weighings represents the graphite.

—A. W.

*A Method for Estimating Carbonic Anhydride.* O. Pettersson. *Ber.* 23, 1402—1406.

This apparatus serves for the estimation of carbonic anhydride either combined or in solution. The solid or solution is introduced into the flask B, and the carbonic anhydride is liberated by means of acid and by boiling, and is measured in D. A piece of iron or aluminium wire is placed in B so as to furnish a small stream of hydrogen. This serves a double purpose, firstly to sweep out the dissolved gas, and secondly to prevent violent ebullition. At starting, the liquid containing carbonic anhydride is introduced into B

up to the mark *x*, the connections are then made, acid is poured into A, F lowered and the acid allowed to flow in through *a* until the mark *y* is reached, when *a* is closed. F is now considerably lowered so as to exhaust C, and *b* is then closed. The reservoir F is raised until the level of the mercury is the same in the two tubes, *d* is opened, the level of the alkali in the Orsat tube E is accurately adjusted to the mark on the capillary and the volume of the air in D noted. The air is now passed into E until the carbonic anhydride is absorbed and is again measured as before. Unless the liquid was rich in carbonic anhydride the result obtained by these first measurements would only represent the carbonic anhydride contained in the ordinary atmosphere. The distillation of the liquid in B is now proceeded with, the bulk of the air in D is expelled through *c*, the rest is transferred to E until the next measurement; F is lowered and *b* partially opened to allow a quantity of gas to pass over. When sufficient is in D, *b* is closed and the carbonic

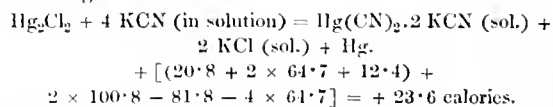
anhydride measured as before. This operation is repeated and the evolution of hydrogen continued until the gas shows no contraction after treatment with the potash in E. The various contractions are then added together and give the total result.—A. W.

*The Estimation of Phosphorus in the "Basic" Siemens Steel Bath.* W. Galbraith. Paper read before the Iron and Steel Institute, May 1890.

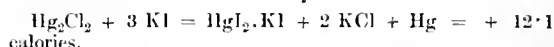
See under X., page 864.

*The Reciprocal Action of Mercurous and Alkaline Haloid Salts.* A. Ditte. Compt. Rend. **110**, 1330—1333.

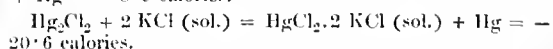
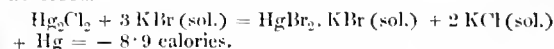
This is a thermo-chemical paper, in which the author describes the results of his investigations on the interaction of mercurous and potassium haloid salts. Potassium cyanide decomposes mercurous chloride in the cold in the following manner:—



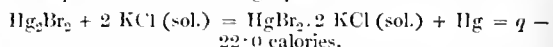
Potassium iodide acts similarly.



In the case of chloride and bromide of potassium heat is absorbed.



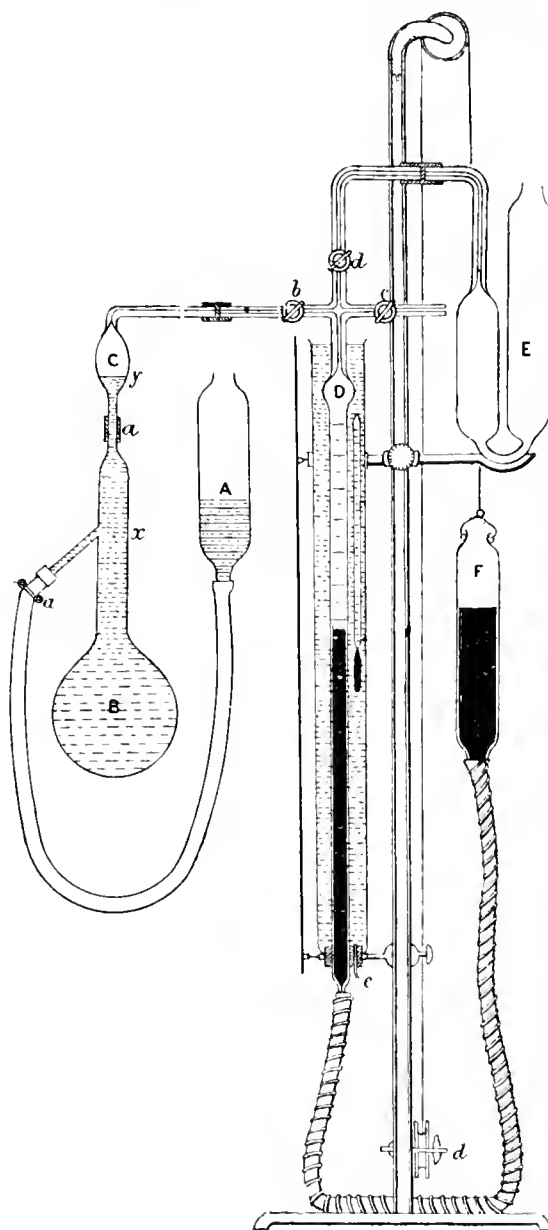
The reactions which take place between mercurous bromide and potassium cyanide, iodide, and bromide are similar to the above, the nett amounts of heat disengaged being + 15·4, + 4·3, and - 12·4 calories respectively. In the case of potassium chloride the maximum thermal effect corresponds to the following equation:—



This reaction is certainly endothermic, since the heat of solution of mercuric bromide in an excess of potassium chloride is less than the difference between the heats of formation of mercuric and mercurous bromides. Mercurous iodide is decomposed by potassium cyanide in the same way as the chloride, the heat liberated being + 5·6 calories. By the action of potassium iodide, bromide, and chloride on mercurous iodide, mercury is liberated, the mercuric iodide formed being simply dissolved in the excess of alkaline haloid salt, without any double decomposition taking place. The amounts of heat are - 8·0, - 13·6, and - 13·6 calories respectively. The heat of solution of mercuric iodide in potassium bromide and chloride is insignificant. The fact that those of the above reactions which are endothermic are realisable in practice may be easily understood, when it is considered that mercurous haloid salts are decomposed by water alone, especially when boiling and often changed, into mercury and mercuric salts. In the presence of an alkaline haloid salt, the mercuric compound forms with it a double salt. The equilibrium existing between the mercurous and mercuric compounds, the mercury and the water is thus destroyed, and a fresh portion of mercurous salt is decomposed in order to restore it. In this manner the action goes on until only mercury is left.—H. T. P.

*The Electrolysis of Metallic Phosphates in Acid Solution.* E. F. Smith. Amer. Chem. J. **12**, 329—336.

THE course pursued in the preparation of the solutions for electrolysis was to first precipitate the metal with an excess of di-sodium phosphate, dissolving the compound obtained in a measured volume of free phosphoric acid, and then exposing the liquid to the action of currents of known



THE ESTIMATION OF CARBON DIOXIDE.

strength. Mercurous, lead and bismuth phosphates being nearly insoluble in phosphoric acid were therefore not available for these experiments.

**Copper.**—To a solution containing 0.0996 gm. of metallic copper as sulphate, 10 cc. of  $\text{Na}_2\text{HPO}_4$  (sp. gr. 1.0358) and 3.5 cc. of  $\text{H}_3\text{PO}_4$  (sp. gr. 1.347) were added. The precipitated copper phosphate dissolved in 0.5 cc. of acid so that the deposition of metal occurred in the presence of 3 cc. of free phosphoric acid. The total dilution with water amounted to 125 cc. The current employed gave 0.15 cc. of electrolytic gas per minute. The current in two other experiments was increased to 0.5 cc. electrolytic gas per minute. Strong currents deposit metallic iron and other metals from their phosphates in phosphoric acid solution. The results obtained appear very satisfactory.

**Copper from Iron.**—A solution containing as before 0.0996 gm. of copper with the addition of 0.1700 gm. of metallic iron, to which 30 cc. of di-sodium phosphate and 4.5 cc. of phosphoric acid solution were added, was decomposed by a current giving 0.6 cc. of gas per minute. The whole of the copper was deposited.

**Copper from Aluminium.**—A copper solution containing aluminium was treated as above with a current giving 0.3 cc. of gas per minute; the copper deposited weighed 0.0995 gm.

**Copper from Chromium.**—A solution similarly treated with the same strength of current as in the separation of the aluminium, deposited 0.0994 gm. of the copper, and converted the chromium into chromic acid.

**Copper from Zinc.**—A solution containing the same quantity of copper with the addition of 0.1500 gm. of zinc, deposited 0.0993 gm. of copper. The strength of current was 0.15 cc. of electrolytic gas.

**Copper from Cobalt.**—With 0.0968 gm. of cobalt, solution as before, with a current of 0.22 cc. of gas, the copper deposited was 0.0995 gm.

**Copper from Nickel.**—The nickel (0.1105 gm.) was present as chloride, the other conditions being the same as in the preceding example. The deposit of copper weighed 0.0996 gm.

In all the above experiments the copper deposited rapidly from cold solutions. The deposition was allowed to continue all night. Before interrupting the current the acid liquid was siphoned off and replaced by water. The deposits were washed with cold and hot water.

**Cadmium.**—Ten cc. of cadmium sulphate (= 0.1827 gm. of Cd) were precipitated by an excess of di-sodium phosphate. The phosphate dissolved in 1.5 cc. of phosphoric acid ( $\text{H}_3\text{PO}_4$ ). The current gave 0.6 cc. of gas.

Two determinations with the strength of current yielded (a) 0.1839 gm. and (b) 0.1820 gm. The cadmium deposit in (a) showed a little sponginess and did not form as rapidly as the copper. It was found advisable towards the close of the experiment to increase the current strength. With a current of 0.40 cc. the deposits obtained were (a) 0.1828 gm. and (b) 0.1833 gm.

**Cadmium from Zinc.**—A solution containing 0.1827 gm. of cadmium and 0.1500 gm. of zinc treated with di-sodium phosphate and phosphoric acid and diluted to 125 cc., deposited 0.1820 gm. of cadmium in 12 hours when acted upon in the cold with a current evolving 0.35 cc. of gas per minute.

**Cadmium from Nickel.**—The experiments were conducted as above and gave good results. The separation of cadmium from iron, chromium and aluminium gave equally good results. The solution in these cases was made up to 100 cc.

The separation of copper from the metals mentioned in connexion with it, is not attended with any difficulty, but with cadmium compliance with the conditions given is absolutely necessary, as the results otherwise vary. In solutions containing free phosphoric acid, cadmium is usually inclined to sponginess, so that concentration of liquid should be avoided, and the poles of the acting

battery should not approach too closely to each other. The most favourable separation was found to be  $1\frac{1}{4}$  in., and the proper dilution of the liquid 100—150 cc.

**Copper from Cadmium.**—A solution containing 0.2452 gm. of copper and 0.1827 gm. of cadmium as sulphates with 20 cc. of di-sodium phosphate and 10 cc. of phosphoric acid diluted to 125 cc., deposited in two experiments 0.2451 and 0.2452 gm. respectively of copper, with a current giving 0.10 cc. of gas per minute. With a current of 0.2 cc. the deposit weighed 0.2451 gm.

Silver phosphate, in a phosphoric acid solution, when electrolysed gives a deposit of spongy metal. When the phosphate is dissolved in the least possible quantity of ammonia, the deposit is coherent.

Manganese is not deposited as dioxide at the positive pole if the phosphate strongly acidified with phosphoric acid be electrolysed. The author utilises these conditions to effect the separation of manganese and copper.—J. B. C.

#### *The Dry Assay of Tin Ores.* Part I. H. O. Hofman. Technology Quarterly, **3**, 112—143.

THE author has studied practically the application of the various methods for the dry assay of tin to the black tin-stone of the Black Hills, where its occurrence is different to that in other parts of the world. He prepared a large quantity of the pure cassiterite for his experiments, and by a careful process of pulverising, screening, roasting to free from sulphur and arsenic, treating with acids and finally washing, a product was obtained which gave by wet assay 86.24 per cent. of  $\text{SnO}_2$ , or 67.84 per cent. of Sn. This material was then subjected to various modifications of the three usual methods of assay. The German method (the reduction with charcoal, black flux, borax and salt) when carefully carried out gave very good results, averaging 67.58 and 67.46 per cent. of Sn. The method was varied by prolonging the time of fusion, using chalk-lined crucibles, and by heating with charcoal first and then adding the fluxes, but in no case were such satisfactory results obtained. The method of fusion with fluorspar, however modified, gave very bad results. The cyanide method when pure cyanide was used (98 per cent. of KCN and 2 per cent. KOCN) gave results averaging 67.30 per cent. Various modifications were tried, such as using salt, potassium carbonate or charcoal with the cyanide and employing lined crucibles, but in every case worse results were obtained. Potassium carbonate lowers the results more and more as the amount present increases, hence, in the assay of tin ore common cyanide should be avoided. The cyanide "pure for gold platers" (92 per cent. of KCN, 5 per cent. of KOCN, and 3 per cent. of other salts) gave average results of 67.10 per cent., and the common fused variety 60.76 per cent. Full details of the experiments and results are given in the paper. The author finds that no advantage is obtained by lining the crucibles in any way, equally good and sometimes better results being obtained in the ordinary Battersea crucible.

—A. W.

#### ORGANIC CHEMISTRY.—QUALITATIVE.

##### *The Detection of Noxious Mineral Substances in Wine.* L. Liebermann. Chem. Zeit. **14**, 635.

**Detection of Copper, Lead, and Arsenic.**—100 cc. of the wine are evaporated down to one-half, 10—15 cc. of hydrochloric acid then added, and the mixture diluted to 100 cc. with strong sulphuretted hydrogen water. In absence of metals of the second group, the wine remains absolutely clear and is unaltered in colour. In presence of these metals a more or less distinct precipitate is formed and the wine is discoloured. The precipitate is filtered off and washed, first with water and then with alcohol. When its quantity is small the paper is only covered with a yellowish, brownish or blackish film. In presence of 0.01 per cent. of copper, lead, or arsenious acid, the reaction is quite strong, and even when only 0.001 per cent. is present, a distinct reaction is obtained. In order to identify the

separate metals, the precipitate is dissolved in dilute nitric acid, and the solution boiled and filtered. In the filtrate, lead is tested for by means of sulphuric acid. The solution freed from lead is boiled to expel nitric acid. In one portion of this solution the copper is identified by adding ammonia or potassium ferrocyanide; to the other portion a little pure zinc is added, and the hydrogen arsenide thus produced is detected by its action on a drop of silver nitrate solution spread on filter-paper. A citron-yellow spot surrounded by a black ring is formed; on the addition of a few drops of water the spot turns black all over.

**Detection of Zinc.**—The wine is evaporated down to one-half, made alkaline with ammonia, and then acid by the addition of an excess of acetic acid, and finally diluted to its original bulk with strong sulphuretted hydrogen water. In presence of zinc, a grey, or in the case of red wines, a violet-grey precipitate is formed. The thoroughly washed precipitate is dissolved in dilute hydrochloric acid, the solution being boiled to expel sulphuretted hydrogen. The solution is again filtered and mixed with excess of caustic potash solution. The liquid is filtered through glass-wool or very closely-woven linen. In the filtrate, ammonium chloride produces in the presence of zinc a precipitate soluble in excess of ammonium chloride.—H. T. P.

**Detection of Diresorcinol in Synthetic Phloroglucinol.**  
J. Herzog and S. Zeisel. Chem. Zeit. 14, 1064.

DIRESCORINOL, when dissolved in concentrated sulphuric acid and treated with glacial acetic acid, and the mixture heated, gives an intense violet colour. By this test the fact has been established that phloroglucinol purified according to Will's method still contains diresorcinol, whilst that prepared according to Skraup was free from it. Phloroglucinol prepared from phloroglucinol acetate was pure, even when the latter was made from material containing diresorcinol.—W. M.

**The Acidity of Vegetable Oils.** Holde. Mitt. königl. techn. Versuchs. 1890, 78—82.

THE author examined many samples, particularly of rape oil, both for mineral and free fatty acid. The former, together with that present as sulphonic acids in the oil, was recognised in the usual way by shaking and heating with water. He records the familiar fact that mineral acids are seldom present even in traces in properly refined oils. More interesting results are afforded by a lengthy table, the mean values of which show an average percentage of free fatty acid in unrefined rape oil of 0.15 per cent., calculated as sulphuric anhydride, corresponding to 1.08 when reckoned as oleic acid, while the numbers for refined rape oil are 0.35 and 2.45, and for cotton-seed oil 0.78 and 5.51. In connexion with these last figures it must be stated that certain of the samples were abnormally acid and thus raised the average.

Further experiments were made to ascertain the rate of increase of acidity suffered by oils exposed to the air for considerable periods. In general, the lapse of about a year and a half caused an increase of 20—40 per cent. of the original acidity in the case of an oil with a low proportion of free fatty acid, while in one instance of a cotton-seed oil, very acid and rancid besides, the increase was from 1.46 per cent., calculated as sulphuric anhydride, in June 1889, to 2.82 in March 1890. (These figures can be very approximately converted into percentages of oleic acid by multiplication by 7.)—B. B.

**New Reactions of Albuminoids.** C. Reichl. Monatsh. 11, 155—165.

IT was shown a short time ago that a blue colouration is produced when an albuminoid is treated with dilute sulphuric acid, ferric sulphate, and an alcoholic solution of benzaldehyde or salicylaldehyde. In these reactions the ferric sulphate plays the part of an oxidising agent and can be replaced by very dilute nitric acid, mercuric oxide, &c. The blue condensation-product is the sulphate of a base; it is decomposed by alkalis with separation of a brownish

precipitate which dissolves in acids yielding a blue or bluish-green, and in alkalis giving a yellowish solution. The reaction seems to be caused either by the skatol- or the indol-group of the albuminoid molecule, most probably by the former, since skatol itself gives colour reactions very similar to those produced in the case of albuminoids.

Benzyl alcohol, and resins containing derivatives of benzyl alcohol, Peru- and Tolu-balsam, for example, give a bluish-green or bluish-violet colouration with ferric sulphate, dilute sulphuric acid, and an albuminoid, amygdalin; on the other hand, gives no colouration unless more concentrated sulphuric acid is employed.

The reaction with salicylaldehyde is carried out as follows: The albuminoid in a solid condition is treated with one drop of a 0.5 per cent. alcoholic solution of salicylaldehyde, the alcohol is allowed to evaporate, and several drops of dilute sulphuric acid containing ferric sulphate are added; after a short time eggs, blood-, and plant-albumen, blood-fibrin, and casein give a violet-blue, legumin a brownish-violet, and plant fibrin a brownish-yellow colouration; sheep's wool and animal membrane are coloured bluish-violet under the same conditions. When the albuminoid is in solution a drop of a 0.5 per cent. alcoholic solution of salicylaldehyde and an equal volume of concentrated sulphuric acid are added, the mixture is cooled, but without shaking, and then a drop of ferric sulphate solution is added; a blue to violet zone is observed after a longer or shorter time according to the diluteness of the solution.

The oil of *Spica ulmaria* gives the same reactions as salicylaldehyde; with salicin, populin, and saligenin the colouration is produced more slowly and is less intense.

Anisic aldehyde, under the conditions described above, gives with egg-albumen, plant-albumen, and casein a violet, with blood-albumen and sheep's wool a violet-red, with blood-fibrin a blue, with legumin a brownish-violet, and with plant-fibrin a reddish-yellow colouration, the reactions being produced more quickly than is the case with benzaldehyde and salicylaldehyde. Anethol, anisol, Indian anise oil, esdragon oil, and fennel oil react like anisic aldehyde, only more feebly; if mercuric oxide be used instead of ferric sulphate the colourations are more intense.

Vanillin, under the same conditions, gives a violet or violet-blue colouration with egg-, blood-, and plant-albumen, casein, blood-fibrin, sheep's wool, and animal membrane, whilst with legumin it gives a brownish red, and with plant-fibrin a pale violet colouration. Vegetable products containing vanillin, such as vanilla, assafetida, &c., give the same reactions.

When wood, which according to Singer always contains vanillin, is heated with a dilute sulphuric acid solution of egg-albumen containing a little ferric sulphate, it becomes green and then darkens, the solution turning violet; the wood is probably coloured by the sulphuric acid, but the colouration of the solution is probably due to the action of the vanillin on the albumen.

Piperonal gives the same colourations as vanillin; para-cuminaldehyde and cinnamic aldehyde give a slight colouration with some, but no reaction with other albuminoids, whilst those obtained with furfuraldehyde are very feeble.

The various albuminoids do not all give these colour reactions with the same intensity; in the case of legumin and plant-fibrin, for example, the colouration is sometimes difficult to detect or is not produced at all. Piperonal, vanillin, anisic aldehyde and cinnamic aldehyde produce a colouration immediately when the albuminoid is in the solid condition or in moderately concentrated solution, but with salicylaldehyde, benzaldehyde, furfuraldehyde, and cuminaldehyde the colouration is not produced for some time; a 0.1 per cent. solution of an albuminoid requires an hour, and more dilute solutions an even longer time before a colouration is produced.

The reactions with aldehydes are not as delicate as some of the older ones, except when the albuminoid is in a solid state, because there is a maximum proportion between the albuminoid and the acid solution which should not be exceeded; this proportion is 1 part of albuminoid to 6,000 parts of dilute sulphuric acid in the case of salicylaldehyde, 1 to 10,000 in the case of benzaldehyde, and 1 to 40,000 in

the case of anisic aldehyde, vanillin, and piperonal. Aqueous solutions should seldom contain more than 1 part of albuminoid in 3,000 parts of water or the reaction will not take place.

Hydrogen peroxide, in presence of dilute sulphuric acid, gives after a short time a reddish colouration with albuminoids, but the colouration disappears on the addition of ferric sulphate.—F. S. K.

### Tests for Cocaine Hydrochloride.

See under XX., page 888.

## ORGANIC CHEMISTRY.—QUANTITATIVE.

*The Influence of Solvent or Medium on the Speed of Reaction.* N. Menshutkin. J. Russ. Chem. Soc. **22**, 347—358 and 393—409.

In his previous investigation on the same subject (J. Russ. Chem. Soc. **19**, 642) the author had come to the following conclusions:—

(1) The speed of a reaction may vary considerably, being carried on in different solvents or mediums; (2) the chemical process could not be considered independently of the solvent or medium in which it is carried on; (3) the latter enters in every chemical reaction with a certain coefficient.

The present investigation concerns the details of the same subject under two principal aspects; *a*, as to the influence of different solvents on a given reaction; and *b*, as to the speed of different reactions in a given solvent.

The author has chosen the reaction of triethylamine with ethyl iodide as most convenient for the purpose. It was carried on in 15 volumes of different liquid solvents which are chemically quite indifferent to either of the reagents. It has been found that under this condition, at the temperature of 100°, and within the same space of time considerably different amounts of tetra-ethylammonium iodide,  $(C_2H_5)_4NI$ , are formed.

These results are expressed in the adjoining table; the first column showing 23 different solvents, the second containing the constants of speed of the reaction, the third, the relative speed of reaction in the corresponding solvent, taking the lowest one, the case of hexane, as 1·0, thus illustrating the quickening influence of different solvents; while the fourth shows the same relative speed of the reaction, taking the greatest speed, in case of benzyl alcohol, as 100, thus illustrating the retarding influence of the solvents.

Solvents.	Constant.	Relative Speed.	
HYDROCARBONS.			
Hexane, $C_6H_{14}$ .....	0·000180	1·0	0·13
Heptane, $C_7H_{16}$ .....	0·000235	1·3	0·17
Xylene, $C_6H_4(CH_3)_2$ .....	0·00287	15·9	2·2
Benzene, $C_6H_6$ .....	0·00584	38·2	4·4
Propyl-chloride, $C_3H_7Cl$ .....	0·00540	30·0	4·0
Chloro-benzene, $C_6H_5Cl$ .....	0·0231	128·0	17·4
Bromo-benzene, $C_6H_5Br$ .....	0·0273	150·0	20·3
Bromo-naphthalene, $C_{10}H_7Br$ .....	0·1129	627·0	84·9
SIMPLE ETHERS.			
Ethyl-amyl ether, $C_2H_5.O.C_5H_{11}$ .....	0·000630	3·5	0·47
Ethylether, $C_2H_5.O.C_2H_5$ .....	0·000757	4·2	0·57
Phenetol, $C_2H_5.O.C_6H_5$ .....	0·0212	117·7	16·0
Anisol, $CH_3.O.C_6H_5$ .....	0·0259	223·9	30·3

Solvents.	Constant.	Relative Speed.	
MIXED ETHERS.			
Isobutyl acetate, $C_4H_9.O.CO.CH_3$ ...	0·00577	32·1	4·3
Ethyl acetate, $C_2H_5.O.CO.CH_3$ .....	0·0223	123·9	16·7
Ethyl benzoate, $C_2H_5.O.CO.C_6H_5$ .....	0·0259	143·9	19·4
ALCOHOLS.			
sobutyl alcohol, $C_4H_9.OH$ .....	0·0258	143·3	19·4
Ethyl alcohol, $C_2H_5.OH$ .....	0·0336	203·3	27·5
Allyl alcohol, $C_3H_5.OH$ .....	0·0333	240·5	32·5
Methyl alcohol, $CH_3.OH$ .....	0·0516	286·6	38·0
Benzyl alcohol, $C_6H_5.CH_2.OH$ .....	0·143	742·2	100·0
KETONES.			
Acetone, $CH_3COCH_3$ .....	0·0608	337·7	45·7
Acetophenone, $CH_3.CO.C_6H_5$ .....	0·1294	718·7	97·3

This table shows that a given reaction goes on in benzyl alcohol 742 times quicker than in hexane; this being the maximum difference of speed shown in the table, the author thinks there may be expected still more favourable solvents for the reaction in alcohols and ketones of the naphthalene series.

The author finds the cause of these differences in the composition and structure of the solvent. The illustrations to be found in the same table. (*a*.) In hydrocarbons and simple ethers the reaction goes on the slowest; quicker in mixed ethers, still quicker in ketones, but the quickest in alcohols (and of course in acids, which could not be tried in this case).

### I.

	Relative Speed.
Hexane, $C_6H_{14}$ .....	1·0
Ethyl ether, $C_2H_5.O.C_2H_5$ .....	4·2
Ethyl acetate, $C_2H_5.O.CO.CH_3$ .....	123·9
Ethyl alcohol .....	203·9
Acetone, $CH_3.CO.CH_3$ .....	337·7

### II.

	Relative Speed.
Benzene, $C_6H_6$ .....	38·2
Phenetol, $C_6H_5.O.C_2H_5$ .....	117·7
Ethyl benzoate, $C_2H_5.CO.OC_2H_5$ .....	173·9
Acetophenone, $C_6H_5.CO.CH_3$ .....	718·7
Benzyl alcohol .....	742·2

Thus the compounds containing hydroxyl (HO), *i.e.*, alcohols, acids, and their derivatives, as mixed ethers and ketones, appear to be the most favourable for the speed of reactions.

(*b*.) Members of the same homologous series, of the same structure, used as solvents, affect the reaction as follows: the greater the molecular weight of the solvent, the smaller is the speed of the reaction.

### I.

	Relative Speed.
Methyl alcohol, $CH_3.OH$ .....	286·6
Ethyl alcohol, $C_2H_5.OH$ .....	203·3
Isobutyl alcohol, $C_4H_9.OH$ .....	143·8

### II.

	Relative Speed.
Anisol, $C_6H_5.O.CH_3$ .....	223·9
Phenetol, $C_6H_5.O.C_2H_5$ .....	117·7

(*c*.) The less saturated is the solvent the greater is the speed of the reaction. The comparison of allyl and aromatic compounds with those corresponding of the paraffin series fully illustrate this.

	Relative Speed.
Ethyl alcohol, $\text{C}_2\text{H}_5\text{CH}_2\text{OH}$ .....	203.3
Allyl alcohol, $\text{CH}_2\text{CH}=\text{CH}_2\text{OH}$ .....	240.5
Hexane, $\text{C}_6\text{H}_{14}$ .....	1.0
Benzene, $\text{C}_6\text{H}_6$ .....	38.2
Ethyl ether, $\text{C}_2\text{H}_5\text{O.C}_2\text{H}_5$ .....	4.2
Phenetol, $\text{C}_6\text{H}_5\text{O.C}_2\text{H}_5$ .....	117.7
Ethyl acetate, $\text{CH}_3\text{CO.O.C}_2\text{H}_5$ .....	123.9
Ethyl benzoate, $\text{C}_6\text{H}_5\text{CO.O.C}_2\text{H}_5$ .....	143.9
Acetone, $\text{CH}_3\text{CO.CH}_3$ .....	337.7
Acetophenone, $\text{CH}_3\text{CO.C}_6\text{H}_5$ .....	718.7
Ethyl alcohol, $\text{C}_2\text{H}_5\text{OH}$ .....	203.3
Benzyl alcohol .....	742.2

There is only one observation supporting author's expectation as to a still greater speed of the reaction in solvents of naphthalene series:—

	Relative Speed.
Bromo-benzene .....	150
$\alpha$ -Bromo-naphthalene .....	627

(d.) The substitution of chlorine for hydrogen increases the speed of the reaction; still more so the substitution of bromine and iodine.

	Relative Speed.
Benzene, $\text{C}_6\text{H}_6$ .....	38.2
Chloro-benzene, $\text{C}_6\text{H}_5\text{Cl}$ .....	128.0
Bromo-benzene, $\text{C}_6\text{H}_5\text{Br}$ .....	158.0
Hexane, $\text{C}_6\text{H}_{14}$ .....	1.0
Propyl chloride, $\text{C}_3\text{H}_7\text{Cl}$ .....	38.0

The substitution of different organic groups for hydrogen in benzene supplied the following results:—

	Relative Speed.
$\text{C}_6\text{H}_6$ .....	38.2
$\text{C}_6\text{H}_5(\text{OC}_2\text{H}_5)$ .....	117.7
$\text{C}_6\text{H}_5(\text{CO.O.C}_2\text{H}_5)$ .....	143.9
$\text{C}_6\text{H}_5(\text{O.CH}_3)$ .....	223.9
$\text{C}_6\text{H}_5(\text{CO.OCH}_3)$ .....	718.7
$\text{C}_6\text{H}_5(\text{CH}_2\text{OH})$ .....	742.2

This table shows that the groups containing oxygen, being substituted for hydrogen, make the solvent more favourable for the reaction than those containing chlorine and bromine.

The physical properties of the solvent have apparently very little importance in this respect. The researches of Kobilansky (*Z. f. phys. Chem.* 4, 29) show that hydrocarbon and simple ether solutions are the worst conductors of electricity, while weak alcoholic solutions are the best conductors.

The above results led the author to the conclusion that in case of a weak reaction it is advisable to use as solvents alcohols and ketones, while energetic reactions require to be carried on in hydrocarbon and ether solutions.

The same results may be applied to the chemical reactions between liquid compounds without any solvents; the excess of either of the reagents may be considered as such. To illustrate this, a series of experiments were made on the reaction of acetic acid on aniline:—

Parts of Aniline for 1 Part of Acetic Acid.	Relative Speed.
1 .....	34.7
2 .....	28.7
3 .....	23.5
4 .....	17.1

Parts of Acetic Acid for  
1 Part of Aniline.

1 .....	34.7
2 .....	57.3
4 .....	78.1

Thus, the excess of the aniline decreases the speed of the reaction, while the excess of the acid increases it.—N. W. T.

### The Flashing Point of Lubricating Oils. A. Martens Mitt. königl. tech. Versuchs. 1890, 71—78.

A SIMPLE plan used by Treumann, of Hanover, for determining the flashing point of lubricating oils—the apparatus consisting merely of a capsule placed in a hemispherical sand-bath, to protect it from air currents, the light being applied from time to time to the uncovered surface of oil in the capsule—is compared with the elaborate method in vogue at the Versuchsanstalten, in which the modified Abel-Pensky apparatus (this Journal, 1889, 734) is employed. The results show that for many practical purposes the simpler process is sufficiently accurate, though suffering from the inherent disadvantages of all “open-test” methods. A communication from Treumann incorporated in the paper shows that discrepancies are possible even with the modified Abel-Pensky, concerning the constancy of the results of which several points are at issue between him and the author.—B. B.

### Action of Reducing Agents upon Gallic Acid and Tannins. C. Böttlinger. Annalen. 258, 252—260.

METALLIC sodium was added in small portions to the solution of gallic acid in absolute alcohol. No reduction products were obtained. Tannic acid similarly treated appeared to undergo simple hydrolysis, the yield of gallic acid amounting to 60 per cent. of the weight of the tannin. Tannin dissolved in amyl alcohol, and heated with the metal at the boiling temperature of the alcohol, undergoes partial resolution into gallic acid, a portion combining with the alkyl to form ethereal derivatives. Acetoquercitanic acid is similarly attacked.

The results of the investigation in respect of its main object, which was to prepare reduction products of these compounds, were negative.—C. F. C.

### Derivatives of Diphenylhydrazine. R. Stabel. Annalen, 258, 242—251.

Diphenylhydrazine has been obtained by the author by distillation under diminished pressure (40—50 mm. at 220°) and recrystallising from petroleum spirit, in colourless tabular crystals belonging to the monoclinic system and melting at 34.5°.

*Compounds with the Sugars.*—The hydrazones are well crystallised bodies only slightly soluble in water.

*Glucosodiphenylhydrazine.*—To prepare this compound 1 part of grape sugar is dissolved in a small quantity of water and added to an alcoholic solution of the base (1.5 parts). The mixture is heated for 2 hours on the water-bath, the alcohol evaporated off, and ether is added to the solution, to remove the excess of base. The aqueous portion solidifies to a crystalline magma. The yield of hydrazone amounts to 75 parts of the theoretical. It is best purified by recrystallisation from its solution in hot water, this property being characteristic of the compound. In the pure state it melts at 161°—162°. The formation of this hydrazone affords an excellent method of identifying dextrose in presence of levulose. In cases where the solution cannot be heated the separation of the hydrazone may be completed by allowing the mixture to stand 2—3 days at the ordinary temperature.

*Mannosodiphenylhydrazine.*—This compound closely resembles the above, melting at 155° to a clear yellow liquid.

*Galactosodiphenylhydrazine* is also similar in properties to the above; melting point 157°.

*Rhamnosodiphenylhydrazine* is formed by heating the reagents in alcoholic solution 3—4 hours; or in a sealed tube at 100° for 2 hours. The product crystallises from its solution in hot water in colourless silky needles (melting point 134°).

### Furfurandiphenylhydrazine—



is rapidly formed when the aldehyde and base are brought together in equal molecular proportions. It crystallises from aqueous alcohol in yellowish needles (melting point 90°).

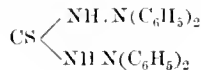


*Salicylaldehydediphenylhydrazone* is similarly obtained, crystallising from its solution in alcohol (80 per cent.) in colourless needles (melting point  $138^{\circ}5$ ).

*Diphenylhydrazine* and *Carbondisulphide* readily react with formation of diphenylthiocarbazine acid—



which crystallises from its solution in the disulphide in long golden yellow prisms (melting point  $109^{\circ}$ ). On heating with diphenylhydrazine the thio-urea of this base is formed—



—C. F. C.

*The Curves of Boiling Temperatures as a Function of the Chemical Nature of the Bodies. Simple Rule for the Determination of the Boiling Points of Organic Bodies at Reduced Pressures.* M. Wildermann. Ber. **23**, 1254—1264.

It is to see whether a relation exists between the pressure and the curves of boiling temperatures on the one hand, and the chemical nature of bodies on the other, the author has compared 20 different homologous series, and has found that within large limits of pressure the proportion of the absolute boiling temperature at M mm. pressure to that at N mm. pressure is almost the same for all members of a homologous series. Therefore the equation is practically correct

$\frac{T_a p}{T_a P} = \frac{T_b p}{T_b P} = \frac{T_c p}{T_c P} = D_1$  where  $a, b, c$  are different members of a homologous series,  $p$  and  $P$  less and higher pressures. This formula shows, first that  $T_a p = T_a P \times D_1$ , viz., that the absolute boiling temperature of a body at a lower pressure  $p$  is equal to its absolute boiling temperature at a higher pressure  $P$  multiplied by  $D_1$ , or by the proportion  $\frac{T_b p}{T_b P}$ , which may be calculated from any other member of the same homologous series. Secondly,  $T_a p : T_b p = T_a P : T_b P$ , viz., the relating of the absolute boiling temperatures of two members of a homologous series is almost the same at any pressure (between 12 mm. and 2 atmospheres). Thirdly—

$$\frac{T_a p - T_a P}{T_b p - T_b P} = \frac{T_a p}{T_a P} = \frac{T_b p}{T_b P}$$

viz., the difference of temperatures between two boiling points of a member of a homologous series at two different pressures is to the corresponding difference of temperatures of another member as the absolute boiling temperatures of these members are to each other at a pressure of M mm.

The author shows that these formulae may be used for the fatty acid series, partly for the aromatic series, and also for certain isomeric bodies, such as isomeric acids, alcohols, &c. He does not claim for these formulae any theoretical value, but has given them as mere rules for practical application in organic chemistry.—H. S.

*The Quantitative Estimation of Furfuraldehyde and Pentaglucooses.* A. Günther and B. Tollens. Ber. **23**, 1751—1752.

HITHERTO furfuraldehyde has been estimated by precipitation as hydrofurfuramide by means of ammonia. The method sufficed to prove that very little furfuraldehyde is obtained by the distillation of the hexaglucooses and their derivatives with hydrochloric acid or sulphuric acid, and that arabinose and xylose yield about 20—30 per cent. of their weight of furfuraldehyde. The method is, however, far from accurate, and yields very contradictory results.

The authors have succeeded in improving the distillation process with hydrochloric acid so that it yields constant quantities of furfuraldehyde, the latter being estimated volumetrically by means of phenylhydrazine, using aniline acetate as an indicator. The details will be given in a later

paper. For the present the authors content themselves by giving some of the results obtained by the distillation of various sugars with hydrochloric acid. Pure arabinose and xylose yield about 50 per cent. of their weight of furfuraldehyde; cherry gum, 15 per cent.; gum arabic, 14 per cent.; straw from wheat and oats, 13 per cent.; starch and sugar, about  $\frac{1}{4}$ — $\frac{1}{5}$  per cent.; vegetable ivory, 1 per cent.

Assuming that pentaglucooses yield half their weight of furfuraldehyde, it will be seen that cherry gum contains 30 per cent. of these bodies; gum arabic 28 per cent.; and wheat and oat straw, 26 per cent. Glycuronic acid gives 46 per cent. of furfuraldehyde, practically the same as arabinose, whilst only a trace is obtainable from casein.

Finally, the authors mention that the non-crystallisable levo-rotatory sugar which Bieler and Tollens have obtained from the "fecus" by the action of sulphuric acid, is considered by them to be different from all sugars hitherto described, including rhamnose.—H. T. P.

*An Adulteration of Linseed Oil.* A. Aignau. Compt. Rend. **110**, 1273—1275.

A COMMON adulterant of linseed oil is rosin oil. Pure linseed oil being, contrary to the assertions of some writers, destitute of rotatory power, rosin oil may be determined in a mixture with linseed oil by its action on polarised light. The rotation is to the right, and varies somewhat with the kind of rosin oil that has been used. If  $[\alpha]_D$  be called the rotation observed in a 100 mm. tube, and  $h$  the weight of rosin oil contained in the mixture, the value of the latter is given by the formulae:—

$$h = [\alpha]_D \cdot \frac{15}{7} \text{ for refined rosin oil.}$$

$$h = [\alpha] \cdot \frac{30}{17} \text{ for best white rosin oil.}$$

$$h = [\alpha]_D \cdot \frac{30}{21} \text{ for fine rectified rosin oil.}$$

The first is the kind commonly used; a 100 mm. tube is better than one 200 mm. in length on account of the somewhat deep colour usually possessed by the oil.

In the case of a paint, a quantity is shaken up with ether, allowed to settle, and the rotation given by the ethereal solution observed in a 200 mm. tube, the formula used

being  $h = \frac{[\alpha]_D}{43}$ . By evaporating a known quantity of the ethereal solution the total weight of oil can be obtained, and thus the percentage of rosin oil readily calculated.—B. B.

*New Process and Apparatus for Sugar Factories.* Dingl. Polyt. J. **276**, 567—572.

See under XVI., page 877.

#### ERRATUM.

This Journal, 1890, page 823, foot of column 1 and top of column 2, for "J. Ribau" read "J. Ribau," and for "thio-sulphate," "thiosulphates," and "thiosulphuric," read dithionate, dithionates, and dithionie respectively.

## New Books.

**THE RIVER IRWELL AND ITS TRIBUTARIES** (A Monograph on River Pollution). By GEORGE E. DAVIS and ALFRED R. DAVIS, Analytical and Consulting Chemists, Chemical Engineers. Manchester: John Heywood, Deansgate and Ridgefield. London: 1, Paternoster Buildings. 1890.

CROWN 8vo. volume, bound in cloth, price 2s. 6d. A work of 96 pages, with map of the whole of the Irwell watershed. It is the outcome of numerous analyses made in the laboratory of the authors, and it is claimed that though on several occasions isolated analyses have been published of various parts of the river, yet never before has a complete account been given of the watershed. The book is divided into eight chapters, preceded by the map of the watershed, on which is marked the principal towns and the position and name of every contributing stream and brooklet of importance.

Following the "Introduction," which gives an accurate account of the Irwell and tributaries, and an explanation of the principal items of the analyses, we find a chapter devoted to the Irwell at Throstle-Nest Weir, which is further summarised on page 22. Then comes the Irwell up stream as Chapter III., in which we find full analyses of the Medlock, Irk, Roeh, Croal, Tottington Brook, Holecombe Brook, River Ogden, Linney Water, and Whitewell Brook, taken at their points of confluence with the Irwell. The effect of drought is plainly set out in Chapter IV., a chapter full of interest to sanitarians, especially as regards the table on page 44, where the various tributaries at their points of confluence with the Irwell are compared with the Leeds and Salford sewage. The Tributaries in Flood form the subject-matter of Chapter V., while Chapter VI. is devoted to an analysis of the source of each tributary. Pages 66, 67, and 68 contain three tables, setting forth a comparison of the sources of the various tributaries, with their normal flow at point of confluence with the main stream, showing thereby the extent of the pollution which each has suffered during its flow. Chapter VII. deals with the various pollutions, while Chapter VIII. describes with minuteness the methods that have been followed in performing the analyses recorded in the work. It is something for the Manchester local authorities and manufacturers to know that the drought flow of the Irk, Medlock and Tottington Brook is excelled in purity by the Leeds sewage.

**ELECTRO-CHEMICAL ANALYSIS.** By EDGAR F. SMITH, Professor of Analytical Chemistry, University of Pennsylvania. Philadelphia: P. Blakiston, Son, and Co., 1012, Walnut Street. London: H. Grevel and Co., 33, King Street, Covent Garden. 1890.

SMALL 12mo. volume, bound in cloth, and containing Preface, Table of Contents, List of Abbreviations, and subject-matter covering 113 pages, terminating with an alphabetical index. The text is embellished with 25 woodcuts. Price one dollar.

The work is subdivided as follows:—INTRODUCTION. ACTION OF THE ELECTRIC CURRENT UPON ACIDS AND SALTS. OHM, VOLT, AMPÈRE. SOURCES OF ELECTRIC CURRENT:—Grenet battery, Leclanché cell, Daniel cell, Meidinger cell, Crowfoot cell, Bunsen and Grove batteries, Magneto-electric machines, Storage cells, Thermopile. REDUCTION OF THE CURRENT:—Rheostat, Resistance frame. MEASURING CURRENTS:—Voltmeter, Ampèremeter. HISTORICAL SKETCH. SPECIAL PART:—1. Determination of the Metals. 2. Separation of the Metals. 3. Oxidations by means of the Electric Current.

**DR. J. FRICK'S PHYSIKALISCHE TECHNIK.** Specieil Anleitung zur Ausführung Physikalischer Demonstrationen und zur Herstellung von Physikalischen Demonstrations-Apparaten mit möglichst einfachen Mitteln. Sechste umgearbeitete und vermehrte Auflage, von DR. OTTO LEHMANN, Professor der Physik an der technischen Hochschule in Karlsruhe. In zwei Bänden: Erster Band. Braunschweig. Druck und Verlag von Friedrich Vieweg und Sohn. London: H. Grevel & Co., 33, King Street, Covent Garden. 1890.

OCTAVO volume, in paper cover, price 15s., containing Preface, Table of Contents, and Text covering 725 pages. The text is illustrated with 708 excellent engravings. The purpose of the book as stated in the preface is, "In the first place, to serve as a Guide in the carrying out of Experiments in Physics, and to supply details, attention to which will ensure their success, as well as to discuss the construction and treatment of apparatus; and, in the second place, general direction and methods will be given for preparing most of the apparatus in the cheapest and most efficient manner." The work is complete in two volumes, and hence the alphabetical index will only appear in the second volume, yet in the press.

The first part is devoted to the treatment of apparatus in general, and may be regarded as a treatise on the carrying out of some of the more frequently occurring pieces of work in the physical laboratory.

Chap. I.—On the arrangement of the department and treatment of apparatus generally. Chap. II.—The cleaning, repairing, and setting up of apparatus.

The second part is a treatise on individual experiments in physics.

Chap. I.—Experiments on the equilibrium of forces—A. Solid bodies. B. Liquids. C. Gases. Chap. II.—Experiments on heat—A. The change of state of bodies by heat. Chap. III.—Experiments in dynamics and thermo-dynamics—A. The movements of solid bodies. B. The movements of liquids. C. The movements of gases. D. Experiments in conduction of heat.

**Die FARBENREACTIONEN DER KOHLENSTOFFVERBINDUNGEN.** Für Chemische, Physiologische, Mikrochemische, Botanische, Medicinische, und Pharmakologische Untersuchungen, bearbeitet von Dr. EMIL NICKEL. Zweite umgearbeitete, vermehrte und erweiterte Auflage. 1890. Berlin: W. Verlag von Hermann Peters. London: H. Grevel and Co., 33, King Street, Covent Garden.

SMALL 8vo. volume, in paper cover, and a considerable enlargement of the first edition. It contains Prefaces to the first and second editions, Table of Contents, Text covering 121 pages, and an Alphabetical Index of Subject-Matter and Authors. The subject is divided for treatment as follows:—

PART I.—Colour reactions with aromatic substances. Chap. I.—Colour reactions with nitrous acid and its derivatives, with exclusion of azo colour formation. Chap. II.—Colour reactions with nitric acid. Chap. III.—Colour reactions with formation of azo colours. Chap. IV.—Colour reactions with formation of triphenyl-methane colours and analogous compounds. Chap. V.—Colour reactions with the aid of iron salts and chromic acid salts. Chap. VI.—Conclusions derived from PART I.

PART II.—Colour reactions without participation of substances containing the benzene nucleus, and colour reactions of unknown character. Chap. VII.—Colour reactions with members of the cyanogen group. Chap. VIII.—Colour reactions with formation of Mnreoxide and similar colouring matters. Chap. IX.—Colour reactions with formation of colours of inorganic character.

## Trade Report.

### TARIFF CHANGES AND CUSTOMS REGULATIONS.

(From the Board of Trade Journal.)

#### THE NETHERLANDS.

##### Classification of Articles in Customs Tariff.

According to the *Moniteur Belge* for the 17th July, the following decisions affecting the classification of articles in the Netherlands Customs tariff have recently been given by the Customs authorities of that country:—

Carbolic acid and creosote, raw or refined, are taxed as oils not specially distinguished, at the rate of 0.55 fl. per 100 kilos. Products known under the name of carbolinum, guaiacol, ereosol, creoline, and creoline powders are liable to the same duty.

#### UNITED STATES.

##### Recent Customs Decisions.

The following decisions affecting the classification of articles in the Customs tariff, and the application of the Customs law of the United States, have recently been given by the United States Customs authorities:—

A weak solution of peroxide of hydrogen in water not similar to bleaching powder either in material or quality, being similar to such powder only as to some uses to which it may be applied, is dutiable as prescribed by paragraph 92 for chemical compounds not otherwise provided for at the rate of 25 per cent. *ad valorem*.

Certain alizarine gray is dutiable at the rate of 35 per cent. *ad valorem*, under the provision in T.L., 82, for "all coal tar colours or dyes, by whatever name known."

#### CAPE OF GOOD HOPE.

##### Modification of Customs Regulations.

By virtue of section 5 of the Customs Union Tariff Act of 1889, the Governor has directed it to be notified that from and after 8th August last rebate to the extent shown in the schedule below, shall be granted in respect to such articles as are in the said schedule whenever any such articles shall be removed overland to British Bechuanaland and the South African Republic, provided that such articles shall only be removed in accordance with such regulations as the Governor may from time to time prescribe.

#### SCHEDULE.

Articles.	Rebate to be Granted.	Duty less Rebate to be Paid.
Brass in bars, for every 100l. in value .....	£ s. d. 12 0 0	£ s. d. Nil.
Candles, per lb. ....	0 0 1½	0 0 1
Copper, per 100l. in value .....	12 0 0	Nil.
Dynamite, per lb. ....	0 0 2	0 0 1
India-rubber, unmanufactured, per 100l. in value .....	12 0 0	Nil.
Oil, paraffin and linseed, per Imp. gall. ....	0 0 9	0 0 3
Oil, other than preceding (not chemical, essential, or perfumed), for every Imp. gall. ....	0 0 6	0 0 6
Do. (chemical, essential, or perfumed), for every 100l. in value .....	10 0 0	5 0 0
Rosin, for every 100l. in value .....	5 0 0	5 0 0
Salt, per ton of 2,000 lb. ....	0 3 6	0 1 6

#### SCHEDULE—cont.

Articles.	Rebate to be Granted.	Duty less Rebate to be Paid.
Soap, per 100 lb. ....	£ s. d. 0 3 5	£ s. d. 0 0 9
Soda, caustic, for every 100l. in value .....	5 0 0	5 0 0
Turmeric, per 100 lb. ....	1 4 0	0 1 0
Turpentine, per Imp. gall. ....	0 0 10	0 0 2
Varnish, per Imp. gall. ....	0 0 6	0 0 6
Vinegar, per Imp. gall. ....	0 0	0 0 1

#### GOLD COAST.

##### Customs Tariff.

The following is a statement of some of the rates of duty now levied on articles imported into the Gold Coast:—

The undermentioned pay 10 per cent. *ad valorem*:—Kerosene and other illuminating oils, perfumery, soap, sugar, and all other goods not enumerated and unexempted.

The following are admitted free:—Acids, blue indigo, candles, chemicals, drugs and medicines, filters, india rubber, scientific instruments, matches, oils (except kerosene and other illuminating oils), paints, pitch and tar, quicksilver, salts, spirits (methylated, unfit for drinking), and tallow.

### EXTRACTS FROM DIPLOMATIC AND CONSULAR REPORTS.

#### EGYPT.

##### Drugs, &c., Imported.

The following gives an idea of the business which is done with Egypt through Alexandria in a few of the staples of our trades:—

Articles.	1889.		1888.	
	Quantity.	Value.	Quantity.	Value.
Olive oil.....	..	£ 105,830	..	£ 83,890
Perfumery and chemical products .....	..	98,082	..	89,722
Soap .....	Lbs. 7,320,992	70,313	6,817,729	72,179
Colours and dyes other than indigo .....	..	61,102	..	43,396
Spices .....	..	56,483	..	44,392

#### Gum Arabic.

The exports of this once-important article are now almost nil, having steadily declined since 1884, when the total exports amounted to 131,000l. Large quantities of gum arabic, ivory, and other valuable products are rotting away in the Soudan, where there is no possibility of exporting or properly storing them.

#### Petroleum.

There was a considerable increase in the imports of petroleum, the total value amounting to 181,291l., against 118,236l. in 1888. The market was, however, overstocked. The use of petroleum has gradually spread throughout Egypt, and has recently been adopted by the Arabs as far south as Wady Halfa, where it was previously almost unknown. Russian oil, on account of its cheapness, has now almost driven American oil out of the market, and, owing to the higher price of the latter, there are instances of the sale of Russian oil as American, the trade-marks being imitated.

## GERMANY.

*Chemicals.*

Our Consul at Frankfurt-on-Main reports that business in chemicals showed a decided improvement in the latter half of 1889. The chemical industry was but little affected by foreign competition, and according to the report of the Chamber of Commerce, some German chemicals have even got the start of English makers, although certain English chemicals still rule the market, and will maintain their position for a long time yet. The trade in pharmaceutical preparations was very satisfactory in the last quarter of the year, the demand having increased, and the profit was fair. In quinine most sales were made by agents, who successfully competed with the manufactories. During the influenza epidemic this article and similar preparations, such as antipyrin, phenacetine, antifebrin, &c., were consumed to such an enormous extent that the manufacturers and dealers were unable to satisfy the demands.

*Cocoa Butter.*

The manufacture of cocoa-butter at Mannheim was less remunerative in 1889 than usual, owing to a deficiency of raw material; nevertheless, the industry may be said to be prospering, having reached a production of 6,000 lb. a day.

## ITALY.

*Lemon Juice.*

The Palermo trade in lemon juice has increased by 5,312*l.*, there having been an augmented demand from France and Great Britain, the two principal markets for this product. France took 4,800*l.*, against 1,186*l.* in 1888. Great Britain raised her demand of 2,115*l.* in 1886, to 3,212*l.* in 1889.

*Sulphur.*

The Consul at Catania reports that the exportation of sulphur underwent a decrease of 9,133 tons as compared with 1888, but the demand became active about the latter part of December for shipment in January and February of the present year. Prices are improving. Sulphur is looked upon as the most important and conspicuous of Sicilian products, and forms the essential resource of the island, being exported to all parts of the world. A new system of fusing, requiring no heavy expenses of plant and no skilled assistance, has lately been introduced by Señor Lion Gil y Ruiz, engineer of the Royal Corps of Engineers in Spain, who first introduced it three years ago in the mines of Buen Viento Corre, belonging to a company in Almeria (Spain), where, after several trials, it obtained a brilliant success. Señor Gil went to Sicily last autumn, seeking a larger field of operation for his invention, and, at his own expense, he planted one of his furnaces on the Mugha mine, where several experiments took place, resulting in a saving of 29 per cent. of sulphur as compared with the old mode of fusing through the Calchione. Referring to "an idea which has found its way that Sicilian sulphur is sooner or later doomed to be excluded from foreign markets," owing to the recovery of sulphur by the "Chance process," the Consul states that Sicilian sulphur, as regards quantities exported, has not suffered from it, and he adds that "Professor Travaglia, of the Mining School of Caltanissetta, a gentleman more than competent in the matter, attaches no importance to the Chance process for obtaining sulphur, and is confident that Sicilian production will still have its markets." Professor Travaglia might change his opinion if he took a summer holiday round about our alkali works in England.

## WOOD-PULP INDUSTRY OF SWEDEN.

Her Majesty's Consul at Stockholm, in his last report on the trade of that capital, says that the manufacture of wood-pulp as a natural product of the country has of late become one of the most important export trades of Sweden. At present there are somewhat more than 120 wood-pulp factories in Sweden, of which about half that number have been started during the last three years.

During 1889 alone 34 new factories have been established. The export from Stockholm compared with the export of last year shows an increase of 6,617 cwt., or about eight times the quantity then exported. The export of the whole of Sweden, which in 1872 only amounted to 114,000 cwt., amounted during 1889 to more than 988,000 cwt. The prices have been constantly declining, but new and improved methods in a like proportion lowered the working cost, and several factories have shown excellent results during latter years. A great number of the factories have been started at places where the iron manufacture has proved a failure. The present improvement in the latter trade, and the heavy increase in the price of charcoal, may, however, cause a decline in the wood-pulp trade.—(No. 772, *Foreign Office Annual Series*.)

## MISCELLANEOUS TRADE NOTICES.

## THE CHEMICAL UNION.

Nearly one hundred of the largest alkali makers were present at the meeting of the alkali trade, called at Liverpool, on Friday, 11th September, at the instance of the promoters of the Chemical Union.

The chairman opened the proceedings by giving a résumé of the labours of the committee from the time of the inception of the scheme to the present. A proof prospectus was handed to each member of the trade present, from which it was gathered that the capital of the Union is to be eight and a half millions sterling, of which seven millions is set against works, plant, land and buildings, and one and a half millions for stocks and working capital.

The capital of the Union will be allotted as follows:—

Two-and-a-half millions in 5 per cent. debentures.

Three millions in 7 per cent. preference shares.

Three millions in ordinary shares.

It is understood that the vendors have agreed to take 70 per cent. of the purchase money in shares, so that it is quite possible that the public may experience a difficulty in obtaining the allotment of any ordinary shares.

The chairman stated that the committee had come to the conclusion to issue the prospectus themselves, without the assistance of any financial intermediary. They were confident of success, and he mentioned that five firms, representing one and a half millions of capital, have stated their intention of taking the *whole* of the purchase money in shares, and it is understood to be the intention of one firm to apply for 100,000*l.* more shares than the amount of their purchase money.—*Chemical Trade Journal*.

## THE ALFA GRASS IN ALGERIA.

According to recent statistics the yearly production of alfa grass is about ten million francs, a sum which could easily be doubled or trebled. But owing to insufficient means of transportation, there are in the Algiers department alone over 15,000 acres of grass running to waste, a loss of 120,000 tons, and as much as 400,000 tons are estimated to be, for the same reason, lost in other parts of the colony. As it is, England purchases nearly four-fifths of the crop, and uses it almost entirely for special qualities of paper. Of the latter paper thus made much is exported to France. The printing paper is especially adapted to fine work and plate-printing. To the use of alfa-grass paper the superiority of English illustrated periodicals is partly to be attributed—if their French competitors are to be believed.—*Chemist and Druggist*.

## MINERAL RESOURCES OF URUGUAY.

The *South American Journal* for the 9th August contains the following information extracted from the *Anuario Estadístico* of the Republic of Uruguay for the year 1888 on the subject of the mineral resources of that State:—

This soil is very rich in minerals, metals, clay, and combustible minerals. Gold, silver, copper, iron, tin, and mercury, are found among the first. Granite, mica, felspar,

various and precious agates, calcareous stone, mountain-rock crystal, marble of different colours, slate, lithographic stones, alum, gypsum, cobalt, calcareous cement, lodestone, marble basalt, and columbite of great value, discovered by Mr. Lettson. A quantity of flints and crystallisations similar to rubies, topaz, zirconite, and emerald, which appear in glittering points in pyramidal shapes, are found in abundance in our mineral kingdom. The mineralogist, Henry Petivenit, found gold, topaz, and diamonds, in the River San Francisco, which runs through Minas, and Mr. Lettson, gold in the departments of Salto and Tacuarembó.

From 1852 up to date, several mines were denounced, and samples of minerals were extracted from Godoy, Barriga Negra, San Francisco de Minas, Arapey, Chico, Aeguaí, and other places.

The working of a lead mine was tried in Soldado, department of Minas, and at present a French company works the gold mines of Cuñapirú in the auriferous region of the department of Tacuarembó. Another company works a copper mine in the department of Maldonado.

In the hills of Arequita, Penitentes, Campanero, Mahoma, and Marincho, since last century the existence of gold has been ascertained.

Gold in veins is found in quartz, and also in nuggets. By the source of the Arapey and Guequay rivers, and especially of the Catalan and Pintado, begins the region of the quartz stone and agates, amethysts, and glittering flints.

Black basalt is plentiful in the hills of Hades and in the vales of Cuareim and Artigas, and on the shores of Invernada. Calcareous stone is also very plentiful, especially in the department of Minas, Maldonado, Florida, and Isla-Mala. Marble is found in the two first departments. It is not rare to find calcareous cement on the left shore of the River Brújás and on the sides of the Cerro of Monte Video; the stones which were employed in the construction of the porticoes of the old city and Cabildo were extracted therefrom. Granite is predominant in these deposits. The felspar in the samples extracted from the hills of San José, in Estanzuela, and near the Aguada, is of pale white or alabastrous, rose or darkish yellow colours. Mica is generally black or dark yellow.

#### THE PETROLEUM DEPOSITS OF PERU.

West from Greenwich 81 deg. 40 min., and south of the equator 3 deg. 40 min., is Zorritos, the centre of the petroleum region of Northern Peru. Lying as it does so near the equator, it would be supposed that the heat would be excessive, but this is not the case, for at a point a little further north commences the rainless region, and as a consequence, the trade winds. These strong breezes are in the morning, from 2 o'clock until 10, from the south-east, and blow strongly, changing at 12 o'clock to south-west. The result is a temperature that does not vary from 70 deg. Fahr. 5 deg. all the year round. The air is perfectly dry, and the climate much recommended for lung diseases.

Abundant surface indications of petroleum exist at and in the vicinity of Zorritos. Some years since a company was formed to develop the district, and they put down some four wells, the largest of which produced 16,000 gallons daily. A refinery was built, and the manufacture of kerosene began. Lack of capital prevented a large development, and it was not until the property came into the hands of the present owner, Mr. Faustino G. Piaggio, that a regular system of work was inaugurated, and it is now one of the best managed properties in South America. There are at present nine wells, and the production is 7,000 cases, or 70,000 gallons, of refined kerosene a month. The specific gravity of the petroleum is 42 deg., producing 22 per cent. of light products, 29 per cent. of illuminating oil, 16 per cent. lubricating, and 33 per cent. of asphalt or brea. The crude petroleum, as well as the distillates, has a sweet aromatic odour, and differs in this respect from many other oils. A very small quantity of acid is used in treating the kerosene, owing to the absence of paraffin, and 2 lb. to 3 lb. of acid is quite sufficient for the purification of 40 gallons of kerosene. The property lies on the shore of the Bay of Zorritos, consisting of 54 pertenencias, or

2,160,000 square metres, having an extension along the coast of 10,800 metres. All of this large tract has surface indications, while the geological formation is all that could be desired, the anticlinal being formed by the shore range of the Andes. The port is smooth and deep, a small railway connects all the offices and shops with the wharf, and the different departments are in communication with the principal office by telephone. No attempt has yet been made to introduce the residuum, after extracting the kerosene, as a combustible for steamers, &c. Good coal is never lower than 15 silver soles a ton, and it has been demonstrated beyond a doubt that three barrels, or 120 gallons, of oil is equivalent to a ton of the best coal. Not only would it seem that the steamers on this coast would use petroleum as a fuel, but the immense saltpetre districts of Arica, Tarapaca, and Antofagasta would be large consumers, once assuring them of a supply. Mr. Piaggio has not been able to extend this branch of the business, as it requires another capital and other direction to carry it through. Attention is being called to Northern Peru, and irrigating companies are being formed. Mines are being developed, and it looks as though a new era had commenced for that country.—*Engineering*.

#### THE PRODUCTION OF QUICKSILVER.

The following statistics of the production of quicksilver in the world have been published by the Statistical Department of the United States, the quantities being in flasks of about 76 lb. each:—

	United States.	Spain, Austria, and Italy.	Total.
1881	60,851	60,082	120,933
1883	46,725	68,394	115,119
1885	32,073	66,281	98,354
1887	33,760	75,027	108,787
1889	26,464	71,772	101,236

From these figures it will be seen that the production of America is diminishing both actually and proportionately, as 10 years ago it was about one-half, whereas now it is little over one-fourth of the whole.—*Industries*.

#### DISCOVERY OF QUICKSILVER.

A fresh rich deposit of quicksilver, at no greater depth than three feet under the surface, has recently been discovered at Mantche, near Wippach, in the Austrian province of Carniola.—*Standard*.

#### EXPORTS OF CHEMICALS FROM THE TYNE.

The following are the exports of chemicals from the Tyne for the month of August and corresponding period of previous year:—

	1890.	1889.
	Tons.	Tons.
Alkali and soda ash.....	1,538	1,102
Bicarbonate of soda.....	6	13
Bleaching powder.....	2,018	1,410
Manure.....	3,612	4,486
Soda crystals.....	990	733
Sulphate of soda.....	64	63
Caustic soda.....	810	2,546
Other chemicals.....	823	
	9,811	10,413

—*Chemical Trade Journal*.

## THE EXPORT OF CAMPHOR FROM JAPAN.

A great impetus was given to the export of camphor from Japan last year by the increase of prices in Europe, and fears were entertained that the supply was being exhausted. Even if this should be the case in some districts, and that it is perhaps so is shown by the fact that the export is being diverted from Nagasaki to Hiogo, new forests of camphor trees appear to become available in others to an extent which prevents any diminution in the aggregate export.—*Industries.*

## THE COTTON-OIL EXPORTS OF THE U.S.A.

The cotton-oil exports of the U.S.A. amounted to 13,384,385 gallons (value 5,291,178 dols.) during the year ending June 30th; that is, five times as much as was exported the year before. In 1875-76 the export was only 281,054 gallons.—*Chemist and Druggist.*

## THE CULTIVATION OF CASTOR OIL IN SENEGAL.

The French *Moniteur Officiel du Commerce* for August 14th publishes a report which the French Colonial Office has received from M. Carstaing, on the subject of the cultivation of the castor-oil plant in Senegal.

In June 1888 M. Carstaing drew the attention of the Governor of the French colony of Senegal to the advantages which would accrue from the cultivation of the indigenous castor-oil plant, particularly the species which spontaneously decorticates itself in the sun, and which M. Carstaing had discovered. The Government agreed to undertake experiments in this direction, and at the end of June 1888, 10,000 seeds were sown in a plantation at Sor, near St. Louis, and 6,500 were distributed to private persons to experiment upon, at the public cost.

The extreme dryness of the climate had prevented the success of several other attempts of a like kind, such as those with arachides nuts, millet, and beans. It was fortunately found that the castor-oil plant was able to resist this obstacle, and in its first season the plantation at Sor produced 300 kilos. of good seeds. In May 1889 other plantations of castor oil were founded at Dakar-Bango, Louga, Kelle, and Thiès. By the close of December 1889 the Government was able to announce that these new areas of cultivation had proved twice as successful as any one had dared to hope. The plants had in six months grown, at Thiès and elsewhere, to a height of from 3 to 4 metres, with a rich crop.

During the rainy season of the present year a great number of applications have been made by planters in various parts of Senegal for seed of the best quality, and fresh plantations have been opened, with 1889 seed, in Rufisque, Gorée, and other places. It has been so clearly shown that the soil and climate of Senegal are favourable to the growth of the castor-oil plant, that preparations are being now made for a very wide cultivation of it throughout the colony in February, March, and April of next year. The principal difficulty in the way of this new industrial enterprise is the resistance of the natives, who cannot be persuaded that arachides nuts and castor oil can be simultaneously grown in the same piece of ground. This is quite a mistake, however; by planting the castor-oil plants of the first year sufficiently sparsely, success is certain.

The principal trading houses of Bordeaux and Marseilles have promised to support the colony of Senegal in the cultivation of castor oil, by offering a market for the product. They consider that the new industry will prove a source of immense wealth to the colony, the present prices being highly remunerative. The accident that the crops of arachides nuts have lately been a failure make the Administration of Senegal only the more anxious to secure this new and valuable product. Offers have already reached Saint Louis from two American houses, and it is believed that the colony will have no difficulty in selling all that it produces.

The numerous uses to which castor oil is now put are very encouraging to the cultivation of the plant. It can be

utilised, besides in its medicinal capacity, in dyeing, soap-making, greasing machinery, lighting, printing, for cement in Java, and for food in China. The Chinese boil it with alum and sugar to remove the bitter taste.

## BOARD OF TRADE RETURNS

## SUMMARY OF IMPORTS.

	Month ended 31st August	
	1889.	1890.
Metals.....	£ 1,718,433	£ 1,863,582
Chemicals and dyestuffs.....	483,048	514,524
Oils.....	679,506	640,373
Raw materials for non-textile industries.....	4,985,328	4,137,800
Total value of all imports....	32,937,088	31,322,897

## SUMMARY OF EXPORTS.

	Month ended 31st August	
	1889.	1890.
Metals (other than machinery) ....	£ 3,329,443	£ 3,629,295
Chemicals and medicines .....	653,452	711,201
Miscellaneous articles.....	2,488,079	2,836,418
Total value of all exports.....	21,390,507	22,817,609

## IMPORTS OF METALS FOR MONTH ENDED 31ST AUGUST.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Copper:—			£	£
Ore..... Tons	10,513	4,613	89,628	34,333
Regulus .....	14,557	7,919	343,114	224,250
Unwrought ....	3,136	3,848	136,386	226,434
Iron and steel:—				
Iron ore.....	290,546	330,913	222,024	250,422
Iron bolt, bar, &c. ..	14,050	9,009	133,750	92,033
Steel, unwrought..	1,199	768	8,186	7,810
Lead, pig and sheet ..	11,580	12,629	147,437	164,081
Pyrites .....	37,082	52,770	65,296	99,836
Quicksilver..... Lb.	77,596	84,396	9,108	11,240
Tin..... Cwt.	28,822	45,548	128,115	214,577
Zinc..... Tons	5,077	4,733	94,484	108,090
Other articles ...Value £	..	..	340,005	420,576
Total value of metals	..	..	1,718,433	1,863,582

IMPORTS OF CHEMICALS AND DYESTUFFS FOR MONTH  
ENDED 31ST AUGUST.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Alkali..... Cwt.	1,396	1,491	£ 3,392	£ 3,786
Bark (for tanners, &c.) .....	35,978	71,773	15,154	33,190
Brimstone .....	68,120	16,434	18,058	4,842
Chemicals..... Value £	..	..	99,507	108,122
Cochineal .....	506	165	3,381	958
Cutch and gambier Tons	2,564	2,849	74,702	78,973
Dyes:—				
Aniline .....	..	..	25,802	24,029
Alizarine .....	..	..	25,753	25,732
Other .....	..	..	1,180	1,340
Indigo .....	744	1,063	8,789	12,482
Madder .....	1,275	456	1,424	573
Nitrate of soda....	75,920	59,675	32,419	24,490
Nitrate of potash ..	24,689	25,056	21,860	22,330
Valonia .....	2,408	2,604	35,136	52,110
Other articles... Value £	..	..	116,493	121,567
Total value of chemicals	..	..	483,048	514,524

## IMPORTS OF OILS FOR MONTH ENDED 31ST AUGUST.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Cocoa-nut..... Cwt.	13,513	12,680	£ 29,654	£ 17,630
Olive .....	2,155	1,349	76,209	54,093
Palm .....	86,185	49,292	88,545	57,578
Petroleum .....	9,196,189	11,023,713	256,460	258,245
Seed .....	1,270	1,670	35,994	43,333
Train, &c..... Tons	2,715	1,978	55,743	42,395
Turpentine .....	53,464	64,434	86,864	100,550
Other articles .. Value £	..	..	58,937	66,549
Total value of oils ...	..	..	679,506	640,373

IMPORTS OF RAW MATERIALS FOR NON-TEXTILE  
INDUSTRIES FOR MONTH ENDED 31ST AUGUST.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Bark, Peruvian .. Cwt.	4,336	5,034	£ 17,261	£ 15,661
Bristles..... Lb.	291,914	267,675	39,448	28,509
Caoutchouc..... Cwt.	13,207	12,296	125,966	135,160
Gum:—				
Arabie.....	4,091	4,383	14,554	19,557
Lac, &c.....	3,419	2,417	11,931	9,757

## IMPORTS OF RAW MATERIALS, &amp;c.—cont.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Gutta-percha ....	7,301	4,736	£ 106,173	£ 55,709
Hides, raw:—				
Dry.....	37,498	28,335	92,193	74,904
Wet.....	62,569	69,888	144,521	156,155
Ivory .....	996	694	46,375	38,045
Manures:—				
Guano .....	2,421	337	16,605	1,745
Bones.....	2,574	3,058	12,728	15,852
Paraffin..... Cwt.	21,435	43,312	24,989	56,198
Linen rags..... Tons	3,104	2,408	30,121	24,167
Esparto, &c. ....	16,074	9,834	79,843	52,468
Pulp of wood ....	19,497	11,384	63,492	66,378
Rosin..... Cwt.	93,481	134,747	17,335	33,113
Tallow and stearin ..	103,487	92,867	131,517	114,586
Tar .....	59,273	43,381	33,358	31,911
Wood:—				
Hewn .....	287,863	248,967	808,883	596,714
Sawn .....	909,715	745,662	2,261,512	1,752,316
Staves .....	18,596	17,854	62,386	59,019
Mahogany .....	2,200	2,479	21,149	20,825
Other articles.... Value £	..	..	822,898	788,101
Total value .....	..	..	4,985,328	4,137,860

Besides the above, drugs to the value of 90,995*l.* were imported as against 80,386*l.* in August 1889.

EXPORTS OF METALS (OTHER THAN MACHINERY) FOR  
MONTH ENDED 31ST AUGUST.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Brass..... Cwt.	9,882	7,746	£ 39,984	£ 37,623
Copper:—				
Unwrought .....	47,948	67,409	106,779	206,443
Wrought.....	33,389	18,557	100,099	67,055
Mixed metal ....	29,216	30,160	72,192	87,376
Hardware .....	..	..	220,984	212,098
Implements.....	..	..	108,093	100,009
Iron and steel..... Tons	383,337	345,557	2,459,212	2,613,662
Lead .....	2,944	5,924	41,870	83,474
Plated wares... Value £	..	..	31,782	30,967
Telegraph wires... ..	..	..	46,432	55,601
Tin .....	6,672	9,511	31,230	46,750
Zinc .....	4,421	12,530	3,945	12,252
Other articles .. Value £	..	..	66,821	75,985
Total value .....	..	..	3,929,443	3,629,295



EXPORTS OF DRUGS AND CHEMICALS FOR MONTH ENDED  
31ST AUGUST.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
			£	£
Alkali..... Cwt.	501,369	540,113	131,391	181,059
Bleaching materials „	112,937	150,748	40,318	42,681
Chemical manures. Tons	38,714	39,117	174,359	181,022
Medicines..... Value £	..	..	74,036	80,173
Other articles ... „	..	..	230,348	226,206
Total value .....	..	..	653,452	711,201

EXPORTS OF MISCELLANEOUS ARTICLES FOR MONTH  
ENDED 31ST AUGUST.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
			£	£
Gunpowder..... Lb.	606,600	1,078,700	14,212	30,129
Military stores.. Value £	..	..	83,067	102,129
Candles..... Lb.	1,231,000	1,166,800	21,557	22,480
Caoutchouc..... Value £	..	..	95,480	110,293
Cement..... Tons	38,404	51,308	74,504	106,031
Products of coal Value £	..	..	66,979	83,266
Earthenware ... „	..	..	173,853	155,972
Stoneware .....	..	..	12,037	12,099
Glass:—				
Plate..... Sq. Ft.	219,717	265,345	16,084	13,563
Flint..... Cwt.	8,402	9,743	19,488	21,103
Bottles..... „	75,251	66,020	35,295	32,281
Other kinds..... „	15,598	15,638	11,132	14,663
Leather:—				
Unwrought .... „	9,825	13,452	91,195	115,628
Wrought ..... Value £	..	..	34,122	30,281
Seed oil..... Tons	4,907	4,621	110,986	109,388
Floor cloth..... Sq. Yds.	1,218,300	1,284,300	60,824	59,098
Painters' materials Val. £	..	..	121,339	125,393
Paper..... Cwt.	74,146	64,720	126,684	114,543
Rags..... Tons	4,069	4,836	31,907	36,833
Soap..... Cwt.	33,616	38,084	36,333	38,556
Total value .....	..	..	2,188,679	2,836,418

## QUICKSILVER.

	IMPORTS.			
	1890.	1889.	1888.	1887.
	Bottles.	Bottles.	Bottles.	Bottles.
August .....	1,125	1,034	27	1,200
Jan.—Aug.....	55,693	55,790	64,662	56,831
	EXPORTS.			
August .....	5,327	5,376	3,366	6,244
Jan.—Aug.....	41,124	40,868	34,750	46,390
Average Price:	£ s. d.	£ s. d.	£ s. d.	£ s. d.
August .....	10 0 0	9 6 6	7 14 0	7 3 0

## Monthly Patent List.

\* The dates given are the dates of the Official Journals in which acceptances of the Complete Specifications are advertised. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the said dates.

I.—GENERAL PLANT, APPARATUS, AND  
MACHINERY.

## APPLICATIONS.

13,084. C. J. Cario. Improvements in pyrometers. Complete Specification. August 20.

13,127. T. Young. Improvements in valves for corrosive and other fluids. August 21.

13,284. J. Foster. Improvements in and relating to evaporating apparatus. August 23.

13,322. R. Harburn. Evaporating and distilling liquids, which he proposes to call Harburn's patent cyclone evaporator and distiller. August 25.

13,390. J. Batten. Improvements in or relating to regenerating furnaces. August 26.

13,930. H. E. Newton.—From W. J. Maedonagh, R. Rock, W. Wood, C. S. Wilkinson, J. Plummet, and R. B. Wilkinson. An improved appliance for injecting liquid and gaseous fuel into furnaces. Complete Specification. September 4.

14,051. H. Rouart. Improvements in sterilising water and other liquids and apparatus therefor. September 6.

14,059. H. E. Hansen. Improvements in apparatus for the complete evaporation of solutions. Complete Specification. September 6.

14,075. J. A. Orme. Improvements connected with refrigerating apparatus or machinery. September 8.

14,104. H. C. Shaw. Improvements in or relating to furnaces or apparatus for heating boilers, evaporating pans, and other structures. September 8.

14,146. W. Orr and P. S. Brown. Improvements in the construction of lead-lined metallic boxes, tanks, drums, and other vessels. September 9.

14,240. W. L. Wise.—From T. L. Stephanon, Mauritius. Improvements in furnaces. September 10.

- 14,244. W. Barnes and E. M. Clymer. Improved means for feeding coal to furnaces. September 10.  
 14,370. A. Dannenberg. See Class II.  
 14,380. L. Johnstone. See Class IX.

## COMPLETE SPECIFICATIONS ACCEPTED.\*

1889.

- 14,338. C. A. Sahlström and J. C. Bromfield. Furnaces for burning liquid fuel. September 17.  
 14,736. G. F. Redfern.—From W. P. Abell. Centrifugal machines. September 17.  
 16,295. E. Gregory. Apparatus for controlling the cold air supply to furnaces, &c. September 3.  
 16,520. J. Price. Apparatus for distilling and condensing solutions. September 10.  
 16,863. A. H. Hobson. Apparatus for making extracts, and treating soluble substances by steam pressure and heat. September 3.  
 16,997. A. Légé, C. A. Remané, and A. T. Job. Air and vacuum pumps. September 17.  
 17,018. W. K. Bruce. Vacuum pans. August 27.  
 18,324. W. W. Bengough. Apparatus for heating, evaporating, and condensing. September 10.  
 18,528. H. H. Lake.—From La Société Anonyme des Parfums Naturels de Cannes. Method and apparatus for the lixiviation of various materials. September 17.

1890.

8544. W. H. Johnson and C. C. Hutchinson. Filter presses. September 3.  
 10,172. H. von Bayer. Method and apparatus for producing cold. August 27.  
 11,246. J. Klein. Process and apparatus for cooling liquids by means of air. September 10.

## H.—FUEL, GAS, AND LIGHT.

## APPLICATIONS.

- 13,033. F. Lennard and E. W. Harding. Improvements in the manufacture and production of artificial fuel. Complete Specification. August 19.  
 13,332. H. Stevenson. An improved method of and apparatus for heating. August 25.  
 13,355. H. Macaulay. Improvements in means or apparatus for regulating the heat and combustion of fuel. August 25.  
 13,364. W. Fenton and W. Graham. An improved manufactured fuel. August 25.  
 13,382. W. Welch. Improvements in purifying coal and water-gas and other inflammable vapours, and apparatus therefor. August 26.  
 13,398. W. H. Nevill. Improvements in the treatment of coal tar and pitch. August 26.  
 13,477. M. Fromont. Improvements in the production of coke, and in apparatus employed therefor. August 27.  
 13,499. F. J. Jones. Improvements in the manufacture of coke, and in purifiers for use therein, and in the manufacture of gas. August 27.

13,574. B. H. Thwaite. Improvements in methods of producing combustible gases for heat and lighting purposes, and in apparatus therefor. Complete Specification. August 29.

13,767. J. Gridley. Improvements in and relating to the treatment of wood, rendering the same highly inflammable. Complete Specification. September 2.

13,962. F. S. Craig and W. J. Cowden. Improved patent fuel. September 5.

14,052. L. H. Armour. Improvements in means employed in the manufacture of hard coke, the distilling of shale, the washing of ores or other operations wherein carbonaceous matter is subjected to the action of heat. September 6.

14,109. J. A. Rossiter. Improved means for generating heat. September 8.

14,112. S. A. Johnson. Improvements in effecting the combustion of fuel, and prevention of smoke in steam boilers, also applicable for domestic fire-grates. September 8.

14,115. H. H. Lake.—From J. Lundström and Co., Sweden. Improvements in the manufacture of artificial fuel. September 8.

14,122. E. P. Jarrard. Improvements in or pertaining to lighting. September 9.

14,297. G. Webb. Improvements in carburettors. September 11.

14,298. G. Webb. A new or improved process and apparatus for supplying gas lighting in a portable form to railway, omnibus, and other vehicles, and for other purposes. September 11.

14,370. A. Dannenberg. Improvement in the methods of firing of periodically and continuously worked chamber and annular furnaces. Complete Specification. September 11.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

12,054. B. H. Thwaite. Method of distributing, heating, and lighting gas. September 3.

14,060. F. N. Mackay. Method and apparatus for separating tar and ammonia from gases. September 10.

17,140. A. A. Fritz. Apparatus for producing an inflammable gas by carburization of air. September 3.

17,232. N. M. Henderson. Apparatus for distilling mineral oils. September 10.

17,467. E. Lund. Improvements in night-lights and holders for same. September 17.

17,673. The Coltness Iron Co., Lim., and W. Young. See Class X.

17,778. H. H. Lake.—From G. W. Wilcox. Apparatus for preventing smoke and economising fuel. August 27.

19,572. W. Wood. Apparatus for the manufacture of illuminating or heating gases. September 17.

1890.

2341. F. Scudder and H. G. Colman. Manufacture or treatment of gases containing carbonic oxide to render them odorous. September 3.

7040. J. J. Cooper. Improvements in carburettors. September 17.

### III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

#### APPLICATIONS.

13,398. W. H. Nevill. *See Class II.*

### IV.—COLOURING MATTERS AND DYES.

#### APPLICATIONS.

12,962. B. Willeox.—From The Farbenfabriken vormals F. Bayer and Co., Germany. Improvements in the manufacture and production of nitroso combinations, and of colouring matters therefrom. August 18.

13,010. W. Majert. Improvements in the manufacture of colouring matters. August 19.

13,165. J. R. Geigy. Production of mono and disulpho acids of tetramethyldiamidodiphenylmethane. August 21.

13,235. B. Willeox.—From The Farbenfabriken vormals F. Bayer and Co., Germany. Improvements in the manufacture and production of azo dyes. August 22.

13,443. B. Willeox.—From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture and production of new sulpho-acids of dioxy-naphthaline or amidonaphthol and of azo colouring matters therefrom. August 26.

13,516. R. Holliday and Sons, Limited, and T. Holliday. The production of a new sulpho acid of naphthylamine. August 28.

13,565. H. H. Lake.—From Wirth and Co., agents of A. Leonhardt and Co., Germany. Improvements in the manufacture of colouring matters. August 28.

13,627. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik, Germany. The manufacture and production of a new indigo blue and sulpho acids thereof. August 29.

13,710. J. Dawson and R. Hirsch. A process for the production of substituted benzidines (diamidotriphenyls) and colours therefrom. September 1.

14,416. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik, Germany. The manufacture and production of new amido-azo compounds and azo dyes therefrom. September 12.

14,432. J. Dawson and R. Hirsch. A process for the production of a substantive cotton colour. September 13.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

15,450. B. Nicholson and T. Palmer. *See Class XIV.*

17,024. O. Imray.—From The Farbwerke Griesheim-on-Main, W. Noetzel and Co. Manufacture of indulin-like colouring matters. September 17.

17,223. H. H. Lake.—From Wirth and Co., agents for A. Leonhardt and Co. Manufacture of colouring matter. September 3.

17,957. O. Imray.—From The Actiengesellschaft für Anilin Fabrikation. Preparation of orange-yellow colouring matters. September 17.

17,971. H. H. Lake.—From A. Leonhardt and Co. Improvements relating to colouring matters. August 27.

1890.

8541. J. J. Hart. Development or treatment of colouring matters in textile fabrics. September 3.

### V.—TEXTILES, COTTON, WOOL, SILK, Etc.

#### APPLICATIONS.

13,701. H. H. Lake.—From Eesales and Hatry, Germany. Improvements in and relating to the weaving of silk waste. August 30.

13,756. A. H. Norman. A cheap method of obtaining fibres from rami, esparto, or similar grasses. September 2.

13,965. K. T. Sutherland and G. Esdaile. Improved treatment of long-staple stalk-fibres, as China-grass, flax, and such like fibres, by which they will have a more even staple when artificially shortened. September 5.

13,997. S. Pitt.—From L. Vignon, France, and L. Cassella and Co., Germany. Improvements in animalising cotton and other vegetable fibres. September 5.

14,246. D. Gantillon. A method or process of treating silk or mixed silk fabrics to give them the appearance of China crepe. September 10.

14,389. A. Watson. An improved process whereby lace curtains and similar articles are treated. September 12.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

16,708. C. Baswitz. Treating textiles by ammoniac copper to make them waterproof and nonflammable. August 27.

1890.

12,689. K. Trobach and G. J. Bruch. *See Class VI.*

### VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

#### APPLICATIONS.

12,907. E. Sykes, D. Sykes, E. Heppenstall, T. Greenwood, and J. W. Whiteley. Improvements in machinery for scouring, dyeing, and drying hanks of yarn, slivers of fibre, and slubbings. August 18.

12,950. H. Thies and E. Herzog. Improvements in bleaching. Complete Specification. August 18.

13,048. S. Pitt.—From A. Smith and Sons' Carpet Co., United States. Certain new and useful improvements in dyeing apparatus. Complete Specification. August 19.

13,184. I. F. Peck. Improvements in apparatus for dyeing yarn. August 21.

13,861. G. Jagenburg. Improved means and apparatus connected with washing, mordanting, or dyeing spun and unspun textile fibres. September 3.

13,963. J. H. Gartside and W. Warr. Improvements in dyeing wool, cotton, silk, or jute, or other fibre, or a mixture of wool and cotton black. September 5.

14,302. T. Ingham. Improvements in dyeing cotton. September 11.

14,372. J. Robertshaw. Improvements in dyeing, sizing, and washing machines. September 12.

14,399. E. T. Aucher. A process for dyeing raw cotton aniline black. September 12.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

19,490. L. Schreiner. Dyeing aniline black. August 27.

1890.

8541. J. J. Hart. See Class IV.

11,873. H. H. Lake. — From The L. Weldon Dyeing Machine Co. Yarn-dyeing machines. September 3.

12,689. K. Trobach and G. J. Bruch. Obtaining and bleaching vegetable fibres. September 17.

## VII.—ACIDS, ALKALIS, AND SALTS.

## APPLICATIONS.

13,240. L. M. C. Folie-Desjardins. Process and apparatus for the simultaneous manufacture of phosphorus and of alkaline silicates by the treatment of the mineral phosphates of lime, alumina, or bone ash. August 22.

13,312. F. Ellershausen. Improvements in the manufacture of sulphide of sodium. August 23.

13,373. F. Ellershausen. Improvements in the manufacture of caustic soda. August 25.

13,504. T. J. Hutchinson. Improvements in the method of obtaining useful products from bleaching powder dregs, and lime mud from paper works. August 28.

13,863. B. E. R. Newlands. Improvements in the treatment of phosphoric acid for facilitating its storage and transport. September 3.

13,871. J. E. Bennett. Improvements in furnaces and flues for evaporation pans used in the manufacture of salt. September 4.

13,929. F. Ellershausen. Improvements in the production of sulphite and hyposulphite of soda. September 4.

14,022. C. Négrier. Improvements in furnaces for concentrating sulphuric acid. Complete Specification. Filed September 6. Date applied for April 26, 1890, being date of application in France.

14,084. E. Meyer. Improvements in the manufacture of hydrate of alumina from silicates of alumina or clay. September 8.

14,483. T. Raynaud. Improvements relating to the production of soda and chlorine. September 13.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

17,074. T. Turner. Treatment of waste pickle from galvanising works, and other such acid liquors, and apparatus therefor. August 27.

17,395. W. Garroway. Obtaining caustic soda or potash by means of boracic acid. September 3.

1890.

11,699. G. Nahnsen. See Class XI.

12,641. C. T. J. Vautin. Producing chlorine gas for the chlorination of gold or other ores. September 17.

## VIII.—GLASS, POTTERY, AND EARTHENWARE.

## APPLICATIONS.

13,134. C. D. Abel. — From the firm of J. Meurin, Germany. An improved manufacture of bricks, slabs, pipes, pipe stones, and other similar articles. August 21.

13,155. L. H. Jahn. Improvements in or connected with the firing of pottery. August 21.

13,222. S. Sambrook. Improvements in enamelling, hardening on, and fret kilns used by earthenware and china manufacturers; also partly applicable in the construction of bakers' and confectioners' ovens. Complete Specification. August 22.

13,282. J. Kirkman. Improvements in the construction of kilns or ovens for burning or baking pottery bricks, tiles, and the like. August 23.

13,331. J. Hamblet. Improvements in and apparatus for the manufacture of ornamental flooring tiles, and other tiles. August 25.

13,374. J. J. Barclay and J. Meikle. Improvements in kilns and apparatus for drying and burning bricks, pottery, &amp;c. Complete Specification. August 25.

13,436. F. C. Roberts. Improvements in pottery kilns. Complete Specification. August 26.

13,707. J. Gill. An improved method for the making of roofing tiles and flooring tiles. September 1.

14,211. D. Rylands and H. F. Boughton. Improvements in the manufacture of glass, and in furnaces for the melting thereof. September 10.

14,328. H. Bonnycastle and T. M. Rymer-Jones. An improvement in the manufacture of refractory and non-conducting bricks, blocks, tiles, slabs, and pipes. September 11.

14,359. L. Fargue. Improvements in the manufacture of stained or ceramic transparent glass windows. September 11.

14,360. C. O. Weber and G. F. Freeman. Improvements in the manufacture of artificial stones for lithographie and similar printing purposes. September 11.

14,383. C. E. Hawley. Improvements in the manufacture of chamber and other ware. September 12.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

16,326. E. Smith. Apparatus for making earthenware socket pipes. September 3.

16,475. W. B. Fitch. Manufacture of metal-coated glass and porcelain ware. August 27.

16,843. A. Drummond. Apparatus for rolling out glass sheets with surface designs. September 3.

17,565. J. G. Sowerby. Manufacture of antique and other sheet glass, and apparatus therefor. September 10.

1890.

11,560. A. Schaaf. Drying stoves for ceramic materials. September 3.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

### APPLICATIONS.

13,105. A. McLean. An improved decorative artificial stone. August 20.

13,216. J. Grainger. A new concrete ventilated building block for building purposes. August 22.

13,227. C. R. Bonné.—From M. Henry, France. Improvements in the manufacture of cement, such as Portland or slag cement. August 22.

13,331. W. S. Lea, J. C. Darby, and R. M. G. Dill. An improved cement or cements, specially suitable for resisting acids, alkalis, combinations thereof, and the manufacture of the same. August 25.

13,384. J. Juogblutb. Improved composition or concrete flooring for stables, public baths, slaughter-houses, and the like. Complete Specification. August 26.

13,595. H. P. Williams and E. G. Williams, trading as Pilkington and Co. An improved artificial asphalt. August 29.

13,620. J. Brown, J. W. Virgo, and H. F. Ainly. An improved composition to be used in the mixing of mortars, cements, and plasters, for building and other purposes. August 29.

14,380. L. Johnstone. Improvements in or connected with rotative furnaces or kilns for treating and recovering waste gases from raw or partially raw materials, used more especially and for example in the manufacture of cement. September 12.

14,398. C. von Forell. A process for manufacturing from Roman cement a composition similar to Portland cement. September 12.

14,439. J. Clemenson and T. Clemenson. An improved method or process of manufacturing Portland cement, and improvements in apparatus employed therein. September 13.

### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

15,406. W. J. Oswald, J. R. Ashwell, and J. A. Cooke. Manufacture of plaster of Paris, and utilisation of waste materials therefor. September 3.

17,758. H. A. Bassett. Manufacture of building material and of a composition for use therewith. September 17.

1890.

6711. F. C. Goodall. Marine cement. September 3.

8606. S. G. T. C. Bryan. Treating slag for manufacture of building blocks, paving blocks, &c. September 17.

10,002. T. Breakell and J. W. Breakell. Colouring and polishing calcareous and other stones, and producing variegated effects thereon. September 10.

## X.—METALLURGY, MINING, ETC.

### APPLICATIONS.

12,912. G. Rodger. Improvements in or connected with apparatus for making steel. August 18.

12,938. S. O. Cowper-Coles. Improvements in coating wires with the heavy metals and alloys of the same. August 18.

13,137. G. W. Clark, J. Clark, and W. G. Dickson. Improvements in obtaining aluminium chloride, and obtaining aluminium therefrom. August 21.

13,174. J. H. Lancaster and M. R. Conley. An improved process for the manufacture of steel, and apparatus therefor. August 21.

13,259. C. T. J. Vautin. Improvements in apparatus for the extraction of gold. Complete Specification. August 22.

13,356. H. Y. Castner. Improvements in the manufacture of sodium and potassium. August 25.

13,393. S. O. Cowper-Coles. Improvements in the deposition of alloys. August 26.

13,413. T. Held. A process for obtaining a new metallic alloy. August 26.

13,474. W. L. Wise.—From L. Benchemans, Belgium. Improved process for roasting ores and metallic compounds. August 27.

13,486. A. Schneller and A. Astfalie. Improved process for the production of pure aluminium. August 27.

13,723. F. H. Molesworth. Improvements in the process of treating metalliferous ores. Complete Specification. September 1.

13,811. J. J. Knight and J. W. Bottomley. Improvements in the treatment of substances containing phosphorus and aluminium. September 3.

13,856. F. Burger and R. Mannheimer. A new product or compound for preventing the formation of pores in molten metals, and the manufacture of the same. September 3.

14,053. F. A. Herbertz. Improvements in smelting furnaces. Complete Specification. September 6.

14,063. H. H. Lake.—From C. F. Claus, Germany. Improvements in and relating to the annealing of wire, sheet metal, and other metal articles. September 6.

14,445. C. While. Melting and mixing iron. September 13.

### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

13,301. W. Deighton. Improvements in steel-making and other "water-gas" furnaces. 27 August.

14,462. A. J. Campion and J. E. Tenison-Woods. Treating tinned or galvanised scrap iron. September 10.

14,880. W. R. Lake.—From E. Thomson. Welding, shaping, and otherwise working metals. September 3.

15,970. W. C. Taylor and T. N. Kirkham. Treating gold quartz and other minerals to facilitate their reduction to powder. September 10.

16,318. H. Marbeau. Manufacture of alloys of nickel with other metals. August 27.

16,338. F. E. Ross. Improvements in cupola and blast furnaces. August 27.

16,868. E. A. Cowper. Recovering float gold. September 10.

17,673. The Coltness Iron Co., Limited, and W. Young. Treating gases from blast furnaces, gas producers, and coke ovens, and apparatus therefor. September 10.

18,062. H. Y. Castner. Purifying the anhydrous double chloride compounds of aluminium. September 17.

19,588. C. A. Faure. Manufacture of ferro-aluminium. September 10.

20,395. T. Brady, T. C. Powers, M. J. Quinn, and A. J. Maher. Apparatus for casting ingots. September 17.

1890.

8606. S. G. T. C. Bryan. See Class IX.

8992. H. W. Lash and J. Johnson. Reduction of ores. August 27.

12,641. C. T. J. Vautin. See Class VII.

## XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

### APPLICATIONS.

13,003. D. Pepper, jun. Improvements in the manufacture of electrodes for primary and secondary batteries. Complete Specification. August 19.

13,006. H. H. Lake.—From E. Thomson, United States. Improvements in welding or otherwise working metals by electricity, and in apparatus therefor. Complete Specification. August 19.

13,013. D. Pepper, jun. Improvements in the manufacture of electrodes for primary and secondary batteries. Complete Specification. August 19.

13,024. G. O. Rennerfelt. Method of and apparatus for producing, by means of electrolysis, decomposed products of melted haloid salts and other combinations of metals. Complete Specification. August 19.

13,261. W. J. S. Barber-Starkey. Improvements in and connected with primary galvanic batteries. August 23.

13,344. H. J. Allison.—From P. Hathaway, United States. Improvements in galvanic batteries. Complete Specification. August 25.

13,421. W. P. Thompson.—From C. L. Coffin, United States. Improvements in apparatus for working or welding metals electrically, and in the method of effecting the same. August 26.

13,463. J. Mosely. Improvements in and connected with zincs for galvanic batteries. August 27.

13,557. T. S. E. Dixon. Improvements in secondary batteries. Complete Specification. Filed August 28. Date applied for, February 6, 1890, being date of application in United States.

13,635. W. P. Thompson.—From C. L. Coffin, United States. Improvements in or appertaining to electric welding. Complete Specification. August 30.

13,636. W. P. Thompson.—From C. L. Coffin, United States. Improvements in electric welding. Complete Specification. August 30.

13,735. C. Hocpfner. Improvements in apparatus for use in electro-metallurgical operations. September 1.

13,807. C. M. Reed. An improved battery compound. Complete Specification. September 2.

13,853. C. A. Faure. Improvements in the production of electricity, and in means or apparatus therefor. September 3.

13,905. M. W. Dewey. Improvements in and relating to the welding of metals by electricity. Complete Specification. September 4.

13,906. M. W. Dewey. Improvements in and relating to the welding and brazing of metals by electricity. Complete Specification. September 4.

14,411. J. T. Niblett. Improvements in secondary batteries. September 12.

### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

11,962. P. Naef. Manufacture of peroxide of lead and by-products. September 3.

15,700. W. B. Sayers. Dynamo machines for electroplating. September 3.

1890.

8086. M. Bailey and J. Warnes. Secondary batteries. September 3.

10,123. M. W. Dewey. Welding, soldering, and otherwise working metals by electricity. September 10.

10,124. M. W. Dewey. Electric heating apparatus. September 10.

11,533. M. G. Farmer. Electrolytic apparatus for forming copper ingots. September 10.

11,680. M. G. Farmer. Apparatus for producing sheets of metal by electro-deposition. September 3.

11,699. G. Nahusen. Obtaining chlorine and bromine by aid of electricity. September 17.

## XII.—FATS, OILS, AND SOAP MANUFACTURE.

### APPLICATIONS.

13,634. C. A. Feuerlein and J. C. Lahusen. Improvements in or appertaining to the production of odourless wool grease. August 30.

13,803. C. A. Jensen.—From P. Moller, Germany. Improvements in the manufacture of cod-liver oil. Complete Specification. September 2.

13,891. D. Rossi. An improved paste or soap for removing grease. September 4.

13,981. G. P. Clarke.—From D. R. S. Galbraith and T. Bell, New Zealand. Improvements in the preparation of soap. September 5.

13,985. G. P. Clarke.—From D. R. S. Galbraith and T. Bell, New Zealand. Improvements in soap powders. Complete Specification. September 5.

14,239. J. Pettigrew. A new composition for cleaning painted or varnished work. September 10.

### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

14,364. E. Mansfield. Method and apparatus for extracting oleaginous and other matters from fibrous spun textiles, &c. September 10.

17,163. W. Brinck. Lubricants. September 3.

18,628. L. T. Thorne and Brin's Oxygen Co., Lim. Oxidation and thickening of oils for use in the manufacture of paints, varnishes, &c. September 10.

## XIII.—PAINTS, PIGMENTS, VARNISHES, AND RESINS.

### APPLICATIONS.

13,294. A. F. Bilderbeek-Gomess. Improved process of devulcanising vulcanised india-rubber, gutta-percha, and similar gums, and reclaiming the rubber therefrom for manufacture without the addition of pure rubber. August 23.

13,719. E. Schmal. Medium for using earth and chemical colours. Complete Specification. September 1.

13,769. A. C. A. Holzapfel. An improved composition for preventing corrosion and fouling on ships' bottoms and other submerged surfaces. September 2.

13,861. A. A. Blandy. Improvements in the combination and treatment of certain materials for the production of substances as a substitute for india-rubber and leather, and for other substances and purposes for which it may be applicable. September 3.

14,106. H. Joyce. Improvements in polishes or compositions for leather and other surfaces. September 8.

14,107. R. F. Metzeler. An improved vulcanised composition. September 8.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

14,150. S. Kenyon. Treating india-rubber to render it soluble. September 10.

15,805. W. L. Wise.—From J. P. Perkins. A new pigment or paint base, and method of producing same. August 27.

18,628. L. T. Thorne and Brin's Oxygen Co., Limited. See Class XII.

#### XIV.—TANNING, LEATHER, GLUE, AND SIZE.

##### APPLICATIONS.

13,603. D. Cielitira. An improved method for the instantaneous production of imitation Russia leather. August 29.

13,794. E. Edwards.—From A. Nicolet, France. Improvements in the preparation and treatment of skins and leather. September 2.

13,796. W. J. J. Hawkins. Improvements in printers' roller composition. September 2.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

15,450. B. Nicholson and T. Palmer. Preserving and recovering the colouring matters from tanning and other vegetable extracts. September 10.

16,595. H. Bogenschield and F. Busch. Process and apparatus for glazing leather. September 17.

#### XV.—AGRICULTURE AND MANURES.

##### APPLICATION.

14,321. A. Buchanan. — From A. W. Tait, Portugal. Improvements in compounds for protecting vines, potatoes, and other vegetable growths from disease. September 11.

#### XVI.—SUGARS, STARCHES, GUMS, Etc.

##### APPLICATION.

13,444. J. Y. Johnson.—From A. L. L. Peek, United States. Improvements in the manufacture of lactose or milk sugar. August 26.

#### XVII.—BREWING, WINES, SPIRITS, Etc.

##### APPLICATIONS.

12,932. W. T. Thorp. An improved beer compound. August 18.

13,257. G. F. Redfern.—From G. Perschied, United States. Improvements in the manufacture of wine, and in apparatus therefor. Complete Specification. August 22.

13,428. H. H. Lake.—From The Handford-Stanford Co., United States. Improvements in apparatus for cooling and aerating wort in the manufacture of malt liquors. Complete Specification. August 26.

13,608. R. Genge. Improvements relating to the production of dry yeast, cattle food, and spirit from maize, wort, and refuse spirit liquors. August 29.

13,694. M. Konig. Improvements in malt curing apparatus. Complete Specification. August 30.

13,862. B. E. R. Newlands. Improvements in the treatment of grain and malt, and the production therefrom of a new product more especially intended for use in brewing and distilling or as a food. September 3.

14,118. G. Roskilly. Refining and heading malt liquors. September 8.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

15,568. J. F. Gallogly. Maturing and mellowing new whisky, brandy, or like alcoholic liquors. September 10.

16,556. D. Wickham. Novel condensing cylinder for gasating malt and other liquors. August 27.

1890.

6337. W. Paterson. Preventing and arresting development of acids during malting or storage of barley and other cereals, and in the infusion of malted barley for the manufacture of concentrated malt extract, or for all brewing purposes. September 3.

10,845. A. Boake and F. G. A. Roberts. Means of applying certain antiseptics to beer, wine, &c. September 17.

11,249. A. Regel. Pasteurising apparatus. September 16.

11,554. G. F. Redfern.—From J. F. Gronwald, E. H. C. Oehlmann, and G. H. Neuhauss. See Class XVIII.—A.

#### XVIII.—CHEMISTRY OF FOODS, SANITARY CHEMISTRY, AND DISINFECTANTS.

##### APPLICATIONS.

##### A.—Chemistry of Foods.

12,955. G. H. Hamrick. The manufacture and preservation of butter. Complete Specification. August 18.

13,067. J. G. Lorrain.—From F. J. Chevet, France. A new or improved alimentary preparation, and method of manufacturing the same. August 20.



13,068. J. G. Lorrain.—From F. J. Chevet, France. A new or improved alimentary preparation, and method of manufacturing the same. August 20.

13,069. J. G. Lorrain.—From F. J. Chevet, France. A new or improved alimentary preparation, and method of manufacturing the same. August 20.

13,070. J. G. Lorrain.—From F. J. Chevet, France. A new or improved alimentary preparation, and method of manufacturing the same. August 20.

13,089. C. L. Bachelier. A new or improved process and apparatus for preserving organic substances, and especially alimentary substances. Complete Specification. August 20.

13,140. W. H. E. Rawlings. A new digestive milk food. August 26.

13,552. W. F. Scutt. Solid milk. August 28.

13,792. A. H. Cone. A baking powder. September 2.

13,824. F. W. Waide. Improvements in mixing and preserving meats, fish, game, and the like, with apparatus for performing the same. September 3.

14,051. H. Rouart. See Class I.

#### *B.—Sanitary Chemistry.*

12,910. A. C. Wilson and A. Ford. Improvements in and relating to the treatment of town, ashpit, or the like refuse, and in the means or apparatus employed therefor. August 18.

14,142. G. V. Alsing. Improvements in the utilisation of sewage sludge for the purpose of vitrification. September 9.

14,253. A. O. Jones and S. Fox. Improvements in the treatment of sewage. September 10.

14,254. A. O. Jones. Improvements in the treatment of sewage, and apparatus therefor. September 10.

#### *C.—Disinfectants.*

13,046. W. P. Thompson.—From J. R. Bate, F. W. Owen, and J. W. Simcock, United States. Improvements in and relating to preserving compounds for embalming purposes. Complete Specification. August 19.

13,488. J. Y. Johnson.—From E. Salomon, France. The manufacture and production of a new antiseptic. Complete Specification. August 27.

#### COMPLETE SPECIFICATIONS ACCEPTED.

##### *A.—Chemistry of Foods.*

1890.

10,950. M. Persson. Preparation of condensed cream. September 3.

11,554. G. F. Redfern.—From J. F. H. Gronwald, E. H. C. Oehlmann, and G. H. Nenhaus. Apparatus for sterilising milk and other liquids, and decanting same. August 27.

11,586. Y. Tobias and H. Fischer. A liquid chocolate, free from fat and preservable. September 3.

##### *B.—Sanitary Chemistry.*

1889.

17,898. A. Young. Apparatus for destroying noxious vapours of drains, sewers, hospitals, &c. September 17.

1890.

4732. W. Birch. Apparatus for filtering sewage, &c. September 10.

#### *C.—Disinfectants.*

1890.

12,323. H. H. Lake.—From W. F. Simes. Compound for disinfecting and other purposes. September 10.

#### XIX.—PAPER, PASTEBOARD, ETC.

##### APPLICATIONS.

13,677. A. Johnstone. Improvements in the manufacture of paper and in mechanism connected therewith. August 30.

#### XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

##### APPLICATIONS.

12,993. J. Y. Johnson.—From F. von Heyden, Germany. Improvements in the manufacture of oxyphthalic acids (phenol dicarbonic acids). August 19.

14,437. M. Freund and M. Heim. Process of producing new derivatives of hydrastine and narcotine. September 13.

#### COMPLETE SPECIFICATION ACCEPTED.

1889.

16,555. O. Imray.—From W. MacKean. Obtaining salts of thorium, zirconium, and like metals, from their ores or from waste residues of hoods used for incandescent gas lighting. August 27.

#### XXI.—PHOTOGRAPHIC PROCESSES AND MATERIALS.

##### APPLICATIONS.

13,262. J. C. Osborne. The removal of the action of light on untuned sensitised and albuminised photographic papers and surfaces. August 23.

13,836. J. J. Aeworth. Improvements in the manufacture of photographic films and in apparatus therefor. September 3.

13,937. C. Ball, P. N. Evans, and Q. Wirtz. Method of rendering photographs on glass, china, or the like, permanent. September 4.

14,189. V. Planchon. Improvements relating to films for use in photography. September 9.

## XXII.—EXPLOSIVES, MATCHES, Etc.

## APPLICATIONS.

13,085. M. Meisgen and M. Hett. An improved fuse or priming machine for kindling fuels. Complete Specification. August 20.

13,801. G. W. Thomas. Improvements in matches, fuses, and the like. September 2.

14,080. C. O. Lundholm, J. Sayers, and W. Cullen. Improvements in the manufacture of explosives. September 8.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

11,678. A. V. Newton.—From A. Nobel. Improvements in cartridges. September 10.

16,028. P. Molyneux. Improvements in wax matches. August 27.

16,426. J. S. Taylor and S. W. Challen. Plant for burning special charcoal for the manufacture of gunpowder. September 10.

1890.

9315. E. N. Todd. Manufacturing thin sheets of nitro-cellulose. September 10.

11,018. W. P. Thompson.—From S. D. Smolianinoff. Explosive compounds. September 10.

# THE JOURNAL

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Dec. 1st.—Mr. Wm. Webster. "The Electrical Treatment of Sewage."

1891.

Jan. 5th:—

Mr. Percy Gilchrist. "The Basic Copper Process."

Mr. W. C. Young. "On Standard Sperin Candles."

Feb. 2nd.—Dr. W. S. Squire. "The Artificial Production of Cold Theoretically and Practically considered."

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Dec. 2nd (Edinburgh).—Mr. R. Irvine. "The Condensation of Smoke Particles."

1891.

Jan. 5th (Glasgow).—Mr. H. Ballantyne. "On the Effect of Exposure, under Certain Conditions, upon some Constants of Oils."

Feb. 2nd (Edinburgh).—Dr. J. B. Readman. "The Manufacture of Phosphorus: Part III."

Mar. 2nd (Glasgow):—

(1.) Dr. E. J. Mills. "Destructive Distillation: Part II."

(2.) Messrs. R. T. Thomson and H. Ballantyne. "On the Revision of Constants Employed in the Analysis of Fats and Oils: Part II."

### ERRATUM.

In the September number of this Journal, page 846, line 19 from top of column II., instead of "had to be done" read "had been done."

## Journal and Patent\* Literature.

### I.—GENERAL PLANT, APPARATUS, AND MACHINERY.

Action of Tannin on Iron. L. Manstetten. Chem. Zeit. **14**, 869-870.

L. VIGNON (Chem. Zeit. **14**, 117; this Journal, 1890, 725 and 767), in a note on tannin as an anti-incrustator, expresses the opinion that its use is injurious to iron boilers on account of its corrosive action. The author attacks this view, and while holding no brief for tannin in this capacity, yet gives reason for belief that it has little tendency to attack iron after the first contact, the metal becoming coated and protected from further action. His experiments with iron as a material for vessels used in the extraction of tanning materials show that the quantity of iron taken into solution is inconsiderable, and that, although the first few batches of liquor are discoloured, and have to be filtered through spent tanning matter, subsequent extracts are unaffected. Further, direct experiments, consisting in boiling slips of sheet iron in tannin solutions for different periods, show the rate of attack to be very slow. He concludes that the risk from the use of tannin as an anti-incrusting agent has been much exaggerated.—B. B.

### PATENTS.

Improvements in Lead Tanks, and in the Method of Manufacturing the same. P. J. Davies, London. Eng. Pat. 12,157A, July 31, 1889, *8d*.

See under XI., page 952.

Improvements in and in Apparatus for Concentrating and Evaporating Liquids, and for Burning off Organic Matter therefrom. C. H. Roeckner, Tynemouth; F. L. and R. L. Roeckner, Hemel Hempstead. Eng. Pat. 12,871, August 15, 1889, *8d*.

THE inventors claim novelties of arrangement in four classes of apparatus:—(1.) When boiling fibrous materials or similar matters in pans or trays, they introduce into the apparatus a mid-feather or division, and, by means of agitators, force the liquid to travel round and round, whereby the heat is to be more fully utilised and evaporation from the surface of the liquid facilitated. (2.) When incinerating partially dried and thickened materials, they place them in ovens with circular floors, from the centres of which sets of rotatory agitators or stirrers are made to work, whereby all organic matter can be burned off to any desired extent or reduced to ashes without manual labour. (3.) When constructing apparatus for evaporating much diluted liquids by multiple effect, they place all evaporating surface of their apparatus diagonally or inclined instead of either vertically or horizontally, with a view to obtaining better economical results through facilitated evaporation. (4.) In tubular apparatus for direct evaporation they also place the tube boxes inclined or diagonally, and they cause the weakest liquor to enter the system nearest the fire-grate so that it may meet with the greatest heat at starting.

The specification is illustrated by four sheets of drawings.—B.

\* Any of these specifications may be obtained by post, by remitting the cost price, plus postage, to Mr. H. Reader Luck, Comptroller of the Patent Office, Southampton Buildings, Chancery Lane, London, W.C. The amount of postage may be calculated as follows:—

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" 2 <i>s</i> . 4 <i>d</i> ., " " 3 <i>s</i> . 4 <i>d</i> . . . . .	2 <i>d</i> .

*Improvements in and relating to Filtering Apparatus.*  
R. C. Gerville, Hamburg, Germany. Eng. Pat. 16,499,  
October 18, 1889. 8d.

THE object of this invention is to provide means for cleaning the surfaces of the filtering material by reversing the direction of the liquid through the filtering substance. This is effected by a tap of peculiar construction, having a hollow plug, by adjusting the position of which the supply of liquid may be shut off, or the filtered liquid drawn off, or the direction of the liquid through the filtering medium reversed. The filter case is preferably circular, and at one point partially encloses the tap. The filtering medium is cemented into the filter casing, and to the top, so as to leave spaces between its front and rear sides, and the top and bottom of the casing, and communicating with the tap. For details of the construction and mode of using the tap the drawings attached to the specification must be consulted. There are six claims.—E. S.

*Improved Apparatus for Use in making Extracts from, and otherwise treating, Soluble or Partially Soluble Substances by Means of Steam Pressure and Heat.*  
A. H. Hobson, London. Eng. Pat. 16,863, October 25, 1889. 8d.

THE improvements relate to apparatus principally designed for use in extracting the soluble constituents of bones, hides, roots, dyestuffs, &c., and generally for treating materials by steam and heat for the elimination of extractive matters and other purposes. The extractor consists of a spherical or other shaped vessel provided with hollow trunnions and mounted to revolve about a stationary tubular axis, and having steam inlet and exhaust passages, and meridionally arranged heating and agitating pipes in connexion with them, and also a perforated jet pipe arranged to inject steam into the mass to be treated.

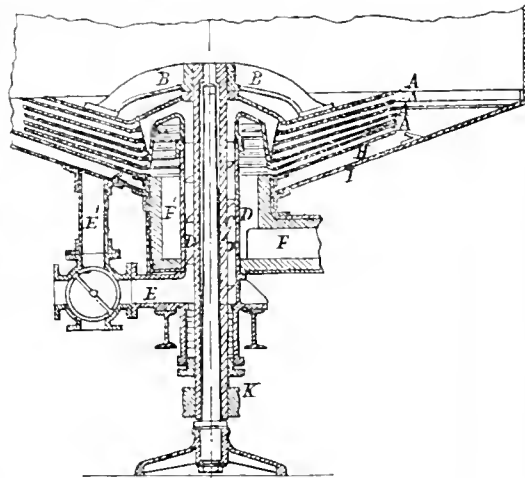
The extractor having been charged, steam at suitable pressure is admitted to the pipes for heating the charge, and to the jet pipe. The difference of pressure between the generator and the extractor necessary to ensure the efficient action of the steam jet is maintained by an automatic steam cock.—E. S.

*Improvements in Vacuum Pans.* W. K. Bruce, Dundee.  
Eng. Pat. 17,018, October 28, 1889. 8d.

THE vacuum pan is constructed of two readily separable parts, which when the pan is in use are held together by the pressure of the external atmosphere, without the use of bolts. The upper part or dome is provided with a pulley and counterbalance weight, and the lower part is supported on a suitable frame by hollow trunnions, through which the steam supply and exhaust pipes pass. By means of a lever the lower part can be turned about its trunnions to permit of the removal of its contents. By this construction ready access can be had to the interior of the pan for cleaning or other purposes. There are five claims.—E. S.

*Improvements in Apparatus for Evaporating Liquids.*  
E. Theisen, Sinzig, Germany. Eng. Pat. 19,222, November 29, 1889. 1s. 1d.

THE inventor constructs apparatus for the rapid evaporation of liquids in such a way as to cause the liquid together with air or gas to pass through centrifugal apparatus consisting of a number of rapidly revolving discs, fans, or plates, in which the liquid and the gases are brought into close contact either whilst passing through the discs or immediately on issuing therefrom. There are also claims for means of raising the liquid to the apparatus from a lower level, for augmenting the propelling action of the fans, for working several sets together in multiple effect, and for other modifications. The general mode of action will best be understood from the accompanying sketch, which shows a vertical section through one class of apparatus.



THE RAPID EVAPORATION OF LIQUIDS.

The dished plates A A are secured by the arms B to a hollow shaft C, which receives rapid motion by means of the pulley K. The spaces between the discs A communicate through channels L with a stationary tube D surrounding the shaft C, which tube is in connexion with a pipe E through which the liquid is admitted, aided by the screw G secured to the rotatory shaft. The cylindrical chamber F1 supplies the air or hot gases through the pipe F and also carries the casing I of the centrifugal apparatus as well as a stationary dished plate H. On the mixture of liquid and gases issuing through the plates A a rapid evaporation takes place, the vapours are led away, whilst the unevaporated liquid returns through the pipes F1 and E for repeated manipulation. The specification covers seven claims and there are nine sheets of drawings.—B.

*Improvements in Filter Presses.* S. H. Johnson and C. C. Hutchinson, Stratford. Eng. Pat. 8544, June 3, 1890. 8d.

THE object of these improvements is threefold:—

(1.) To facilitate the escape of the filtered liquid after having passed through the filtering cloths. This is done by constructing the partition plates with their surfaces formed of projecting studs with reticulated interspaces between them. This construction also increases the accessibility to the surfaces for cleaning.

(2.) To preserve the filtering cloth by preventing undue wear at certain points. This is effected by the employment of hydraulic lipped packings in the rims of the filter plates, whether moulded with the lips or the lips formed by the operation of closing the joints.

(3.) To secure tight joints in the inlet, outlet, and washing passages. This is done by "an elastic packing ring with lips projecting so as to come into gentle contact with the joint surfaces of the adjoining lugs and so contrived that the pressure due to the working of the machine becomes the effective agent."—E. S.

*Improvements in Pulverising Mills.* H. H. Lake, London.  
From V. L. Rice, New York, U.S.A. Eng. Pat. 9809,  
June 24, 1890. 8d.

A CHAMBER made of iron, and in the form of a pan, contains a steel ring which forms one of the grinding surfaces. Round the interior of the ring three rolls suitably mounted on shafts are caused to revolve. The centrifugal force generated by the revolution of the rolls and their shafts causes them to swing outwardly into contact with the ring, and at the same time causes them to turn on their axes as they revolve round the chamber. When the rolls are at rest they fall away from the wall of the chamber. Some of

the rolls are provided preferably with a spiral flange which lifts the pulverised material and causes it to be distributed over the surface of a circular screen, mounted on the upper edge of the chamber and constructed with a slight outward taper. When pulverised to the required fineness, the material passes through the screen and falls into the hopper. The two drawings accompanying the specification must be consulted for details of construction. There are eight claims.—E. S.

## II.—FUEL, GAS, AND LIGHT.

*On a Violet Colouring Matter from Residues of Gas Purifiers or Illuminating Gas.* R. Gasch. Jour. f. Gasbeleuchtung, **33**, 304—306.

This colouring matter is the iron salt of potassium carbonyl-ferrocyanide. Illuminating gas in its passage from the purifier to the gas holder is led through an alkaline solution of ferrous oxide; the solution is acidified and the ferrocyanide precipitated by zinc or ferrous sulphate. The filtrate gives the violet colour on the addition of ferric chloride before the sulphocyanide reaction appears. If, however, the solution contains only a little of the former, the sulphocyanide reaction appears first and the violet later. The colouring matter may be obtained by precipitation with sodium chloride. The violet colouring matter is obtained from gas purifier residue by extraction with weak soda or strong ammonia and precipitation of the acidified solution with zinc or ferrous sulphate. If the latter is used the sulphate should be added until the blue precipitate ceases and a white one appears. The violet is precipitated from the filtrate by ferric chloride.—A. L. S.

*On Purifying Material.* E. Schilling. Jour. f. Gasbeleuchtung, **33**, 322—326.

It is well known that an analysis of a gas purifying mass does not indicate its value. Factors that require to be taken into consideration besides the composition of the material, are: weight, size of grain, moisture, the substance for rendering the mass more penetrable, the depth of the layers, the velocity and difference in pressure of the gases previous to entering and after leaving the purifier. In order to find the highest value of a certain purifying mass, the author passed a current of pure sulphuretted hydrogen over the finely powdered material, retaining the water which was produced by phosphoric anhydride. Even after passing the current through for 20 hours, complete saturation did not take place. The chief action was over after three hours. Pure ferric hydroxide absorbs on an average after the first saturation, 30.77 of sulphur, different varieties of bog-iron ore 11.2—28.41; the theoretical amount of sulphur corresponding to the amount of iron oxide present varied from 18.06—42.69. From this it will be seen that in no case, not even that of pure ferric hydroxide, was the maximum absorption equal to that theoretically possible. And it does not appear that there is any relation between the amount of iron in the material and the amount of sulphur absorbed.

Referring to the difference in absorbing capacity as influenced by certain physical conditions the author points out that the quantity of sulphur taken up was doubled by interspersing wood shavings in the mass.—A. R.

*Cyanogen in Gas Manufacture.* W. Leybold. Jour. f. Gasbeleuchtung, **33**, 336—341. (See Appendix, "The Estimation of Cyanogen in Gas," page 979.)

The amount of nitrogen contained in coal varies from about 1.06 to 1.50 per cent. Knnblanch found that for every 100 parts of nitrogen in the coal there were found: 31.36 in the coke, 10—14 as ammonia, 1.5—2 as ferrocyanogen (in the purifying material), 1.0—1.3 in the tar; the remaining 46—56 parts would probably be in the illuminating gas.

The cyanogen in the crude gas exists as ammonium cyanide, which being very volatile even at 36° C. passes on with the gas through the condensers and scrubbers till it reaches the purifiers without much loss; a little however being found in the liquors condensed as ammonium sulphocyanide, and owing to the power of the cyanide to dissolve the iron of the apparatus, as ferrocyanide. In one case there was found in the liquor from the hydraulic main per litre 23 grms. of ammonium chloride, .88 gm. of sulphocyanide, and .11 gm. of ferrocyanide; in the liquor from two pair of atmospheric condensers .52 gm. and .33 gm. of sulphocyanide, .05 gm. and .11 gm. of ferrocyanide, .12 gm. and .09 gm. of unaltered cyanide; in the water condenser 1.27 grms. of sulphocyanide, .44 gm. of ferrocyanide, and .25 gm. of cyanide. In the scrubber even, very little cyanogen was found, the ammonium cyanide, though readily soluble in water, being decomposed by the carbonic acid into ammonium carbonate and free hydrocyanic acid.

The following tables indicate the amount of hydrocyanic acid retained by the various parts of the gasworks apparatus. The coals employed were of five different sorts, carbonised in separate retorts, and so arranged that the gas mixed in the hydraulic main.

TABLE I.—CYANOGEN IN THE GAS.

Source.	Grms. HCN per 100 cbm.	Per Cent. HCN by Vol.	Removal of the HCN	
			By Weight.	Per Cent.
Hydraulic main.	203.4	0.166	Grms. 16.3	8.02
After condensers	187.1	0.152	13.5	6.55
After scrubber ..	173.6	0.142	114.1	65.15
After purifiers ..	59.5	0.048	39.7	19.55
Holders .....	19.8	0.016	19.8	9.73
			203.4	100.00

TABLE II.—CYANOGEN IN THE GAS.

Source.	Grms. HCN per 100 cbm.	Per Cent. HCN by Vol.	Removal of the HCN	
			By Weight.	Per Cent.
Hydraulic main.	265.9	0.217	Grms. 10.0	3.76
After condensers	255.9	0.209	4.3	1.62
After scrubber ..	251.6	0.205	119.9	45.00
After 1st purifier	131.7	0.107	48.4	18.20
After 2nd purifier	83.3	0.067	21.7	8.16
After 3rd purifier	61.6	0.050	20.4	7.67
Holders ....	41.2	0.033	41.2	15.50
			265.9	100.00

These tables show that from 56 to 71 per cent. of the total amount of cyanogen formed is retained in the iron-oxide purifiers; Table II. also proving that the largest amount is found in the first purifier. The more purifiers employed, the larger is the yield of cyanogen, for even in the absence of sulphuretted hydrogen it is still absorbed by oxide that has been used, whether revived or not.

The cyanogen is recovered as prussian blue; and to obtain a spent oxide containing a considerable amount, it is advisable to retain one purifier as No. 1 for a long time after it has ceased to take up sulphuretted hydrogen, the rest of the purifiers being managed as usual. By this means it is possible to obtain from 8 to 10 per cent. of prussian blue. The speed of the gas has great influence on the amount recoverable; the purifiers, therefore, should be as large as possible.



When hydrocyanic acid is passed through a layer of iron oxide as employed in gasworks, it is not absorbed; but if the material is first saturated with sulphuretted hydrogen, so as to form iron sulphide, the greater part is taken up, even after revivification. Iron cyanide is produced, which, in presence of air, is converted into iron ferrocyanide or prussian blue; this, however, is not of constant composition, but is a mixture of various compounds, some of which, especially when the yield is high, contain ammonia in combination. A trace of sulphocyanogen is generally found at the same time, and when the scrubbing has been inefficient, so that the gas enters the purifiers containing large quantities of ammonia, the yield of sulphocyanogen is often higher than that of blue.

To imitate these latter conditions, a mixture of hydrocyanic acid and ammonia was passed through an oxide purifier previously saturated with sulphuretted hydrogen. The following figures were obtained, the speed of the gas being lower in the second case:—

	Per Cent.	Per Cent.
Water.....	26.62	32.33
Sulphur.....	24.98	18.98
Prussian blue.....	1.71	3.57
Ammonium sulphocyanide.....	3.03	6.25
Ammonia.....	2.05	2.08

Ammonia has also the effect of increasing the yield of sulphocyanogen at the expense of the ferrocyanogen. By substituting ammonium sulphide for the free ammonia, the following result was obtained:—

	Per Cent.
Water.....	33.02
Sulphur.....	11.39
Prussian blue.....	5.38
Ammonium sulphocyanide.....	4.40
Ammonia.....	0.75

A revived purifier was moistened with ferrous sulphate solution (10 per cent.), to keep it in an acid condition, and the mixture of hydrocyanic acid and ammonia passed over it. The amount of prussian blue found was 3.32 per cent., and of ammonium sulphocyanide 2.61 per cent., thus showing that a neutral condition is the most favourable for the formation of the former. By making the oxide alkaline with soda, and then treating with hydrocyanic acid, the reactions became more complicated:—

	Per Cent.
Water.....	32.52
Sulphur.....	6.39
Prussian blue.....	0.93
Sodium sulphocyanide.....	0.73
Sodium cyanide.....	1.13
Sodium ferrocyanide (cryst.).....	0.89

The author's experiments show that the percentage of blue contained in the spent oxide, and, therefore, its marketable value, may be greatly increased by removing the free ammonia from the gas as thoroughly as possible before it enters the purifiers. In one works where new scrubbers were erected the yield rose from 3.0 per cent. to 6.23 per cent., the sulphocyanogen falling from 5.96 per cent. to 3.22 per cent., while with suitable oxide, by allowing the gas to remain in contact with the material for some time, it may rise as high as 10.32 per cent.

From the above tables Nos. 1, and 11., it will be seen that of the total cyanogen reaching the purifiers, only 65 and 85 per cent. is recovered. To avoid this loss, which in large works is considerable, a process has been devised by Knublauch, in which the gas is passed through a scrubber containing a mixture of soda and ferrous sulphate solution, the best position for which is next the hydraulic main.

—F. H. L.

*On the Use of Magnesium Chloride Solution for filling Gas Meters.* W. Leybold. Jour. f. Gasbeleuchtung, **33**, 424.

The author recently examined an old gas meter, which had been in use for several years, the drum of which was made of britannia metal. The solution was found to be of

the same specific gravity as when first put in (22° B.), and was perfectly clear; but on the walls of the meter there was a heavy brown deposit consisting of zinc sulphide, iron sulphide, iron cyanide, lead hydrate, and tin oxide. The zinc must be derived from the vessels used to prepare and keep the magnesium chloride, none entering into the composition of the meter. On opening the drum, the britannia metal was found to be unattacked, but the leaden U-tube as well as the soldering was eaten away.

The author recommends the use of magnesium chloride only when the gas is free from ammonia, the action of the latter being to form magnesium carbonate, which is insoluble in the solution of the chloride and separates out in thick crusts. Meters filled as above should be distinctly marked, to avoid subsequent addition of glycerin.—F. H. L.

*The Weathering of Coal.* Busse. Deutsche Töpfer und Ziegler Zeit. 1890, **21**, 353.

COAL, when exposed to the weather, is acted on by the oxygen of the air up to a definite limit; it combines with the free hydrogen of the coal to form water, and with a part of the carbon to form carbonic acid. When the limit is reached a certain amount of oxygen is yet taken up and mechanically held by the coal. Thus, as the result of weathering, coal loses some of its combustible constituents, and gains in weight owing to the absorption of oxygen. This may amount to as much as 4 per cent. on the original weight of the coal. The effect of weathering is greater the smaller and the more porous the lumps are.—A. L. S.

## PATENTS.

*Improvements in and Connected with the Manufacture of Illuminating Gas from Coal.* J. H. R. Dinsmore, Liverpool. Eng. Pat. 7714, May 27, 1887. Amended May 24, 1890. 8d.

THE emendations cannot be clearly understood without reference to the original specification, which must be consulted for details. Briefly stated, the process now claimed is carried out by "distilling the coal in closed retorts, heating the gas so produced, and then subsequently cooling it upon leaving the apparatus."—O. H.

*Improvements in or Appertaining to Appliances for Burning Hydrocarbons.* J. Wilson, New York, and A. Mason, Brooklyn, U.S.A. Eng. Pat. 10,660, July 2, 1889. 8d.

THE improvements relate to contrivances for burning hydrocarbons (petroleum and other oils and fatty substances, gases and vapours, &c.) in steam boiler and other furnaces, for details of which the original specification must be consulted.—F. S. K.

*Improvements in the Manufacture of Illuminating Gas.* F. J. Jones, St. Albans. Eng. Pat. 11,624, July 20, 1889. 8d.

THE hot gases (water-gas) resulting from the action of superheated steam on coke are passed through a charge of coal moving continuously downwards. The mixture of gases, tarry matter, and other vapours so obtained is at once passed through a superheater and the resulting gas then purified in the ordinary way. The apparatus employed is described in Eng. Pat. 19,721 of 1889.—H. K. T.

*Improvements in or Relating to the Manufacture of Gas from Atmospheric Air.* J. F. L. Monier, Paris, France. Eng. Pat. 12,043, July 29, 1889. 8d.

THE object of the present application is to provide a new apparatus for making gas by means of the earthenware of atmospheric air, and which offers certain advantages over the various patterns of the same style of apparatus at present in use.

"The fundamental principle of the apparatus consists in the application of an ejector based upon the Giffard system with a draught of air by means of a jet of steam, and planned in such wise that, given an effective minimum pressure, for example, equivalent to  $1\frac{1}{2}$  kilos., while the volume of air drawn along shall correspond to the required supply, the carburetted air thus transformed into illuminating gas will issue at the outlet of the apparatus under a pressure of water equivalent to at least 70 mm., whereby gas can be carried to great distances and up to any desired altitude."

The gas is produced as follows: Air, by means of a steam ejector, is passed into a water-cooler, where the steam is condensed, and then into a dome or bell fitted at the upper part of the apparatus and suspended over a pulley; the weight of the bell must be proportionate to the pressure of steam forcing air into the carburettor, and its vertical guidance is effected by means of an iron rod, fitted to the bottom of the reservoir, and extending into a pipe fixed to the upper part of the bell; the lower extremity of this pipe is perforated to allow the surplus air to escape at a time when, owing to pressure, the bell might be caused to ascend too high. From the bell the air is conducted into the carburettor through a pipe fitted at the lower part of the apparatus and immersed in the hydrocarbon. This pipe is made in the shape of a double coil and is provided along its entire length with a number of perforations through which the air escapes, and, passing upwards through a perforated tray immersed in the hydrocarbon, is finally carried off to the gas supply pipe.

The apparatus is easy of application in establishments where a steam engine is fitted; for application where no steam engine is available a small steam generator has been designed.—F. S. K.

*Improvements in Air and Gas Comminglers.* W. W. Popplewell, London. From J. M. Mitchell, New York, U.S.A. Eng. Pat. 13,526, August 27, 1889. *Sd.*

This apparatus consists of two rotary blowers coupled together by which air and gas are charged into the same conduit. An arrangement is provided by which the relative volumes of air and gas can be altered.—H. K. T.

*Improvements in and relating to Gas Washers.* G. E. Saville, Millwood. Eng. Pat. 15,740, October 8, 1889. *Sd.*

The gas enters a trough or bell inverted in a closed tank through which ammoniacal liquor flows and is drawn through perforations in the lower sides of the inverted trough by the exhauster. Should the speed of the latter become accelerated, the liquor, which is supplied freely, will be drawn up between the sides of the trough and the tank and so prevent excessive exhaustion in the retorts, &c., and consequent mixing of furnace gas with the coal gas. The lower part of the tank communicates with a cylindrical vessel open at the top for regulating the height of the liquor. In the centre of this regulator is a funnel, over the edge of which the liquor overflows and is led away.

—H. K. T.

*Process for obtaining Salts of Thorium, Zirconium, and like Metals from their Ores or from Waste Residues of Hoods used for Incandescent Gas Lighting.* O. Imray, London. From W. MacKean, Paris, France. Eng. Pat. 16,555, October 19, 1889. *6d.*

See under VII., page 941.

*Apparatus for Producing an Inflammable Gas by Carburation of Air.* A. A. Fritz, Brussels, Belgium. Eng. Pat. 17,140, October 29, 1889. *1s. 1d.*

A liquid hydrocarbon is supplied under pressure to a reservoir, passing thence to a boiler, the amount so passing being regulated by a tap actuated by the bell of a gasometer. From the boiler, which is heated by Bunsen

burners, the hydrocarbon issues in the form of vapour through injectors and draws air with it, the air entering through check valves, which close against any outflow. The mixed air and vapour pass into the gasometer, thence to a scrubber composed of layers of coke, supported on wire gauze, and so to the main. The rate of consumption determines the yield of hydrocarbon, since, when the gasometer rises, the supply of hydrocarbon to the boiler is cut off. Any liquid which drains from the scrubber is returned to the reservoir. Drawings descriptive of the different forms of the apparatus accompany the specification.

—H. K. T.

*Improvements in Saturating Coal Gas, Air and other suitable Gases with Hydrocarbon Vapour for Limelight and other Purposes.* A. W. Scott, Weston-super-Mare. Eng. Pat. 17,378, November 2, 1889. *8d.*

This apparatus consists of a closed vessel filled with cotton-wool or other porous material, and furnished with an inlet and outlet tube. A clear path is maintained between the two tubes by means of a skeleton tube of wire similar to those used for lining india-rubber tubing. The vessel is immersed in a water-bath. It is charged by filling it with benzene or commercial "benzoline," or any hydrocarbon boiling between  $100^{\circ}$  and  $200^{\circ}$  F., any liquid unabsorbed by the cotton-wool being drawn off. The apparatus is used for saturating oxygen previous to its passage through an ether saturator or for saturating hydrogen or coal-gas for the usual oxy-hydrogen limelight.—H. K. T.

### III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

*Nononaphthene and its Derivatives.* M. I. Kononov. J. Russ. Chem. Soc. 22, 4—23 and 118—148.

THIS is a first detailed study of one of the members of the naphthene series recently found in Caucasian naphtha. The fraction of Baku kerosin boiling at  $135^{\circ}$ — $137^{\circ}$  was found to consist of the hydrocarbon  $C_{10}H_{18}$ , and served the author as material for the study. Three specimens of the same hydrocarbon differed but slightly in their specific gravity, having at  $20^{\circ}$  respectively a density of 0.7652, 0.7647, 0.7664. Like all other naphthenes it does not deviate the plane of polarisation; it is insoluble in water, but very hygroscopic, and can be distilled almost without decomposition.

Not only fuming sulphuric acid on heating, but even dilute sulphuric acid at the ordinary temperature acts upon nononaphthene very considerably, evolving sulphur dioxide and becoming black and thick. Thus Mendeléeff's advice to avoid chemical purification of naphtha hydrocarbons by means of sulphuric acid appears to be well founded.

The final result of the reaction of fuming sulphuric acid on nononaphthene is, according to the author, undoubtedly pseudocumene mono- and di-sulphonic acids, accompanied by some tar-like oil.

Bromine acts on nononaphthene in direct sunlight or on heating strongly. In presence of a small quantity of aluminium bromide, bromine forms with nononaphthene a crystalline bromide, as with all naphthenes containing more than  $C_7$  in the molecule. These needle-like crystals are formed even when the nononaphthene is obtained by hydrolysing chloronaphthyl, i.e., when it is absolutely free from any aromatic substance. The crystalline bromide proved to be  $C_{10}H_{17}Br_2$ , melting at  $227^{\circ}$ — $228^{\circ}$ ; tribromopseudocumene ( $C_{10}H_{15}Br_3$ ) melts at  $225^{\circ}$ — $226^{\circ}$ . The author assumes this slight difference in the temperature of melting is caused by the admixture of  $C_{10}H_{17}Br_2$  with the former, which melts at  $240^{\circ}$ — $241^{\circ}$ . It is not improbable that a methyl-group is eliminated by the action of bromine on the naphthene, and the action of nitric acid confirms this.

Chlorine combines with nononaphthene under ordinary conditions, whilst when in excess and in direct sunlight it reacts with a slight explosion; the outcome being mono- and dichloride,  $C_9H_7Cl$  and  $C_9H_6Cl_2$ .

Fuming nitric acid hardly affects the hydrocarbon, the nitro products are formed, but in so small quantity that they may be ascribed to the impurities of the hydrocarbon. The author tried dilute nitric acid (sp. gr. = 1.38 + 2 vols. of water), and succeeded in obtaining, on heating it with the hydrocarbon in sealed tubes, a nitro-product containing only  $C_9$  in a molecule— $C_9H_7NO_2$ , besides an acid solution containing products of oxidation. The former is a yellowish oil, boiling very constantly at  $218^\circ$ — $220^\circ$ , and having a feeble smell, dissimilar from that of aromatic compounds. Under the reducing action of hydrogen, it becomes a volatile alkaline liquid, resembling *conine* and of the same formula (propylpiperidine),  $C_8H_{15}NH_2$ . It is strongly alkaline, fuming like ammonia on neutralising it with hydrochloric acid; boils at  $172^\circ$ — $177^\circ$ , specific gravity at  $0^\circ$  = 0.8727, at  $20^\circ$  = 0.8717 (*conine* boils at  $167^\circ$ — $168^\circ$ , specific gravity at  $0^\circ$  = 0.8625). This amine combines with hydrochloric acid, platinum chloride, and sulphuric acid, giving respectively fixed salts; chloride,  $C_8H_{15}NH_2HCl$ , crystallising from hot aqueous solution in yellow tablets and needles. The platino-chloride,  $(C_8H_{15}NH_2 \cdot HCl)_2PtCl_4$ , is hardly soluble in water.

The sulphate,  $[C_8H_{15}NH_2]_2H_2SO_4$ , on being analysed proved this formula. The author ascribes a great importance to the study of these two classes of nitro- and amido-compounds, as they may serve for obtaining a series of homologous hydrocarbons.

On the other hand, the splitting of the molecule by the action of nitric acid, bromine in presence of aluminium bromide, and probably also by the action of sulphur, suggests to the author an explanation of the oxidation of the hydrocarbon. By the action of oxidising agents, nononaphthene never gives acids containing  $C_9$  in the molecule; the acid solution accompanying the nitro-product was found to be a mixture of acids, such as terephthalic, acetic, probably butyric, and some others.

All efforts to reduce nononaphthene into a corresponding paraffin by the action of hydriodic acid failed, although the author still believes such reduction possible.

**Derivatives of Nononaphthene.**—The *Monochloride*,  $C_9H_7Cl$ , is formed by passing dry chlorine into the vapour of nononaphthene from Caucasian naphtha. At least two isomeric varieties are obtained:—

1. Boiling at  $185^\circ$ — $188^\circ$ , sp. gr.  $D_{20}^{20}$  = 0.9339.
2. Boiling at  $182^\circ$ — $184^\circ$ , sp. gr.  $D_{20}^{20}$  = 0.9304.

Probably it contains the admixture of corresponding paraffin chlorides boiling at the same temperature. They are oily liquids with a smell like camphor, and a very sharp taste. They are also lighter than water, and can be distilled under the ordinary pressure.

On heating with caustic alkali, hydrochloric acid is obtained, and  $C_9H_{16}$  with products of decomposition. Water produces a similar effect, partly exchanging hydroxyl (HO) for chlorine, and giving an alcoholic compound. Bromine in presence of aluminium bromide acts upon the chloride and gives a crystalline substance, probably tribromopseudo-cumene.

**Dichloride**,  $C_9H_6Cl_2$ , is capable of yielding 2 mols. of hydrochloric acid, and becoming  $C_9H_{14}$ —the first hydrocarbon of the terpene series obtained from naphthenes.

**Iodononaphthene**,  $C_9H_7I$ , is formed on heating the chloride ( $185.5^\circ$ ) with hydriodic acid. It is a heavy colourless liquid, almost insoluble in water, boiling at  $108^\circ$ — $111^\circ$ , and could not be solidified. Sp. gr.  $D_{20}^{20}$  = 1.4041.

**Nononaphthyl Acetate**,  $C_9H_7CO_2CH_3$ , formed on heating silver acetate with the chloride of the hydrocarbon; boils at  $208.5^\circ$ , sp. gr.  $D_{20}^{20}$  = 0.9200. Along with it is formed a considerable amount of unsaturated hydrocarbon,  $C_9H_{16}$ , boiling below  $120^\circ$ .

**Nononaphthyl Alcohol**,  $C_9H_{17}OH$ , is obtained by the action of silver monoxide on the acetate of the hydrocarbon. Its agreeable smell resembles that of the tertiary alcohols; it boils at  $189^\circ$ — $192^\circ$  and its sp. gr.  $D_{20}^{20}$  = 0.8972. Some other alcohols homologous with this one had been already obtained, such as  $C_8H_{15}OH$ , by Pautinsky and Jakevkin, and  $C_{11}H_{23}OH$  by Markovikov and Ogloblin. It is scarcely soluble in water, but is very hygroscopic and forms naphthylates with sodium, barium and, probably, silver.

**Nononaphthyl Ether**,  $(C_9H_{17})_2O$ , is obtained by the action of silver monoxide on the iodide; it boils at  $294^\circ$ — $297^\circ$ . It is a thick liquid, and can be distilled over metallic sodium; sp. gr.  $D_{20}^{20}$  = 0.8662 (lower than that of alcohol).

**Nononaphthylene**,  $C_9H_{16}$ , was first obtained and named by Markovikov. It is formed from the chloride of nononaphthene by the action of silver acetate. There are at least two isomeric hydrocarbons: 1, boiling at  $135^\circ$ — $137^\circ$ , sp. gr. =  $D_{20}^{20}$  = 0.7962; and 2, boiling at  $131^\circ$ — $133^\circ$ ; colourless liquids with a smell similar to that of turpentine and quite different from that of naphthenes; it resembles in its physical properties campholene from campholic acid. It combines readily with 2 atoms of bromine, showing a marked increase of temperature, but the resulting bromide readily yields hydrobromic acid. On standing with water in presence of a small amount of sulphuric acid it forms nononaphthyl alcohol. In strong sulphuric acid it dissolves completely. On heating with hydriodic acid in sealed tubes it forms ordinary nononaphthene. In presence of suitable oxidising agents it gives the same products as the above alcohol, i.e., a mixture of acids containing less than  $C_9$  in a molecule, such as acetic, succinic, and others. In short, these hydrocarbons of the general formula,  $C_nH_{2n-2}$ , of the naphthene series stand in the same relation to naphthenes as olefines stand to paraffins in the normal series.—N. W. T.

#### *The Occurrence of Phenol and Pyridine in Brown Coal-Tar.* T. Rosenthal. Chem. Zeit. 14, 870.

ALTHOUGH it is stated in the text-books that phenol is a constituent of brown coal-tar, yet the preparation of the pure crystalline substance therefrom appears not to have been hitherto effected. It may be obtained by the following process: Light oil or first runnings is shaken with 0.5 per cent. of its weight of caustic soda dissolved in water to make a 10 per cent. solution. After removing traces of oil clinging to the alkaline solution of phenol thus obtained, by blowing steam through it, the tar acids are liberated by the addition of dilute sulphuric acid, and purified by repeated distillation, towards the end over chromic acid, and finally over white lead. A considerable proportion distils below  $190^\circ C.$ , and from it crystals of phenol separate on standing, which melt at  $36^\circ C.$  after recrystallisation. The purity of the substance was proved by the analysis of the derivatives tribromophenol and the potassium salt of paraphenol-sulphonic acid.

Pyridine has not previously been obtained from this source; it is present in very small quantities. Six thousand kilos. of light oil shaken out with 50 kilos. of sulphuric acid of 20 per cent. strength gave about 10 kilos. of the crude base. By repeated rectification, using a four-bulb dephlegmator, 200 grms. boiling below  $120^\circ C.$  were obtained. Further purification by means of the ferrocyanide gave a specimen of pyridine boiling at  $115^\circ$ — $117^\circ$ , the purity of which was verified by analysis of its double salts with platinum and gold chlorides.—B. B.

#### *Notes on the Manufacture of Mineral Lubricating Oil.* A. Veith. Chem. Zeit. 14, 901—902 and 942—943.

THE importance of the mineral lubricating oil industry may be gauged by the fact that there are now prepared from the crude material oils of all grades, and adapted for every kind of machinery, and capable, if judiciously chosen, of completely replacing the more costly fatty oils hitherto in use. The mere distillation of the less volatile portion of

petroleum failed to yield a thoroughly satisfactory product, as a certain amount of "cracking" took place in consequence of the contact of the vapour of the oil with the heated sides of the still causing the formation of evil-smelling products of low viscosity. About 1870 the use of superheated steam was adopted, improvement in the quality of the material at once taking place, and fairly good results were obtained by even the early and more primitive appliances, though the oil still left a good deal to be desired. The cause of the efficacy of superheated steam seems to be its mechanical action separating and protecting the particles of oil vapour from local overheating. Consideration of the best material for the retort, temperature and means of condensation led to the evolution of modern plant. Turning first to the still proper, it was found that cast iron when of the necessary thickness was too slow in heating and cooling, and too poor a heat conductor for economical use, and wrought iron was therefore substituted; vertical retorts worked badly owing to the considerable condensation that took place on their upper parts, and the consequent tendency of the oil to trickle back down the heated walls of the still, and retorts of elliptical section set horizontally with a wide tube leading quickly away and downwards for the conveyance of the evolved vapours were adopted in place of them. The form of superheater varies in different factories, but speaking broadly the question of the best material for the tubes is the most weighty, and may be answered thus: Cast iron does not readily oxidise and burn away, but is liable to crack with sudden changes of temperature; the reverse is true of wrought iron; in consequence of these properties the author recommends the use of wrought iron tubes protected with cast iron cast round them as best meeting the requirements of the case. The temperature of the superheated steam is  $220^{\circ}$ – $270^{\circ}$  C., and empirical rules rather than the pyrometer are relied on for obtaining and maintaining it. The condensation of the oil is effected in a large worm with thin walls cooled in air instead of water, and having openings at intervals along its length; by the use of this device, instead of obtaining a single product whose viscosity and flashing point are not particularly high, several different oils are got, the heaviest portion condensing in the portion of the worm nearest the still, and the lighter products after further condensation in the order of their volatility. The lightest of all is condensed in a second worm cooled by water and collects together with the water from the superheated steam. The automatic separation of most of the oil from the water with which it is associated saves time, inasmuch as the oil can be at once refined without waiting for separation by ordinary means.

—B. B.

*The Distillation of Rosin in Vacuo.* C. A. Bischoff and O. Nastvogel. Ber. 23, 1919–1923.

ROsin consists chiefly of a mixture of a hydrocarbon  $C_{20}H_{32}$ , which is probably the same as that described by Deville and Ribau under the name of "colophene," and of an acid  $C_{20}H_{32}O_2$  isomeric with sylvic and pimaric acids, which may be called "isosylvic acid."

The products obtained from the distillation of rosin under atmospheric pressure are the result of complete decomposition occurring during the process, the average yield being as follows:—60 grms. rosin distilled:—

	Grms.	Boiling Point.
Spirit (and trace of water).....	8.0	$108^{\circ}$ – $200^{\circ}$
Viscid, clear oil .....	9.8	$206^{\circ}$ – $360^{\circ}$
Heavy oil.....	16.6	
Pitch (insoluble in alcohol).....	19.0	
Uncondensable gas.....	6.6	

Both the spirit and the oil are readily soluble in caustic soda, from which solution hydrochloric acid throws down white precipitates.

For the distillation in vacuo, an initial pressure of 50 mm. was employed, which owing to the evolution of gas quickly rose to 110 mm. From 24.3 grms. of rosin distilled, 19.8 grms. of distillate were obtained, together with 1.4 grm. of pitch insoluble in alcohol, and 3.1 grms. of gas. The distillate was almost insoluble in cold caustic

soda, but soluble in hot alcohol, being reprecipitated on the addition of water; it was also found to be insoluble in ether and benzene. It was then dried and distilled in air, the first drops coming over above  $360^{\circ}$ , whereas ordinary rosin oil starts at  $108^{\circ}$ . The distillate was collected in three portions, no decomposition being noticeable during the process. The middle fraction was the largest, and consisted of a tacky oil with a smell resembling turpentine; analysed it gave results coinciding with a formula  $C_{44}H_{66}O_3$ . This formula contains two molecules of water less than the so-called "abietic acid,"  $C_{44}H_{64}O_3$ . The substance had neither the character of an acid, aldehyde, ketone, or alcohol, but appeared to be an anhydride; by repeated distillations in vacuo it seemed to be split up into several components.

On repeating the experiments with rosin that had been kept over concentrated sulphuric acid and phosphoric anhydride for three weeks, it was found that water was always obtained on distillation, showing that it must be chemically combined in the rosin. This was then distilled at a pressure of 30 mm., and found to melt at  $69^{\circ}$ – $130^{\circ}$ . First distillation:—

	Per Cent.
Water and spirit.....	11.4
Oil.....	75.2
Pitch.....	6.9
Gas and loss.....	6.6

On heating to  $150^{\circ}$  to remove water, the oil lost 1.5 per cent. Second distillation:—

	Per Cent.
Up to $200^{\circ}$ .....	5
Up to $250^{\circ}$ .....	65
Up to $300^{\circ}$ .....	13
Up to decomposition.....	9
Pitch.....	5
Gas and loss.....	3
	100

The second portion ( $200^{\circ}$ – $250^{\circ}$ ) was redistilled and gave:—

	Per Cent.
Up to $200^{\circ}$ .....	6.4
Up to $216^{\circ}$ .....	19.0
Up to $219^{\circ}$ .....	9.3
Up to $223^{\circ}$ .....	5.4
Up to $225^{\circ}$ .....	6.3
Up to $245^{\circ}$ .....	8.2
$248^{\circ}$ – $250^{\circ}$ .....	35.0
Loss.....	10.4

The fraction  $248^{\circ}$ – $250^{\circ}$  rectified 10 times and analysed, gave results coinciding with the formula  $C_{40}H_{58}O_3$ , and is probably isosylvic anhydride. It solidified in a short time to a hard colourless mass of very fine crystals, easily soluble in alcohol and ether. The alcoholic solution rotated the ray of polarised light to the right  $[\alpha]_D = +63^{\circ}$  (sylvic acid gives  $[\alpha]_D = -53^{\circ}$ ; Haller, Ber. 18, 2166; this Journal, 1886, 25). By repeated solution in caustic potash and precipitation with dilute acetic acid, a white powder was obtained, melting between  $60.5^{\circ}$  and  $62.5^{\circ}$ , and soluble in weak ammonia, which on analysis gave results coinciding with a formula  $C_{20}H_{30}O_2$ . This acid is therefore isomeric with sylvic acid. In common with its isomer, it has the property of giving a red-violet colour on the addition of concentrated sulphuric acid to its solution in acetic anhydride, which is removed by water, colourless flocks separating out.

The next largest fraction distilled at  $216^{\circ}$ – $219^{\circ}$ , and consisted chiefly of hydrocarbons of the formula  $C_{10}H_{16}$ ; by repeated distillation it can be so far purified as only to contain about 1 per cent. of oxygen.

"Colophene" boils at  $318^{\circ}$ – $320^{\circ}$ , in vacuo at  $218^{\circ}$ – $220^{\circ}$ , apparently showing that this substance is a diterpene,  $C_{20}H_{32}$ .

It is specially to be remarked that "colophene" and isosylvic acid, the two chief constituents of rosin, the latter of which is possibly formed during the distillation from the original sylvic acid, are very similar in their composition.

—R. H. L.

*Contributions to the Study of the most Volatile Portion of Coal Tar.* J. Biehinger. Jour. f. Gasbeleuchtung, **33**, 341—345 and 358—369; also Dingl. Polyt. J. **276**, 78 *et seq.*

THE contradictory statements published as to the components of this portion of tar, are probably due to the fact that the investigations have been conducted on products which have been previously submitted to the action of acids and alkalis. To avoid errors introduced in this way, the author's experiments were carried out on that portion of 90 per cent. English benzene which boils between  $30^{\circ}$  and  $80^{\circ}$ , and afterwards on some specially prepared fractions boiling, No. 1 between  $50^{\circ}$  and  $60^{\circ}$ , and No. 2 between  $60^{\circ}$  and  $70^{\circ}$ , sent him by Dr. Häussermann from the chemical works at Griessheim.

In common with previous experimenters, the author found that fractional distillation through Le Bel-Henninger's tubes, or through a six-bulbed apparatus filled with glass beads, even when repeated several times, was not competent to split up the substance satisfactorily, decompositions appearing to occur which altered the boiling points.

By allowing the crude products to settle, or better by fractionation, they were found to separate into two distinct layers, the lower consisting chiefly of carbon bisulphide. The separation, however, was not sufficiently complete to allow of exact results being obtained. The only other methods available were to remove one well-known constituent at a time, or to separate the substances into groups.

To test for carbon bisulphide, the first runnings, or impure benzene, are treated with alcoholic ammonia, and allowed to rest for a time. If the amount present is not extremely small, ammonium sulphocyanide separates out in crystals, which may be extracted with water, and after acidification identified by ferrie chloride. Or by adding caustic potash dissolved in absolute alcohol, the carbon bisulphide may be converted into potassium xanthate, which may be dissolved out by water and identified by the formation of the yellow cuprous xanthate on the addition of copper sulphate solution.

The presence of carbon bisulphide is a great hindrance to the investigation of the first runnings, for it has the property, in common with several other substances present, of not being changed by concentrated acids and alkalis, for which reason most of the benzene of commerce is contaminated with it.

The following methods of estimating carbon bisulphide may be employed:—

*1st. By means of triethylphosphine.* The carbon bisulphide is dissolved in absolute ether, and an ethereal solution of triethylphosphine added in excess; after a time the liquid becomes red, and deposits small needle-like crystals; after the colour has disappeared, the whole is thrown on to a tared filter, the mother-liquor being used to rinse out the vessel, the crystals finally washed with pure ether, dried in *vacuo* and weighed. Working with pure bisulphide, the author found the results a good deal too low.

*2nd. Estimation as a salt of xanthic acid:—*

The solution containing the carbon bisulphide is added drop by drop to a concentrated solution of caustic potash in absolute alcohol, with constant stirring. The mass of crystals should be thoroughly broken up, and the whole warmed slightly. The xanthate so obtained may be analysed volumetrically either by means of a copper solution or by iodine. For the first process, the copper sulphate solution should be of decimal strength, each cc. of which represents 0.03202 grm. xanthate or 0.0152 grm.  $\text{CS}_2$ . The best method is that of Macagno, who neutralises the solution of the potassium xanthate with acetic acid, and titrates with the copper solution as long as any turbidity is apparent, the end reaction being obtained by testing a drop of the liquid with potassium ferrocyanide. As the cuprous precipitate masks the colour of the copper ferrocyanide, this may be done by putting one drop of the liquid on to a double thickness of filter paper and adding the ferrocyanide to the solution that passes through to the lower layer. Experiments tried by the author on pure  $\text{CS}_2$  gave results 0.54 per cent. too low by this process. Tried on the two crude products mentioned above:—No. 1 gave 19.8 per

cent.  $\text{CS}_2$ , and No. 2, 19.17 per cent.  $\text{CS}_2$ . These figures show that it is impossible to concentrate the carbon bisulphide into one fraction by distillation, and strengthen the idea that it exists in the crude liquid in loose combination with some other substance, which compound is only broken up gradually by boiling.

*The third method of estimation, by means of standard iodine,* depends on the formation of ethyldisulphocarbonic sulphide as discovered by Desains (Annalen [3], **20**, 496). The solution containing the potassium xanthate is exactly neutralised with acetic acid, starch added, and  $\frac{N}{20}$  iodine run in till the blue colour appears. The author's experiments show that this method is far less exact than the former.

The author has tried to estimate carbon bisulphide by converting it into ammonium sulphocyanide by means of alcoholic ammonia: after  $3\frac{1}{2}$  days only, 78.3 per cent. was found to be changed into the sulphocyanide.

He also tried converting the bisulphide into diphenylsulphocarbamide, by warming it with aniline and caustic potash: after several days' heating only 61 per cent. was changed.

To remove the carbon bisulphide from the light "benzole," the best method is to proceed as follows:—1,000 to 1,200 grms. of the fraction are treated with from one-fifth to one-sixth their weight of ordinary alcohol, and to avoid loss of the most volatile portions, put into a freezing mixture. Dry ammonia vapour is then passed through the mixture, care being taken that the leading tube does not become blocked. After a time crystals begin to separate out, and the liquid divides into two distinct layers, the lower consisting of alcohol and water, with the various products of the action on the bisulphide in solution, and containing a small amount of the hydrocarbons, which may be removed by the addition of more water. If the whole liquid is filled with separated solid matter it may be strained through linen, when the division can be easily seen. The top layer should be treated with more alcohol and ammonia passed again, the operation being repeated till crystals no longer separate. The residual liquid is then thoroughly washed with water, dried by means of calcium chloride and distilled.

By this treatment the specific gravity of the fraction No. 1 fell from .8148 to .7401.

The author hopes to continue his researches on the product freed from carbon bisulphide.—F. H. L.

## PATENT.

*Improvements in the Distillation of Mineral Oils and like Products, and Apparatus for that Purpose.* J. Dewar, Cambridge, and B. Redwood, Finchley. Eng. Pat. 13,016, August 17, 1889. *Sd.*

THIS is an apparatus for the distillation of mineral oils under reduced pressure, a current of air or inert gas being passed continuously through the boiling liquid. It consists of a retort, condenser and receiver. The latter is connected with an air-pump and with a second receiver placed beneath it which can also be put into communication with the air pump or with the external air. When a sufficient quantity of the distillate has collected in the first receiver, the two receivers are put into communication and the liquid runs from the first into the second. The communication is now cut off, air is admitted to the second receiver and the distillate run off by a tap at the bottom. A third receiver communicates with the bottom of the retort and serves to withdraw the high-boiling residue. The retort is also furnished with a tube which reaches nearly to the bottom of the retort and serves to admit air or other gas which bubbles through the liquid and assists the distillation.—H. K. T.

## IV.—COLOURING MATTERS AND DYES.

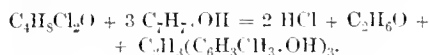
On a Violet Colouring Matter from Illuminating Gas or from the Residues of Gas Purifiers. Jour. f. Gasbeleuchtung, **33**, 304—306.

See under II., page 923.

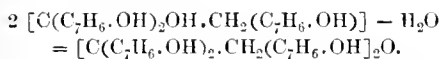
*The Condensation of Dichlorether with the Cresols.*

C. Brückner. Annalen, **257**, 322—330.

*Ethenyltrieresol.*—The cresols react with dichlorether similarly to the phenols (Ann. **243**, 151, et seq.), ethenyltrieresols being formed according to the equation—



These compounds are prepared by warming a mixture of dichlorether (1 mol.) with cresol (3 mols.) and dropping the solution of the product in glacial acetic acid into a large excess of water. They are white uncrystallisable powders reddened when damp by the air; they are insoluble in water, chloroform, or light petroleum, but dissolve readily in alcohol, ether, glacial acetic acid, ethyl acetate, &c.; on evaporating the solutions they are left in resinous masses. The ortho-compound is soluble in dilute solutions of potash, the meta- and para-compounds only in warm concentrated solutions; they are all reprecipitated unchanged by acids, even by carbonic acid. They do not melt when heated, but form soft resinous masses which become brittle and glassy on cooling. The *o*-compound undergoes this change at 85°, the *m*-compound at 90°, and the *p*-compound only at 100°; at 130° decomposition ensues. The triacetates are pale yellow amorphous powders insoluble in water and in alkalis. When the ethenyltrieresols are boiled for six hours with a little more than a molecular proportion of ferric chloride, and the product is poured into an excess of water, violet-brown flocculent precipitates separate, the alcoholic solution of which directly dye wool and silk violet-brown. The precipitates are purified by repeatedly dissolving them in glacial acetic acid and precipitating with water. By desiccation in vacuo they are obtained as dark powders which may be heated without change to 120°, but decompose at 200°. They are soluble in alcohol, ether, glacial acetic acid, ethyl acetate, acetone, and alkalis, but not in carbon bisulphide, benzene, &c. From aqueous alkaline solutions they are reprecipitated by acids, even by carbonic acid. The constitution of these dyestuffs is slightly different from that of the analogous phenol, naphthol, resorcinol, &c. derivatives, 2 mols. of the corresponding phenol first formed condensing with elimination of water—



The acetates of these dyestuffs are yellow powders which decompose at 100°, the *o*- and *p*-compounds have the composition  $[C(C_7H_6.OC_2H_3O)_2.CH_2(C_7H_6.OC_2H_3O)]_2$ , but the *m*-compound appears to be a mixture of several substances.

—S. B. A. A.

*Hydrazobenzenedisulphonic Acid.* H. Limpricht.

Ber. **23**, 1052—1057.

THE compound formerly known as hydrazobenzenedisulphonic acid has been proved to be benzidinedisulphonic acid. The author has, however, succeeded in preparing the proper hydrazobenzenedisulphonic acid, by heating a mixture of lead azobenzenedisulphonate and water on a water-bath and passing sulphuretted hydrogen through the mixture. On evaporation *m*-amidobenzene sulphonic acid and benzidinedisulphonic acid were precipitated, and finally white needles easily soluble in water were obtained, forming the desired compound. It is a strong reducing agent; mineral acids convert it into benzidinesulphonic

acid and its alkaline solution is changed on evaporation into azobenzenedisulphonic acid.

The barium salt—



forms white or light yellow crystals.

The potash salt,  $C_6H_4(SO_3K)NH.NH(SO_3K)C_6H_4$ , also forms light yellow crystals.—A. L.

*Researches on Azo Derivatives of Phenyl-β-Naphthylamine.* T. Zincke. Ber. **23**, 1314—1325.

BENZENE-AZOPHENYL-β-NAPHTHYLAMINE yields on oxidation with chromic acid the chromic salt of a well characterised ammonium base,  $C_{22}H_{16}N_4.OH$ , which, on careful reduction reproduces benzene-azophenyl-β-naphthylamine. If the aqueous solution of this compound be heated on the water-bath a red resinous matter is gradually precipitated, whilst the bitter taste of the solution decreases in intensity. On treatment with alcohol a base is dissolved from the resin, and the residue consists of benzene-azo-β-naphthylphenylamine. The same reactions take place if the chloride of the base is heated with caustic potash. The alcoholic solution precipitates on dilution with water a body of the formula  $C_{22}H_{16}N_4O$ , which, after crystallisation from benzene, forms yellowish needles, melting at 215°—217° C. It is easily soluble in alcohol, glacial acetic acid, and benzene, and shows strong greenish fluorescence especially in dilute solutions. It forms salts. The author discusses at great length the constitution of the base obtained from the azo compound, without however arriving at a final conclusion as to its constitution.

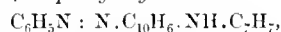
—A. L.

*Azo Derivatives of Secondary-β-Naphthylamine Derivatives, and their Oxidation Products.* P. Matthes. Ber. **23**, 1325—1334.

*p-Toluene-azo-β-naphthylphenylamine*, prepared from β-naphthylphenylamine and *p*-diazotoluene chloride, forms, after crystallisation from alcohol, dark red needles of a beautiful metallic lustre, easily soluble in benzene, less in alcohol and glacial acetic acid. Melting point = 120° C.

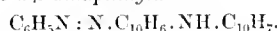
*Ammonium Base from p-Toluene-azo-β-naphthylphenylamine.*—The azo-compound is dissolved in 10 times its weight of hot glacial acetic acid. The solution is then cooled to 40°—45° C., and a hot concentrated solution of potassium bichromate is gradually added until the liquid becomes brown and yields, on dilution with water, a yellow precipitate, the chromic salt of the new base. The nitrate, picrate, the platinum, mercury, and tin double salts are described.

*Benzene-azo-β-naphthyltolylamine*—



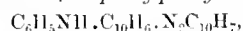
forms long dark red needles easily soluble in hot glacial acetic acid and benzene, with more difficulty in alcohol and ether, melting at 152° C. It yields on oxidation an ammonium base, of which several salts are described.

*Benzene-azo-α-β-dinaphthylamine*—

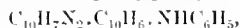


Two isomerides are formed. The β-compound obtained by crystallisation from benzene forms red needles, melting at 167° C. On boiling with alcohol in hydrochloric acid, Witt's asymmetrical azine was obtained, melting at 283°—284° C. On carefully oxidising the azo-compound, the ammonium base was obtained, of which several salts are described.

*α-Naphthalene-azo-β-naphthylphenylamine*—



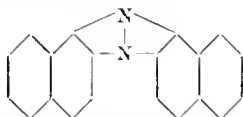
forms dark red needles, melting at 140° C., with difficulty soluble in alcohol, more easily in benzene and glacial acetic acid. The ammonium base is prepared by oxidising the azo-compound with chromic acid.

*β-Naphthalene-azo-β-naphthylphenylamine—*

forms long brick-red needles, melting at 154°–155° C. On oxidation with chromic acid the ammonium base is obtained.

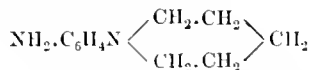
*Benzene-azo-β-β-dinaphthylamine—*

is only formed if the solutions are kept very cold and if the diazo solution does not contain an excess of free acid. Red needles, melting at 139° C.

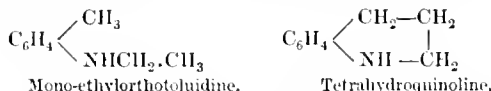
*Symmetrical-β-β-naphthazine—*

is obtained if a strongly acid solution of diazobenzene chloride is added to a warm solution of β-β-dinaphthylamine. It forms yellowish needles, melting at 242°–243° C., and showing blue fluorescence in benzene and green fluorescence in acetic acid.—A. L.

*The Formation of Colouring Matters from Tetrahydroquinoline.* E. Lellmann and H. Boye. Ber. **23**, 1374–1383.

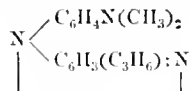
*p-AMIDOPHENYLPYPERIDINE—*

shows the same colour reactions as *p*-amidodimethylaniline. Tetrahydroquinoline can be considered as a mono-ethyl-orthotoluidine, in which the two alkyl groups are joined together—



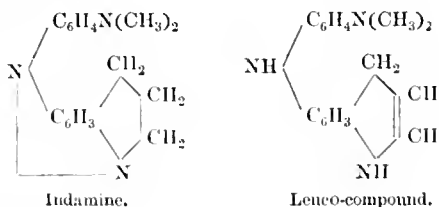
This supposition originated the idea of submitting the tetrahydroquinoline to some tests which, when applied to dimethylaniline, produced colouring matters; the result proved the theory to be correct.

If a cold solution of equal molecular proportions of tetrahydroquinoline hydrochloride and amidodimethylaniline hydrochloride are oxidised with the theoretical quantity of potassium bichromate, an intensely green solution is obtained, which contains an indamine of the formula—

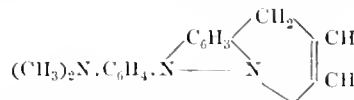


the same product is formed by gently heating aqueous solutions of nitrosodimethylaniline hydrochloride and tetrahydroquinoline hydrochloride. This indamine, which shows all the characteristic reactions with acids and alkalis has, however, some characteristic properties, being converted by intramolecular reduction into a leuco base. If its solution is allowed to stand or is gently heated, the green colour disappears gradually, and after 1–2 hours a colourless solution is formed.

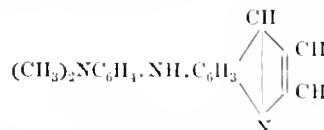
The authors explain this reaction by the following formulae:—



On addition of potassium bichromate to a solution of the leuco base the solution is again coloured green, but of a more brownish tint than the original solution. It probably contains an indamine of the following constitution:—

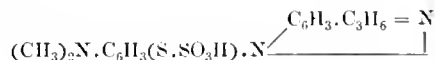


But its solution is again soon decolourised, and probably a body formed of the constitution—

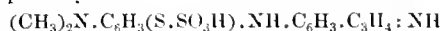


If molecular quantities of amidodimethylanilinesulphonic acid and tetrahydroquinoline hydrochloride are oxidised with potassium bichromate, the indamine of the sulphonic acid settles down in small green needles of metallic lustre, with difficulty soluble in water. They cannot be recrystallised without decomposition. The reaction takes place according to the equation—

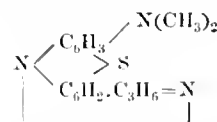
$\text{C}_9\text{H}_{11}\text{N} + \text{C}_8\text{H}_{12}\text{O}_3\text{N}_2\text{S}_2 + \text{O}_5 = \text{C}_{17}\text{H}_{19}\text{O}_3\text{S}_2\text{N}_3 + 2 \text{H}_2\text{O}$ ,  
and the new compound, the indamine of *Tetrahydroquinolinedimethylanilinesulphonic acid* has probably the constitution—



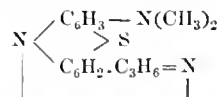
On standing this compound is converted into a leuco base, probably—



On reducing the tetrahydro-indamine with zinc dust and hydrochloric acid, sulphuretted hydrogen is evolved, and the solution precipitates on addition of ferric chloride an abundant quantity of a blue colouring matter, which, according to its formation and properties, belongs to the class of the thionines and is analogous to Methylene blue. Its formula ought to be—



Tetramethylindaminesulphonic acid produces on boiling with water leucomethylene blue. When the leuco base of the dihydro-indamine is treated in the same manner for a day the sulphite of a new leuco base is formed, which on oxidation with ferric chloride produces a new blue colouring matter—



differing in shade and intensity from the Tetrahydro blue.

Kairolin or methyltetrahydroquinoline is also capable of forming indamines and blue colouring matters.—A. L.

*On some Piperidine Bases.* E. Lellmann and H. Büttner. Ber. **23**, 1383–1388.

AROMATIC halogen compounds act on piperidine at a high temperature; bromobenzene, for instance, produces with piperidine at 250° C. *Phenylpiperidine*. The authors have prepared—

*Tertiary-α-naphthylpiperidine*,  $\text{C}_{10}\text{H}_7\text{NC}_5\text{H}_{10}$ , oil, boiling at 185°–190° C.



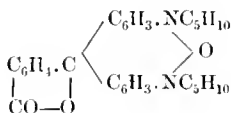
*Tertiary-β-naphthylpiperidine*,  $C_{10}H_7NC_5H_{10}$ , crystals, melting at  $57^{\circ}$ — $58^{\circ}$  C.

*Tertiary anthracylpiperidine*,  $C_{14}H_9NC_5H_{10}$ , yellow needles.

*Tertiary phenanthrylpiperidine*,  $C_{14}H_9NC_5H_{10}$ , crystals, melting at  $113^{\circ}$  C.

*p-Nitro-α-naphthylpiperidine*,  $NO_2C_{10}H_6NC_5H_{10}$ , yellow needles melting at  $17^{\circ}$  C.

#### Piperidylhydramine—



obtained by acting with 6 molecules of piperidine on fluorescein chloride at  $220^{\circ}$  C. The contents of the tube were heated with alcohol and hydrochloric acid. The solution was evaporated to dryness and the residue dissolved in dilute hydrochloric acid. Alkalis precipitate the rhodamine base as a violet floccular precipitate, easily soluble in alcohol and very stable towards alkalis.—A. L.

*On Paramidotriphenylcarbinol*. A. Baeyer and R. Löhr. Ber. 23, 1621—1628.

*Paranitrotriphenylmethane*,  $C_{19}H_{15}NO_2$ , is obtained by allowing a mixture of 5 grms. of *p*-nitrobenzaldehyde, 20 grms. of benzene, and 20 grms. of sulphuric acid to stand for 24 hours. The benzene solution is then washed with sodium carbonate, sodium bisulphite, and water; the excess of benzene is evaporated, and the condensation product is obtained as a thick oil, crystallising after some time. After repeated crystallisations from petroleum spirit it is obtained in beautiful colourless crystals, melting at  $93^{\circ}$  C. On treatment with fuming nitric acid it is converted into *Triparanitrotriphenylmethane*.

*Paranitrotriphenylcarbinol* is prepared from the hydrocarbon by oxidation with chromic acid. The hydrocarbon is dissolved in glacial acetic acid, and 4—5 times its weight of chromic acid are added, until a sample poured into water separates in crystals. The hydrocarbon is removed by petroleum spirit and the carbinol purified by dissolving it in glacial acetic acid by precipitation with water. It melts at  $136^{\circ}$  C.

*Paramidotriphenylmethane* was obtained from the nitro-compound by heating it on the water-bath for several hours with tin and hydrochloric acid until the solution became colourless. The free base, when crystallised from benzene, contains benzene of crystallisation, and forms large prisms of the formula  $C_{19}H_{15}NH_2 + C_6H_6$ . The body is soluble in ether, alcohol, and petroleum spirit, and melts at  $83^{\circ}$ — $84^{\circ}$  C.

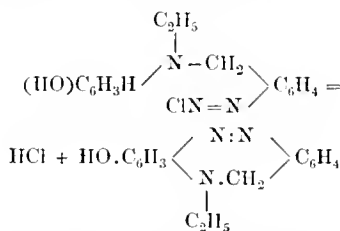
Its *Acetyl-compound*,  $(C_6H_5)_2CHC_6H_4NH(C_2H_5O)$ , forms needles or prisms melting at  $157^{\circ}$  C. This compound yields, when carefully oxidised with chromic acid in a solution of glacial acetic acid, *Acetylparamidotriphenylcarbinol*  $(C_6H_5)_2C(OH)C_6H_4NH(C_2H_5O)$ , soluble in glacial acetic acid and alcohol, and melting at  $176^{\circ}$  C. On saponification of this product with hot dilute sulphuric acid the sulphate of *paramidotriphenylcarbinol* is obtained as a dark red solution. The hydrochloride forms also a red salt, crystallising in long red needles. The free base is colourless, soluble in ether, alcohol, and benzene, and crystallising from toluene or a mixture of ether and petroleum spirit in small colourless crystals melting at  $116^{\circ}$  C.

On reducing *paranitrotriphenylcarbinol*, *amidobenzophenone*,  $C_6H_5COC_6H_4NH_2$ , melting at  $123^{\circ}$ — $124^{\circ}$  C., is formed.

*p-Monamidotriphenylcarbinol* forms red coloured salts, but they have no affinity for animal fibre.—A. L.

*On a Molecular Interchange between a Diazo and a Phenolic Group*. E. Lellmann and H. Boye. Ber. 23, 1781—1783.

ALCOHOLIC solutions of 1 mol. of orthonitrobenzylchloride and 2 mol. of ethylmeta-amidophenol are heated on the water-bath, the alcohol distilled off, and the dark, viscous oil which remains extracted repeatedly with water, to remove the hydrochloride of ethylamidophenol. On treating the residue with dilute hydrochloric acid, it gradually changes to a light grey powder, which the authors assume to be the hydrochloride of orthonitrobenzylethylmeta-amidophenol. This substance was reduced with stannous chloride, and after diluting the solution was poured into ammonium sulphide, when the corresponding amido compound separated out. This was dissolved in dilute hydrochloric acid and treated with 1 mol. of sodium nitrite. A brown colour was produced, which after some time was precipitated from its solution with sodium acetate. Its formula is  $C_{15}H_{15}ON_3$ , and the formation of the dye may be interpreted by assuming that the diazo-chloride of orthoamidobenzylethylmeta-amidophenol is first formed, which then is converted into the azo-dye by the elimination of hydrochloric acid.



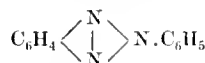
The new dye, which can also be regarded as azobenzyl-ethylamidophenol, dyes silk a yellowish-brown shade which is hardly altered by acids or alkalis.—A. R.

*Orthonitrodiphenylamine and some of its Derivatives*. M. Schöpf. Ber. 23, 1839—1844.

THE best method of preparing this body is to act on orthobromonitrobenzene with an excess of aniline. It is impossible to prevent the formation of coloured by-products. To obtain a fair yield, the substances should be allowed to react for three hours, and then be poured into water. After acidifying with hydrochloric acid, the bromonitrobenzene which has not been attacked is distilled over with steam. On now cooling, orthonitrodiphenylamine separates out and may be crystallised from weak alcohol. Orange-coloured leaflets are obtained, melting at  $75^{\circ}$ . A description of the preparation of orthonitrodiphenylparatolylamine is also given. *Phenylorthophenylenediamine* is readily prepared by heating nitrodiphenylamine to  $120^{\circ}$  with alcoholic sulphide of ammonium. After evaporating most of the alcohol, the residue is treated with hydrochloric acid, boiled to separate out the sulphur, then cleared with some animal charcoal and the base precipitated with ammonia. After recrystallising from alcohol with addition of animal charcoal, needles melting at  $79^{\circ}$ — $80^{\circ}$  are obtained. The amine is soluble in much hot water. A solution of the hydrochloride is coloured deep red by ferric chloride, and a brown precipitate is produced, which, if the solution be dilute, is deposited in the shape of brown needles, with green reflex; this body probably belongs to the group of the azines.

On adding sodium nitrate to a solution of phenylphenylenediamine in dilute hydrochloric acid, *phenylazimidobenzene* is obtained. It is recrystallised from weak alcohol, when reddish needles, melting at  $89^{\circ}$ — $90^{\circ}$ , separate out.

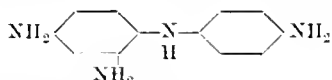
A by-product obtained in the manufacture of amidoazobenzene has been described by Gattermann and Wichmann, which yields aniline and orthophenylenediamine on reduction; they have therefore assigned to that substance the formula—



which would be identical with that of phenylazimidobenzene. Now, the latest researches of Zincke and Campbell point to the existence of two series of these imido-compounds. That class which they distinguish by the term pseudo-azimides is produced by oxidation of orthoamidoazo bodies, the other azimides result from the action of nitrous acid on orthodiamines. The by-product of amidoazobenzene is evidently the pseudoazimide. It differs from the one described by the author of this paper in melting 20° higher. —A. R.

*Derivatives of Diphenylamine and of Phenazine.* R. Nietzki and O. Ernst. Ber. **23**, 1852—1856.

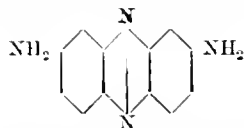
This research was undertaken with a view to preparing the simplest analogue to Toluylene-blue, viz., triamidodiphenylamine—



By the interaction of molecular proportions of dinitrochlorobenzene and paraphenylenediamine in alcoholic solution, amidodinitrodiphenylamine is obtained in quantitative yield, the equivalent quantity of sodium acetate being added after the reaction is complete. The new body is with difficulty soluble in hot alcohol, more readily in benzene or chloroform, from which it crystallises in lustrous brown leaflets melting at 177°. It dissolves in hot fairly concentrated hydrochloric acid. On cooling, the hydrochloride crystallises out. The pieric acid additive compound, as well as the mono- and diacetyl derivatives are also described.

On reducing with stannous chloride and hydrochloric acid, triamidodiphenylamine is readily formed and may be separated as the stannio-chloride by passing hydrochloric acid gas into the solution. The hydrochloride of the base is rapidly oxidised in the air, the sulphate being somewhat more stable. Triamidodiphenylamine is the leuco base of the simplest analogue to toluylene-blue. On oxidation it yields a violet-blue dye which shows the characteristic properties of the dyes of this group, but is much less stable than Toluylene-blue.

For preparing the azine, 20 grms. of the stannio-chloride compound of triamidodiphenylamine are dissolved in half a litre of water, 12 grms. of calcium carbonate added, and then 20 grms. of manganese dioxide (about 60 per cent. strength). The mixture is gradually heated on the water-bath and then boiled until it is coloured yellowish-brown and a drop on blotting paper is no longer coloured violet on the border. The solution is now filtered, a few drops of hydrochloric acid added, and the dyestuff salted out. The crude hydrochloride may be dissolved in boiling water. By adding ammonia while hot, the base crystallises on cooling in long dark yellow needles melting at 280°, readily soluble in alcohol or ether to yellow solutions which exhibit strong fluorescence. The formula of the new azine is—



Concentrated sulphuric acid dissolves the base with a green colour which on diluting changes from blue and violet to red. The pierate, platino-chloride and diacetyl derivative were also prepared. In dilute acids the base dissolves with a red colour. The salts crystallise well. They dye silk and tannin-mordanted cotton red, much yellower than the simplest safranines, more like scarlets. By alkalis the red is very easily changed to yellow.

Diamidophenazine may also be obtained by joint oxidation of para- and metaphenylenediamine or by boiling the latter with quinone-dichlorimide in alcoholic solution.

The connexion of the new azine with phenazine was proved by splitting off the amido group in the usual manner. The resulting body was in every way identical with Claus and Riz's phenazine.—A. R.

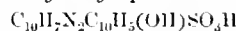
*On some Azo-Dyes from α-Naphthylamine, Dimethylaniline, and α-Hydroxynaphthoic Acid.* C. A. Bischoff. Ber. **23**, 1908—1911.

*Dimethylamidobenzene-α-azonaphthalene—*



is prepared by diazotising a salt of α-naphthylamine, filtering, and combining with dimethylaniline in alcoholic solution. The dye is purified by treatment with hot dilute acid and then with ammonia. It is quite insoluble in water. From a solution in acetone prismatic crystals were obtained which appear black in reflected and red in transmitted light. For dyeing, the azo-compound was sulphonated with acid containing 20½ per cent. anhydride. According to the degree of sulphonation, the sodium salts of the resulting acids dyed silk and wool yellow, orange, or brown.

*α-Naphthylazo-α-hydroxynaphthoic acid—*



is best prepared by allowing a cold solution of α-diazo-naphthalene chloride to flow in a thin stream into a well-cooled solution of α-hydroxynaphthoic acid in sodium carbonate. The dye which separates is washed, dried at 100°, and crystallised from glacial acetic acid. A very good yield of leaflets of gold-like lustre are obtained, melting at 198°. The new acid is sparingly soluble in boiling water and the usual solvents except glacial acetic acid and chloroform, in which it dissolves readily. Concentrated sulphuric acid gives a blue solution and green flakes appear on adding water. Alkalis give two series of salts; the neutral ones are deep red and very soluble, the acid salts dissolve with greater difficulty in cold water and they are coloured yellow. The latter do not dye unmordanted cotton. Wool was treated with warm dilute acetic acid after dyeing. It is coloured a brilliant orange yellow. The shades on silk are not fast to soap.

Below are given some of the more recently discovered yellow dyes, commencing with the greenest and passing from the orange to the brown dyes. Quinoline-yellow, Auramine, Carbazol-yellow, Primuline, Piazzine-yellow, Tartrazine; Brilliant yellow, Helianthine, Metanil-yellow; Bronstein's orange, Jacobsohn's orange I., Brown II. and III.

The most unstable to light are, Carbazol-yellow, Piazzine-yellow (from diphenyl piperazine and sulphanilic acid) and Primuline-yellow.

On moistening with concentrated hydrochloric acid, Auramine becomes light violet; Quinoline-yellow a slight brownish-yellow; Carbazol-yellow at first green then a black-violet; Primuline orange; Piazzine-yellow carmine; Tartrazine is hardly changed; Brilliant yellow turns a deep violet; Helianthine violet-blue; Metanil-yellow a deep blue-violet; Bronstein's orange is first coloured greenish then a slight brown; Jacobsohn's orange I. carmine, Brown II. and III. violet. All these tests refer to the colours on wool.

—A. R.

*On the Sulphonation of Aniline and Naphthylamine with Acid Potassium Sulphate.* C. A. Bischoff. Ber. **23**, 1912—1914.

ANILINE or α-naphthylamine and about triple the quantity of acid potassium sulphate are intimately mixed and heated in an oil-bath to 200°. The yield of sulphanilic or of naphthionic acid in the best cases did not exceed 19 per cent., calculated on the base originally used. In the case of β-naphthylamine as much as 86 per cent. sulphonie acid was obtained.

Much better yields of sulphanilic or of naphthionic acid were obtained by the so-called "baking" process. After intimately mixing base and acid sulphate in the above proportions the mass was cut up into thin cakes, these were allowed to soften at 60—80°, pressed with hot plates, and then heated for three hours to 200°; a 50 per cent. yield of the sulphonated product was obtained in this case. From α-naphthylamine α-naphthionic acid is produced, β-naphthylamine gives β₁-β₂-naphthionic acid with traces of β-naphthylamine sulphonie acid. The results therefore are identical (apart from the yield) with the ordinary process of heating the acid sulphates of the respective bases.—A. R.

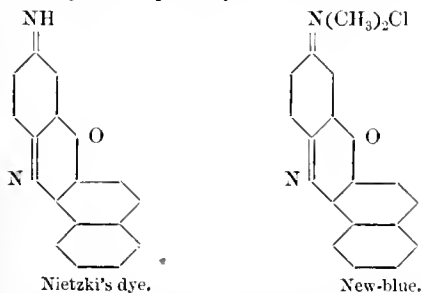
*On the Composition of Diphenyl- and Phenyl-naphthylamine-Blue.* A. Hausdörfer. Ber. **23**, 1961—1966.

THOUGH it has always been assumed that diphenylamine-blue and triphenylrosaniline-blue are identical in composition, analytical proofs have not yet been forthcoming, and this research was mainly undertaken to establish the above fact on a firmer basis. Diphenylamine-blue was prepared by Schoop's method, which consists in heating diphenylamine to 110° and gradually adding double its weight of crystallised oxalic acid. The temperature is kept for eight hours at 130°—132°. Oxalic acid is now removed from the melt with hot water, the residue extracted with alcohol and the requisite quantity of concentrated hydrochloric acid added. The hydrochloride, which separates after 12 hours' standing, is repeatedly boiled with alcohol and finally converted into the base with alcoholic caustic soda. After filtering, the hydrochloride is re-precipitated with hydrochloric acid. The dye possesses the same properties as triphenylrosaniline-blue and an analysis gave figures which correspond to the composition of the latter body. The identity of this blue with the one prepared in the above manner is now placed beyond all doubt.

*Phenyl- $\alpha$ -naphthylamine-blue* was prepared in the same manner as the previously described dye, only substituting phenyl- $\alpha$ -naphthylamine for diphenylamine. In this case, however, the blue-base is almost insoluble in alcohol. After extracting with water and alcohol the base is precipitated with alcoholic soda, dissolved in acetone and the dye precipitated with concentrated hydrochloric acid. It is with difficulty soluble in the usual solvents, and dissolves best in warm aniline. Its properties resemble those of triphenylrosaniline blue, and its formula is proved by analysis to be  $\text{CCl}(\text{C}_6\text{H}_4\text{NHC}_{10}\text{H}_7)_3$ .—A. R.

*On Cyanamides, a New Group of Dyes.* O. N. Witt. Ber. **23**, 2247—2252.

By the action of  $\beta$ -naphthol on the hydrochloride of nitrosodimethylaniline, Meldola obtained a bluish-violet cotton-dye in 1879, which has been manufactured on an industrial scale since 1883, and is known as New-blue, Fast-blue, Cotton-blue, &c. Another dye was prepared by Nietzki by the action of  $\beta$ -naphthol on quinonedichlorimide. The formulae of these two dyes most probably are—



The various products in the market known as New-blue differ considerably in their solubility, and the shade with which they dye. Apart from intentional additions, different samples of New-blue will dye from bluish-violet to violet-red shades, and this must be set down to the product being actually a mixture of different dyes. Nietzki's dye, however, prepared from quinonedichlorimide, appears to be a uniform product dyeing a magenta-red.

One property which all New-blues possess is that they are "*day blues*," i.e. with artificial light the blue caste seems to give way to a red one; in fact the products of different makers when dyed on cotton and examined in yellow light all appeared reddish-brown.

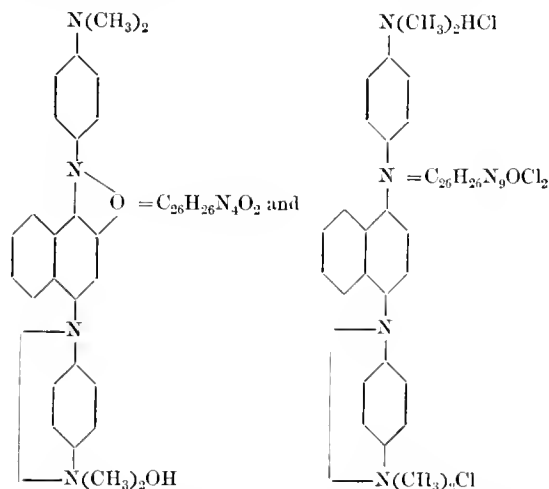
Now by the action of alkalis these dyes undergo a remarkable change. Their bases, at first readily soluble in ether, become more and more insoluble the further the action of the alkali is allowed to proceed. On dissolving the base in acid again, dyes of a pure greenish-blue shade

are obtained which resemble Methylene blue. They are night-blues, i.e. their shade is hardly changed by gas-light. The dye prepared in this manner from Meldola's violet does not differ from the one prepared in a similar manner from Nietzki's red.

The new dyes have been named *Cyanamides* by the author. The following is a method of preparation which gives good results: 56 grms.  $\beta$ -naphthol are heated on the water-bath with 74 grms. nitrosodimethylaniline hydrochloride and 160 cc. alcohol, until the violent reaction at first ensuing has ceased. 40 grms. caustic potash in 240 cc. alcohol are now added and the mixture heated until it becomes more fluid and crystals begin to separate out. The crystals are filtered off hot, washed with alcohol and water, again boiled with alcohol and filtered. The base is finally dissolved in dilute acid, filtered from a small quantity of an insoluble violet by-product and reprecipitated with ammonia. The base is best recrystallised from chloroform. In alcohol, ether, or benzene it is almost insoluble. The solutions are coloured a pure violet-red, whereas solutions of the base of New-blue are coloured reddish-brown. Concentrated acids dissolve the latter with a green colour, whilst the new base forms brownish-orange solutions. By dissolving in dilute acids, the blue coloured salts are obtained; they separate from their solutions in lustrous tarry masses, which become hard on drying. From alcoholic solutions the salts may be obtained in the crystalline form. Dilute aqueous solutions of the chloride or sulphate gelatinise on cooling. If the former be very dilute the salts are partially dissociated, basic insoluble salts separating out. One portion of the acid in the molecule is therefore bound more intimately than the other.

The Cyanamide salts are distinguished by a special absorption spectrum, even in very dilute solutions. The entire spectrum from red to green is extinguished, leaving the other part unaffected. Now since the different kinds of New-blue have practically no effect whatever on the yellow or red portion of the spectrum, this proves that even the bluer shades of certain specimens of that dye are not due to an admixture of Cyanamide.

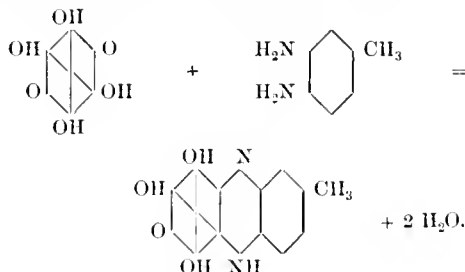
In attempting to apply the results of an analysis of the hydrochloride and of the base to the construction of formulae for these new compounds the author was led to conclude that this is only possible by assuming that 2 mols. of nitrosodimethylaniline react with 1 mol. of naphthol, whence the following probable formula for base and hydrochloride, respectively—



By oxidation with chromic acid Cyanamide-blue is converted into violet-blue dyes which show a brilliant cinnabar-red fluorescence.—A. R.

*On the Constitution of Eurhodines, Indulines and Related Dyes.* F. Kehrmann. Ber. **23**, 2446—2454.

WHILE it is not astonishing that azines are produced from orthotolylendiamine and rhodizonic acid, which is an orthoquinone, the same cannot be said with regard to the similar reaction which takes place between the diamine and tetrahydroxyquinone, doubtless a paraquinone. The author believes the reaction in the latter case is best explained by assuming that one quinone-oxygen atom and the adjoining hydroxyl-group react with the amido-groups—



It appears that other hydroxy-para-quinones are also able to react in a similar manner with orthodiamines; this however is restricted to those paraquinones that do not contain in the hydrocarbon nucleus radicles with a high molecular weight. Thus chloraniline and nitraniline acid do not give azine derivatives, but very stable diamine salts of the hydroxyquinones.

The toluylene-azine of tetrahydroxyquinone is the only one that has hitherto been described. The corresponding phenylene compound was prepared by the author by acting with one molecule of the sodium salt of tetrahydroxyquinone on one molecule of a phenylenediamine salt in dilute aqueous solution. On acidifying with acetic acid greenish-brown needles separate out, which when damp oxidise rapidly in the air; this azine crystallises more readily than the corresponding toluylene-azine, it dissolves with a dark olive-green colour in dilute mineral acids and is oxidised to the rhodizonic derivative by dissolving in fixed alkalis, a violet-blue colouration appearing at the same time. The azine from chloroparadihydroxyquinone is also described. This new body dissolves in alkalis or alkaline carbonates with a reddish-yellow colour almost identical with the colour of alcoholic solutions of O. Fischer and Hepp's diamidophenazine.

By the action of hydroxynaphthoquinone-isoide on orthophenylenediamine,  $\alpha$ -amidonaphthophenazine, described by Fischer and Hepp, is produced. Dihydroxyphenazine, which has also been prepared by these authors, is probably identical with the condensation product of dihydroxyquinone and phenylenediamine.

For preparing  $\alpha$ -naphtho-eurhodol one molecule of the sodium salt of hydroxynaphthoquinone is dissolved in boiling water, acidified with acetic acid, and treated with one molecule of phenylenediamine sulphate. The mass is finally heated on the water-bath, filtered, dissolved in hot but very weak caustic soda, again filtered and the blood-red filtrate precipitated with quarter of its volume of concentrated caustic soda solution. The golden-yellow metallic leaflets of the sodium salt which separate are filtered, dissolved in water and the eurhodol precipitated with acetic acid. The latter is at once crystallised from boiling acetic acid. The crystals that separate are now almost insoluble in acetic acid. This product is identical with the one prepared by converting Fischer and Hepp's amidonaphthophenazine into the corresponding eurhodol.

By replacing phenylenediamine with orthodiamidotoluene (1:2:4) in the above mentioned case, the eurhodol which corresponds to the eurhodine-base C<sub>17</sub>H<sub>13</sub>N<sub>3</sub> is obtained; the latter was first prepared by Witt from orthoamidoazotoluene and  $\alpha$ -naphthylamine.—A. R.

# PATENTS.

*Improvements in Producing Yellow Colouring Matter suitable for Dyeing and Printing.* W. W. Grieve, Glasgow. From "The Farbenfabriken vormals F. Bayer & Co.," Elberfeld, Germany. Eng. Pat. 9606, July 1, 1884. Amended March 27, 1890. 4d.

THE original specification has already been abstracted (this Journal, 1885, 491). In the amended specification the claim is confined to the production of a yellow dyestuff by the action of tetrazo-ditolyl on salicylic acid. (See also this Journal, 1885, 400, and 1890, 497.)—T. A. L.

*Improvements in the Preparation of a Beta-Naphthylamine Sulphonic Acid and of Colouring Matters therefrom.* A. G. Green, London. Eng. Pat. 15,849, November 2, 1888. Reprint. 4d.

ALREADY abstracted (this Journal, 1889, 878). The Comptroller's amendment is as follows:—"I (the patentee) am aware of the specification of Letters Patent 5846 of 1886 (this Journal, 1887, 436) granted to J. Y. Johnson, and I wish it to be understood that I do not claim anything described and claimed therein." A further amendment is the insertion of the word "when" in claim 3.—T. A. L.

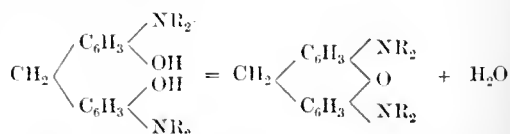
*Manufacture of Colouring Matters Derived from Para-Toluidine,  $\alpha$ -Metylglidine, Pseudocumidine and Mesidine with Tetramethyldiamido Benzhydrol.* O. Imray, London. From "La Société Anonyme des Matières Colorantes et Produits Chimiques de St. Denis," Paris, France. Eng. Pat. 12,784, August 13, 1889. 6d.

A METHOD for obtaining triphenylmethane derivatives which dye green to blue shades by condensing *p*-toluidine,  $\alpha$ -metylglidine,  $\psi$ -cumidine or mesidine, with tetramethyldiamidobenzhydrol by means of sulphuric acid or hydrochloric acid. The leuco bases thus obtained are oxidised with lead peroxide, yielding dyestuffs which may be diazotised and converted into hydroxy-derivatives. Previous to oxidation the leuco bases may, as an alternative, be benzylated and sulphonated when greenish-blue colouring matters are obtained. These dyestuffs are said to possess a great affinity for wool which is easily dyed in an acid bath.

—T. A. L.

*Improvements in the Manufacture of Colouring Matters.* H. H. Lake, London. From A. Leonhardt and Co., Mühlheim, Germany. Eng. Pat. 13,217, August 21, 1889. 6d.

By acting with formaldehyde on dimethyl- or diethyl-*m*-amidophenol colourless condensation products are formed, namely, tetramethyl- or tetra-ethyl-diamidodihydroxydiphenylmethane. On treatment with a dehydrating agent these products undergo a further condensation according to the general equation:—



These substances on oxidation yield pink dyestuffs which may be employed for dyeing or printing on cotton. The following is one of the methods given for obtaining a colouring matter from dimethyl-*m*-amidophenol. A solution of 28 kilos. of dimethyl-*m*-amidophenol in 200 litres of 60 per cent. alcohol is treated with 10 kilos. of a 30 per cent. aqueous solution of formaldehyde. After standing some time the condensation product separates out in small plates melting at 180° C.; 10 kilos. of this are heated to about 100° C. with 50 kilos. of ordinary sulphuric acid until a sample dissolved in water, neutralised with caustic soda and filtered, does not give any precipitate when acetic acid

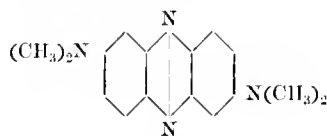
is added to the filtrate. The whole melt is then poured into water and the tetramethyldiamidodiphenylmethane oxide precipitated with caustic soda. In order to oxidise it, 2·7 kilos. are dissolved in 120 litres of water, 4 kilos. of hydrochloric acid added and when cold a solution of about 0·8 kilos. of sodium nitrite is run in. The colouring matter obtained is precipitated with zinc chloride and common salt.—T. A. L.

*Improvements in Producing Azo-Colours on or in Cotton or other Textile Fibre or Fabrics.* T. Holliday, Huddersfield. Eng. Pat. 16,156, October 14, 1889. 6d.

See under VI., page 939.

*Manufacture of a Grey Basic Colouring Matter Soluble in Water.* J. Dawson, Huddersfield. Eng. Pat. 16,448, October 18, 1889. 6d.

WHEN amidodimethylaniline is oxidised with chromic acid in presence of one molecule of hydrochloric acid or under certain other conditions, it is converted into two basic colouring matters, one blue and easily soluble, the other greyish-violet and less soluble in water.\* The following proportions are given: 10 kilos. of nitrosodimethylaniline hydrochloride are suspended in 700 litres of water and reduced with 10 kilos. of zinc dust at 40°. When decolourised the solution is neutralised with soda ash, filtered, and 7 kilos. of 30 per cent. hydrochloric acid or 5·8 kilos. of sulphuric acid or 4 kilos. of zinc chloride added; 5 kilos. of potassium bichromate and 4·5 kilos. of hydrochloric acid, or 3·6 kilos. of sulphuric acid, or 2·5 kilos. of zinc chloride in 100 litres of water are then added to the boiling solution. When the oxidation is complete the solution is filtered hot, and the blue colouring matter precipitated cold by the addition of 2·5 kilos. of zinc chloride and 100 kilos. of salt. The residue containing chromium left after filtering the hot solution is suspended in 400 litres of water and boiled with 5 kilos. of hydrochloric acid. After filtering, a greyish-violet colouring matter is precipitated by the addition of zinc chloride and salt. The blue colouring matter is a dark blue powder dissolving in sulphuric acid with a green colour. It is easily soluble in alcohol and water, and dyes cotton blue with a tannin- and black with an iron-mordant. The base is supposed to have the following formula:—



The greyish-violet colouring matter is "only perfectly soluble in acidulated water," and dissolves in sulphuric acid with a dirty brown colour. Cotton mordanted with tannin is dyed a greyish-violet, and it can also be used with unmordanted cotton.—T. A. L.

*The Preparation of Amidonaphtholsulphonic Acid, and of Colouring Matters therefrom.* S. Pitt, London. From L. Casella and Co., Frankfort-on-the-Maine, Germany. Eng. Pat. 16,699, October 22, 1889. 6d.

By fusing the  $\beta$ -naphthylamine- $\gamma$ -disulphonic acid, described in Eng. Pat. 816 of 1884 (this Journal, 1884, 567) with caustic soda at a temperature not exceeding 200° C., an amidonaphtholmonosulphonic acid is obtained, which combines with diazo compounds to form dyestuffs. The combination can take place in an acid or alkaline solution, and in the latter case the resultant compound can be rediazotised and again combined with amines or phenols. The colouring matters obtained are reds, browns, and violet-blues, and are said to be fast to milling and scouring. The amidonaphtholsulphonic acid itself appears to form a diazo compound of a yellow colour scarcely soluble in

water. This forms azo colours with amines and phenols, and by the action of an alkali it is converted into a violet colouring matter.—T. A. L.

*An Improved Manufacture of Colouring Matter.* H. H. Lake, London. From A. Leonhardt and Co., Muhlheim, Germany. Eng. Pat. 17,223, October 30, 1889. 4d.

A PROCESS for obtaining a nitrosodihydroxynaphthalene from the dihydroxynaphthalene melting at 186° C. by adding sodium nitrite to its solution in dilute sulphuric acid. The reddish-brown precipitate of the nitroso compound which is formed, gives coloured lakes with metallic oxides, that with iron being green, and on wool fast to light and fulling. With a chromium mordant the shades are brown. The colours are of no value for dyeing cotton.

—T. A. L.

*Improvements relating to Colouring Matters.* H. H. Lake, London. From A. Leonhardt and Co., Muhlheim, Germany. Eng. Pat. 17,971, November 11, 1889. 4d.

By condensing formaldehyde with *m*-toluylendiamine a tetra-amido derivative of diphenylmethane is obtained, which by elimination of ammonia and subsequent oxidation yields a yellow dyestuff. The following method is employed for its preparation: 13 kilos. of *m*-toluylendiamine and 26 kilos. of its sulphate are dissolved in 200 litres of water and treated when cold with 10 kilos. of a 30 per cent. solution of formaldehyde. After standing a short time the sulphate of the condensation product separates and may be filtered off. 10 kilos. of this are then heated in an enamelled autoclave with 30 kilos. of hydrochloric acid (containing 30 per cent. HCl) and 100 litres of water for six hours to 160° C. The brownish-red liquid thus obtained is filtered if necessary, and oxidised with 10 litres of a solution of ferric chloride of sp. gr. 1·4 and heated to about 90°. The colouring matter separates as a yellow crystalline powder which is filtered off and washed with dilute hydrochloric acid or brine. It dyes mordanted cotton yellow, and is faster to soap than benzoflavine. On silk or in an aqueous or alcoholic solution it shows a green fluorescence.—T. A. L.

## V.—TEXTILES: COTTON, WOOL, SILK, Etc.

*Shoddy.* J. Herzfeld. Chem. Zeit. 14, 903—904.

THE title "Shoddy" is intended to apply to all artificially recovered wool, although there is no exact English equivalent to the German "Kunstwolle," which includes all the recovered material from mungo shoddy and "extract," or wool from mixed goods. The industry was introduced from England into Germany by Gustav Kober in 1851 at Cologne, and after some early struggles flourished at Mannheim, spreading thence to all parts. To Kober is due the method of separating woollen from vegetable fibres by treatment with sulphuric acid. In such a manufacture the collection, sorting, and cleaning of the raw material are of much importance, but present little technical interest. The separation of vegetable fibres calls, however, for a more detailed account. Two processes are in use, the wet and the dry; in the former the raw material is soaked in sulphuric acid of 4°—7° B. for 12 hours, the greater part of the acid removed from it in a centrifugal machine and dried at 40°—50° C., after which it is carbonised at 90°—95° C. In the latter it is treated with gaseous hydrochloric acid. In either case the carbonised vegetable fibre is beaten out. The recovered wool felts badly.

It is estimated that at least 100—150 million kilos. of recovered wool are produced yearly in Europe. Ten years ago only 34 per cent. of the cloth made in England was prepared from pure new wool, and 9 per cent. of woven fabrics, and these figures are considerably too high for the present state of the trade.—B. B.

*Report on Textile Fibres and derived Products.* Relazione nel. Lab. Chim. Centr. delle Gabelle, 385—402.

THE import duty in Italy on textile fibres and tissues varies according to the proportion of their components; thus on mixed tissues of cotton or wool with silk or tinsel the duty is different according as the proportion of silk is greater or less than 12 or 50 per cent., and on mixed tissues of cotton and wool according as the proportion of wool is greater or less than 50 per cent. Thus in cases of dispute not only a qualitative identification of the tissue is necessary, but also quantitative determinations. The methods adopted for the micro-chemical investigation are based upon the work of von Höhnelt; the direction of the laboratory is now engaged upon an examination of an exact and rapid method for the quantitative separation of various tissues, which is to form the subject of the next publication. Meanwhile the following processes have been adopted: First, for mixtures of cotton and wool, the colouring matter and size are extracted by water containing a trace of sodium carbonate and the tissue then washed with hydrochloric acid, dried and weighed; another portion is digested with 10 per cent. potash solution, which dissolves the wool. Secondly, for mixtures of wool and silk, the size and colouring matter are extracted as before, and the tissue digested in Löwe's solution, which dissolves the silk, but this method is not applicable for Yamamaya, Tussah, and Cyenthia silks, in which cases the method proposed by Höhnelt is used; experiments are being made upon its applicability.

Analyses are given of various mixtures of wool, cotton, and silk sent to the laboratory, and also answers to inquiries as to the nature of various tissues sent for examination. Among the latter the following frauds were detected: a sample denominated velvet plush of silk and cotton was found to consist entirely of Tussah silk, another denominated wool contained 21 per cent. of silk, while a third, supposed to be silk and wool, was only silk. The varnish of various samples consisted principally of resinous matters mixed with oil, but some contained mineral substances such as lead and manganese oxides, barium sulphate and sand; the red colouring matter consisted of ferric oxide and the black of tar. A certain sample of buckram was covered with a layer two millimetres thick, consisting of a mixture of powdered cork, sand, and ferric oxide, the whole cemented together with a resinous substance and a little oil.—V. H. V.

#### PATENT.

*Improvements in Imparting to Fabrics, Threads, and other Articles a Silk-like Lustre.* W. Green, Ramsgate. Eng. Pat. 9879, June 15, 1889. 6d.

It is known that cellulose, suitably treated, forms a substitute for silk. Certain parts of young wood prepared in a state of great purity form the best material for this. It is converted by nitric acid into a pure octo-nitrocellulose which is dissolved in a mixture of 38 parts of ether and 42 parts of alcohol in a proportion of 6.5 per cent. The cellulose thus treated is put in a tinned copper vessel, in which it is kept under a pressure of several atmospheres. The material is forced from the copper vessel through glass tubes with capillary apertures, and in contact with water forms thin threads or filaments which are wound on to bobbins. The nitric acid is at the same time disengaged: "some pyroxylin lose their nitric acid in pure water, but more completely in dilute nitric acid of no greater density than 1.32, and the temperature of which should be caused to decrease slowly from 35° to 25° C. The cellulose thus treated becomes gelatinous and capable of absorbing various substances, particularly colouring matters and salts." The object of the patentee is to extend the use of this article for various purposes. He coats and treats other threads, fabrics, paper, lace, ribbons, buttons, flowers, vases, jewellery, and many other articles with octo-nitrocellulose as above described. In coating threads they are first passed through the octo-nitrocellulose solution, secondly through an aperture by which the superfluous liquid is removed, then through water, and finally wound on to bobbins. A closed vessel must be used to prevent as far as possible the escape of the ether

and alcohol. Another method is to bring the threads into contact with the octo-nitrocellulose, as the latter is forced from a capillary tube, and to cause adherence by giving a twist to the threads. In either case the threads are then to be treated as those of pure cellulose as described above. In treating fabrics, &c. an arrangement is made so that the fabric will pass under or over the tubes through which the cellulose is forced either as threads or as ribbons, the cellulose thus covering the surface of the fabric wholly or in part. By different motions and arrangements of the tubes as well as of the fabrics various patterns may be produced. In some cases adhesive matters or pressure may also be applied to ensure the more perfect union of the cellulose to the fabric. When the material is to be coated on both sides, it is brought into the solution and then passed between rollers. Fabrics thus coated may be used for numerous purposes in lieu of goods made of silk or of silk and other materials. For some purposes a "denitro-cellulose compound, or collodium, or a collodium flexile" may be used instead of octo-nitrocellulose. Moulded, hollow, or other irregular-shaped articles are treated by placing them on trays in a copper vessel. The solution is then run in, until the articles are sufficiently immersed, the superfluous liquid run off, and the coated articles removed and treated as above described. Buttons and many other articles may thus get a silk-like surface. If only a portion of the surface is to be coated with cellulose, this may be effected by pressing the fabric against an engraved roller revolving in the solution. For this purpose, however, the patentee prefers that an ordinary printing arrangement with proper arrangements for coating the engraved roller be fixed in a closed vessel provided with suitable inlets and outlets for the fabric.—H. S.

## VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

*On the Manufacture of Dye-wood Extracts.* C. Feuerlein. Chem. Zeit. 14, 887.

OWING to the great difference in the raw products, it is impracticable to judge of the value of the extract by the percentage of logwood contained in it. Different methods of determining the latter give totally different results, and the quality must depend entirely on the use which the extract is put to. Soxhlet and Brühl have stated that the reason why the dye-wood-extract industry has made so little progress in Germany is that it has been carried on in a less scientific manner. The author, while refuting these remarks, traces the cause to the large duty on extracts in France and America. He advocates an increase in the duty to Germany, which at present amounts to 3 M. per 100 kilos.—A. R.

*Determination of the Value of Dye-wood Extracts.* L. Schreiner. Chem. Zeit. 14, 961—962.

See under XXIII., page 976.

*Test for Fastness to Light and Resistance to Lime of Colouring Matters.* H. Stössel. Chem. Zeit. 14, 903.

MANY colouring matters are now upon the market, consisting of some "base," such as chalk or barium sulphate coloured with an aniline dye (this Journal, 1890, 301), and their fastness cannot always be depended upon. For ascertaining how far they may be expected to resist the action of lime, 1 grm. of the substance is shaken at intervals for six hours with 10 cc. of a normal solution of caustic soda. A check test may be made by shaking another portion of 1 grm. with sugar solution of the same specific gravity (1.046), and comparing the difference in colour. Limewash of the consistency likely to be used is considerably more alkaline than



normal alkali, but any colours likely to fade show tendency to do so when treated as described above.

The fastness to light of such colours may be gauged by exposing them, partly covered with a piece of dark paper, to sunlight for six hours, and comparing the covered and uncovered portions.—B. B.

*Diamond Black.* Farbenfabriken F. Bayer and Co. Chem. Zeit. Rep. 14, 216.

BEFORE dyeing, the wool or other textile fibres are boiled an hour with 10 per cent. glauher salt, and after washing, boiled another half-hour with 2 per cent. potassium bichromate. Both baths may be used for an indefinite time, and the dye-bath may be shaded with other benzidine dyes.

—A. R.

*Anthracene Yellow.* Farbenfabriken F. Bayer and Co. Chem. Zeit. Rep. 14, 216.

THIS dye belongs to the alizarin group, and is correspondingly fast. Instructions for dyeing: mordant with 3 per cent. potassium bichromate and 2 per cent. cream of tartar or 1 per cent. tartaric acid; boil one hour, wash, and dye in a fresh bath with 2 per cent. acetic acid.—A. R.

*The Influence of Olive Oil used in Oiling Wool on the subsequent Dyeing Operations.* A. Horvitz. Färb. Zeit. No. 11, 1890.

ALTHOUGH the oiling of wool necessary before carding is merely a preliminary operation to spinning, it is, nevertheless, of great importance to the dyer, in so far as a proper selection of the oil is essential to the production of an even shade. It is usually supposed that an admixture of resinous oils, mineral oils, and drying oils to the oiling substances creates disturbances in the dyeing process, and this supposition has proved to be correct, although the action of the substances mentioned has not yet been scientifically established. In practice their presence renders the production, especially of light shades, much more difficult, and has not unreasonably been considered as one of the causes of unevenness. The resinous oils and the mineral oils being unsaponifiable are certainly injurious, because they cannot be removed from the woollen fibre by the treatment usual in the manufacture; and in the subsequent dyeing process they are obstructive to an even penetration of the colouring matter into the wool fibre. But this effect being probably produced by all other unsaponifiable fats, the condition made hitherto, that the oils are to contain no resinous or mineral oils, may be extended so far as this: that they are to be free of any unsaponifiable fatty ingredients.

An unsaponifiable fat, which the author frequently found in the oiling substances, is cholesterol. The author repeatedly observed that the presence of cholesterol is more obstructive to an even fixation of the colouring matter than that of any other unsaponifiable ingredient; and he, therefore, recommends that all oils used for wool should be tested for cholesterol.

The detrimental effect of the drying oil may be caused by the wool-fibre becoming enveloped by a coat of varnish, which would partly prevent the entrance of the colouring matter.

The author next discusses the behaviour of olive oil. This substance is obtained as a purer product by pressing the olives in the cold, while the second yield is obtained by boiling the olives and by hot pressing. It is this latter product which is chiefly used for oiling wool.

It consists chiefly of triolein (about 72 per cent.), and contains besides palmitin, arachin, arachinic acid, and very little stearin. According to Schaedler, it invariably also contains cholesterol.

With the usual methods of preparing the oil, the quantity of cholesterol passing into the olive oil will be very small, so that the presence of such traces will scarcely have a perceptible influence. But if the amount of cholesterol in an oil should be as much as 4 per cent., which the author frequently observed, the oil is of the poorest quality, and

has been obtained by unusual methods of preparation. In one instance the author, having extracted cholesterol from dyed pieces that were spotted, examined the olive oil that had been used for oiling the wool. The quantity of cholesterol obtained by saponifying the oil, drying the soap produced, and extracting it with petroleum, was about 3 per cent.

The olive oils of commerce are never pure oils. Adulterations with less expensive oils are very common. An addition of non-drying vegetable oils can only interest the spinner, while in dyeing it can scarcely have any bad effect. An admixture of drying oils, resinous or mineral oils, will, on the contrary, create more or less conspicuous disturbances in dyeing.

The chemist will detect an adulteration with these substances easily by a change in the iodine and saponification equivalents, as well as by deviations in the melting and solidifying points of the fatty acids, while the practical man is limited to qualitative reactions which, as the author remarks, can only be considered as perfectly reliable if, at the same time, confirmed by quantitative tests.

The presence of drying oil may be shown qualitatively as follows:—A well-cleaned copper wire is placed in a mixture of 2 ccm. of nitric acid and 5 ccm. of oil; the mixture is then stirred with a glass rod. If the sample contained a drying oil, the wire, if withdrawn after half an hour, will have a pink shade.

The test for resinous or mineral oil requires the oil to be saponified, the dried soap to be extracted with petroleum ether, and a further examination of the residue after evaporation of the ether. According to Allen, a mixture of 10–12 drops of resinous oil with bromide of tin is coloured a brilliant purple. Bromide of tin may be rapidly prepared by adding bromine, after having shaken it with sulphuric acid for the purpose of removing the water from it, drop by drop on tin filings kept in a well-cooled bottle.

For cholesterol the unsaponifiable portion may be tested by the following reactions, proposed by Schulze:—

1. If a small quantity of cholesterol be carefully evaporated to dryness on the lid of a crucible with a few drops of nitric acid, a yellow spot remains, which, if ammonia be poured on it, assumes a yellowish-red colour.

2. If a sample of cholesterol be rubbed on the lid of a crucible, with a drop of a mixture of three volumes of concentrated sulphuric acid and one volume of solution of ferric chloride, then carefully evaporated to dryness, the undissolved particles assume a violet-red colour which gradually turns more towards blue.

3. If a sample of cholesterol be rubbed together with a little concentrated sulphuric acid, and chloroform be then added, a blood-red solution is produced, which on the admixture of concentrated nitric acid becomes violet, blue, and finally colourless.

If an olive oil destined for oiling wool contains several of these injurious admixtures at the same time, it becomes a matter of great difficulty to answer the question so frequently put in practice as to what extent the oil may have an injurious influence in the further course of the manufacturing operation. Researches in which the quantity of the admixed substances, as well as the amount of damage caused by them, are taken into account, have not yet been made.

The author mentions a point which is not, indeed, of immediate interest to the dyer, but in other respects it is of paramount importance—namely, that all oiling materials for wool should not contain any free acid since it strongly attacks the cards, destroying them in a short time.

## PATENTS.

*Improvements in Machines for Dyeing, Bleaching, and otherwise Treating Yarn in Cop or other Compact Form.* A. Graemiger, Manchester; W. T. Whitehead, Radcliffe; S. Mason and E. A. Leigh, Manchester. Eng. Pat. 9692, June 12, 1889. 8d.

THE improvements relate to the cop-dyeing machine patented by Graemiger (Eng. Pat. 11,497 of 1887), and have for their object the lessening of friction between the



cop-carrier and carrier-body in the same. A double disc cop-carrier, *b b* (Figs. 1 and 2), capable of being intermittently rotated, is now employed. The discs are fitted

against a central stationary carrier body *c*, which is divided into four chambers, *h*<sup>1</sup>, *h*<sup>2</sup>, *h*<sup>3</sup>, *h*<sup>4</sup> (Fig. 1), communicating successively on the rotation of the discs, with the cops *g*

Fig. 1.

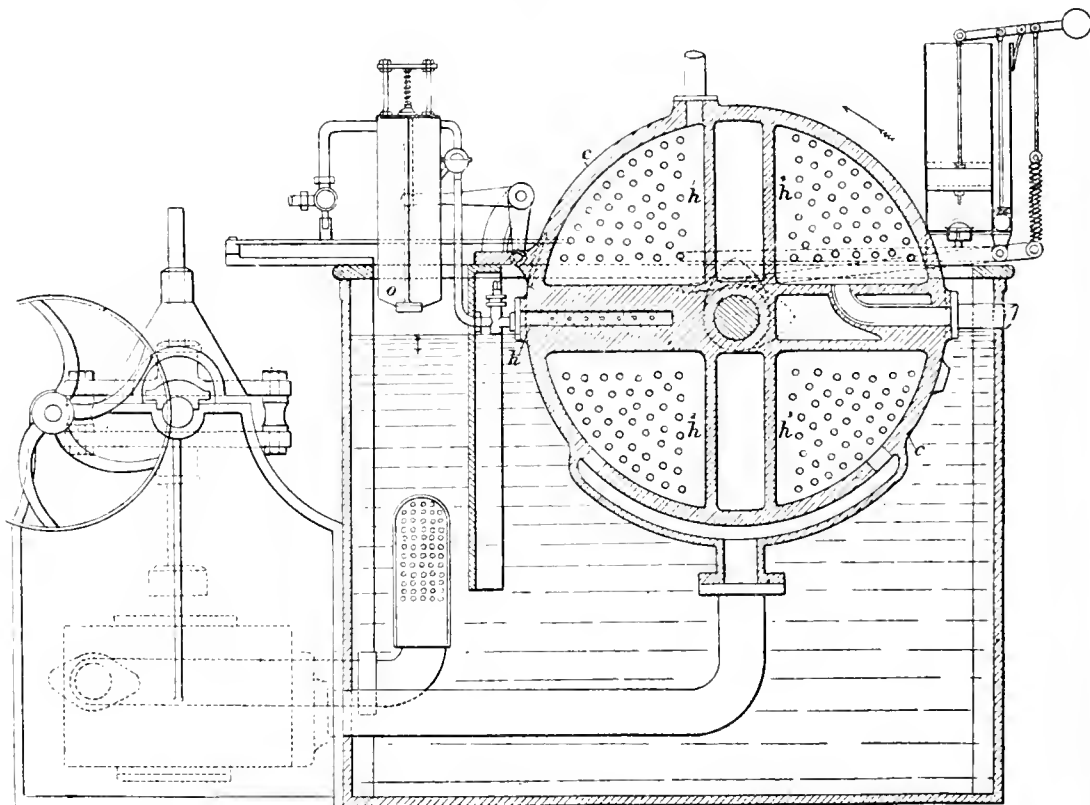
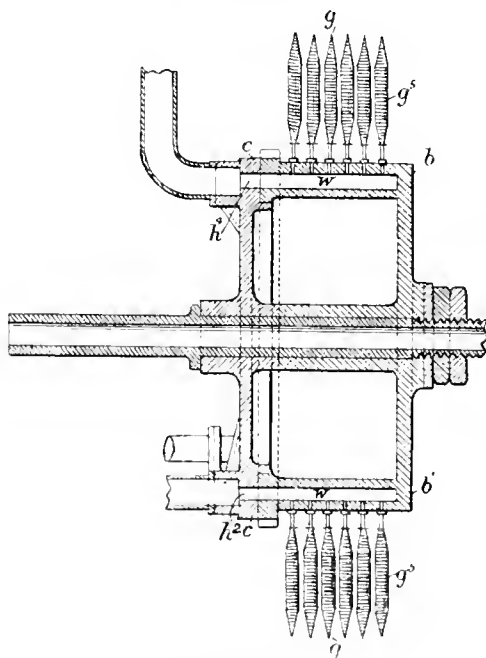


Fig. 2.



(shown in Fig. 2) fixed on perforated injection tubes *g* projecting from the outer faces of the discs. Chamber *h*<sup>1</sup> serves for liquor extraction and substitution of the cops, *h*<sup>2</sup> and *h*<sup>3</sup> for saturation and impregnation, and *h*<sup>4</sup> for liquor extraction. Instead of the dead face hitherto left in the carrier body between chambers *h*<sup>1</sup> and *h*<sup>2</sup>, a preliminary extraction chamber, *h*, is formed, connected with an air and liquor separator, *o*, which prevents liquor from entering the air-pump and allows it to be returned to the tank *a* free from air. By means of the cylindrical apparatus *n* the liquor is supplied to the tank in regulated quantity and its level in the same is kept constant.

Fig. 2 is a longitudinal section of a cylindrical machine. The cop-carrier *b* is in this case fitted to rotate against the end of the carrier body *c* instead of around it. The carrier body is formed with segmental chambers for effecting the above-mentioned operations. The cops *g* are placed in rows at equal distances apart round the periphery of the carrier, and each row of the same is connected with a separate segmental cavity, *w*.

In the original specification details are given of the construction and mode of working of the automatic air and liquor separator, liquor charging vessel, and means of closing the vacuum valve, all of which are applicable to the cylindrical as well as to the disc machine.—E. B.

*Improvements in Sizes and Methods for Fixing Colours in Printing and Staining Paper, Cloth, and other Surfaces.* R. Leigh, Bolton. Eng. Pat. 11,938, July 27, 1889. 4d.

Two processes are included in this specification. In the first a size or vehicle for fixing colours is prepared from a

mixture of (a) a solution of rosin in borax, ammonia, or other alkali; (b) a solution of casein in alkali; or a solution of gum, glue, or starch; and (c) a chrome salt. The colour mixed with this vehicle is printed on the fabric and dried in the usual way.

The second process consists in adding powdered gum, rosin, sulphur, or other gummy, resinous, or oily substance not soluble in water, to a mixture of chrome salt and gum, glue, starch, or other binding material, or alternatively to silicate of soda, gluten, or size. The resinous body is either ground up with the colour direct, or first dissolved in turpentine, petroleum spirit or other solvent. In the latter case the resin is precipitated in a finely divided state on mixing with the thickening. In the use of this vehicle the fixation of the colour is effected by the heat of the drying-plates over which the printed fabric passes. By an alternative process the thickening and colour alone are printed and the rosin or gum dusted or padded on to the moist fabric and then dried as before.—W. E. K.

*Improvements in the Production of an Indigo Vat.* A. Ashworth, Manchester. Eng. Pat. 14,246, September 10, 1889. 4d.

A CLEAR vat for the reduction of indigo is prepared by acting on zinc with bisulphite of soda and, when the reduction is complete, precipitating the zinc by addition of sodium sulphide.—W. E. K.

*Improvements in Machinery or Apparatus for Dyeing or Washing Hanks of Yarn.* J. Ashworth, F. Scholes, and R. Hunt, Radcliffe. Eng. Pat. 14,257, September 10, 1889. 6d.

THIS invention relates to that class of machines in which revolving rollers are employed for supporting the yarn in the vat or dyeing cistern. The machine is made double with two sets of "winces" and two dyeing cisterns. A central shaft runs the whole length of the machine between the two cisterns, and is provided with worms at suitable distances apart. These gear into wheels keyed on to shafts set at right angles to the driving shaft. On each end of these counter shafts is fixed a disc carrying three metal prongs or rods for supporting the hanks of yarn.—W. E. K.

*Improvements connected with or applicable to Baths or Vessels containing Liquids used in Electro-plating or Dyeing or the Electrolyte of Storage Batteries and the like.* E. Hopkinson and D. Appleton, Manchester. Eng. Pat. 14,871, September 20, 1889. 6d.

See under XI., page 952.

*Improvements in Hank-dyeing Machines.* R. Gee, Huddersfield. Eng. Pat. 15,455, October 2, 1889. 8d.

FOR the purpose of dyeing hanks of yarn in such a manner that every part is equally exposed to the dye liquor without the felting or intermingling of the threads, an ordinary dye-vessel is provided with two endless chains arranged lengthwise, one at each side of the vat. These are supported and driven by toothed guide pulleys fixed on a moveable framework. To each chain are attached bobbins or "creels" placed in a vertical position, the hanks of yarn being distended horizontally across the machine between the two sets of bobbins. The pulleys are placed at such distances apart that the hanks are fully distended whilst the chains are traversing the upper ones, but when the chains pass down under the lower guide pulleys, they are caused to converge so that the hanks are slackened and open out and thus the threads are fully exposed to the liquor. During the upper traverse of the chain the bobbins carrying the hanks are caused by frictional contact with leather-covered bars to turn a half revolution or thereabouts in order that the portion of yarn around the bobbins may be constantly

changed. The framework carrying the guide pulleys, together with the endless chains and bobbins, are bodily lifted clear of the liquor when the operation is over, this being effected by hydraulic rams.—W. E. K.

*Improvements in Hank-dyeing Machines.* R. Gee, Huddersfield. Eng. Pat. 15,512, October 3, 1889. 8d.

THE hanks of yarn to be dyed are supported vertically between two rectangular frames, one at the bottom the other at the top of the dye vat. The yarn passes around bobbins or "creels" fixed in the two frames, and is distended by springs acting on the bearings of the lower series of bobbins. The axes of the upper creels are geared by a train of toothed wheels which impart a rotary motion to the hanks. The lower frame, by an arrangement of levers worked by an eccentric, is alternately raised and lowered, the effect being that the hanks of yarn are slackened intermittently and the threads opened out, this action allowing the dye to penetrate to the individual fibres.

—W. E. K.

*Improvements in or relating to Bleaching and Methods for Producing Bleaching Agents.* L. Schreiner, Stuttgart, Germany. Eng. Pat. 15,671, October 5, 1889. 6d.

A BLEACHING material, termed "ozonin," is prepared by dissolving turpentine in a potash-resin soap, and oxidising the product by atmospheric air or by peroxide of hydrogen. "Ozonin" also acts as a powerful antiseptic.—W. E. K.

*Improvements in Producing Azo-Colours on or in Cotton or other Textile Fibre or Fabrics.* T. Holliday, Huddersfield. Eng. Pat. 16,156, October 14, 1889. 6d.

THIS invention relates to a process of dyeing textile fibres by the combination of  $\alpha$ - or  $\beta$ -naphthol with the diazotised various thio-compounds derived from aniline, toluidines, xyldines, emidines, or naphthylamines, such combinations taking place in the presence of the fibre. For example: hydrochloride of thioparatoluidine is converted into the diazo-compound by treatment with hydrochloric acid and nitrite of soda, the cotton dipped into or padded with this solution, and then (with or without drying) passed into a solution of one of the naphthols dissolved in caustic soda; or the order of these operations may be reversed. To produce coloured patterns, as in calico-printing, the cloth is impregnated with the naphthol solution and dried, and the diazo solution, suitably thickened, is then printed on.

—W. E. K.

*A New or Improved Method of Bleaching Wood, Bobbins, Reels, and other Articles made from Wood.* A. Greg, Bolton. Eng. Pat. 16,368, October 17, 1889. 4d.

THE articles are treated successively with baths of lime or other alkali, chloride of lime, and sodium hyposulphite. They are then stopped with burning sulphur, or treated with a solution of sulphurous acid, and finally treated with water containing acetic acid "for the purpose of replacing the natural acid displaced by the before-mentioned baths."

—W. E. K.

*A New or Improved Method of Dyeing Aniline Black.* L. Schreiner, Stuttgart, Germany. Eng. Pat. 19,490, December 4, 1889. 4d.

ALBUMEN, casein or analogous glutinous bodies are mixed with the solution of aniline salt used in dyeing aniline black, and these are coagulated by the reagents subsequently employed for the oxidation of the aniline.—W. E. K.

*Improvements in the Development or Treatment of Colouring Matters in Textile Fabrics or Materials.* J. J. Hart, Lowell, Mass., U.S.A. Eng. Pat. 8541, June 3, 1890. 8d.

This invention relates to those processes in calico-printing in which a colouring matter and mordant or other reacting body, after having been printed and steamed, require a supplementary treatment with a chemical reagent for the development or complete fixation of colour; as for example, the treatment with bichromate of potash in the case of catechu colours, or with tartar-emetic in the case of aniline colours fixed by means of tannic acid. It is the usual practice to pass the fabric through a hot bath of these supplementary reagents, but this process is objectionable on account of detached colour-lake, and foreign bodies contaminating the bath and staining the fabric. To avoid such damage, by this invention the fabric is passed through a cold solution of the reagent, the excess of solution squeezed out by pressure rollers, and the necessary chemical reaction is then effected by passing the fabric over cylinders heated by steam or through a steam-chest.—W. E. K.

*An Improved Blue for Laundry and other Colouring Purposes.* G. R. B. Kempton, West Ham. Eng. Pat. 10,616, July 9, 1890. 4d.

To a suitable proportion of a soluble salt, sufficient "Aniline blue, Prussian blue, Chinese blue, Indigo blue, or Ultramarine blue" in solution is added, and the whole evaporated to dryness, after which the resulting crystalline mass is ready for use.—K. M.

*Improvements in Centrifugal Leather-Dyeing Machines.* J. Kristen, Brum, Moravia. Eng. Pat. 11,255, July 18, 1890. 6d.

OVER a circular horizontal revolving table, the upper surface of which is formed of a glass plate (in order to facilitate the adhesion of the hide) a radial perforated pipe is fixed, and this is connected with a tank that supplies the dyeing liquor. The surplus liquor projected beyond the edge of the plate is received in a conical flanged tank which surrounds the table, and is returned to the liquor tank by means of a pump.—W. E. K.

*Improvements in Yarn-Dyeing Machines.* H. H. Lake, London. From The Leonard Weldon Dyeing Machine Company, Amsterdam, U.S.A. Eng. Pat. 11,873, July 29, 1890. 6d.

THIS invention relates to that class of dyeing machines in which the skeins of yarn are carried on bars affixed to the interior of a frame or reel which rotates within the dye vat. The improvement consists in disposing the respective sets of yarn-carrying bars in planes oblique to the radius of the rotating wheel, so that the yarn is drawn partly endwise and partly side-ways through the liquor, and in consequence the yarn is dyed more evenly and the strain upon it reduced.—W. E. K.

—W. E. K.

## VII.—ACIDS, ALKALIS, AND SALTS.

*The Relative Basic Properties of Aluminium and Ferric Hydroxides.* A. Schneider. *Annalen*, 257, 359—380.

Already abstracted from *Ber.* 23, 1349—1354 (this Journal, 1890, 837).

*Potassium Tetra- and Penta-thionate.* A. Foek and K. Klüss. *Ber.* 23, 2428—2432.

RAMMELSBERG (*Kryst. Phys. Chem.* I., 495) described a potassium pentathionate, which it was suggested in Graham-Otto's book (5 Edition III., 125) was thiosulphate or tetrathionate. Debins (*Ann.* 244, 76) placed the existence of

pentathionic acid beyond doubt by preparing and analysing with Lewes the pure potassium salt. The authors suggest that Lewes (*J. Chem. Soc.* 39, 68; 41, 300) and Shaw (*J. Chem. Soc.* 43, 351) worked with an impure preparation.

The crystals of tetrathionate prepared by the authors belong to the monosymmetric system, and measurements are given in detail. Both in Rammelsberg's original preparation and in the authors' a peculiar hemimorphic or hemihedral formation was observed in some of the crystals, which will be more fully described in the *Zeits. für Krystallographie*.

The authors prepared the pentathionate as follows:—Sulphuretted hydrogen was led for two hours every day for 10 days in a slow stream through a litre of a nearly saturated solution of sulphur dioxide. At the end of this time the smell of sulphur dioxide had gone. The Wackenroder's solution prepared in this way was concentrated in a water-bath at a temperature of 35°, and then filtered from precipitated sulphur; 85 cc. of sp. gr. 1.241 were obtained, which according to Kessler's tables would contain 33 per cent. of  $S_5O_3$ . The calculated quantity of potassium acetate was then added, the liquid put into a large watch-glass, and the water and acetic acid evaporated by a current of air. The residual mass after drying by pressure weighed about 25 grms., and was dissolved in 30 cc. of a 1 per cent. solution of sulphuric acid at a temperature of 45°, the solution filtered from sulphur and crystallised over sulphuric acid. The first two crops consisted nearly entirely of tetrathionate. The following crops contained pentathionate. By fractional crystallisation about 10 grms. of pure tetrathionate and 6 grms. of pure pentathionate were obtained. The crystals of the latter belong to the rhombic system and measurements of them are given in the original paper.

—H. S. P.

*On the Red Colouring of Sulphuric Acid.* R. Nörrenberg. *Chem. Ind.* 13, 363—364.

SOME consumers of vitriol object to a product possessing a red colour. This tinge is often produced in perfectly colourless vitriol when kept in iron vessels, and is due to the action of ferrous sulphate in solution on nitrous compounds; these are reduced into nitrous oxide, which dissolves in sulphuric acid with a red colour. The same observation can be made in the leading chambers of a set, the rose colour or the dark red appearance of the vitriol being here traceable to the iron, which is carried forward with the sulphurous acid from the burners. Addition of ammonium sulphate destroys the nitrous compounds and thus effectually prevents any red colour.—H. A.

## PATENTS.

*Improvements in Apparatus for Generating Carbonic Acid Gas.* F. Foster, Hoxton. Eng. Pat. 17,569, December 1, 1888. Reprint. 6d.

THE apparatus has been already described in this Journal, 1889, 983.—H. A.

*Improvements in and Connected with the Utilisation of Sulphate of Iron Galvanising Pickle.* T. Parker and A. E. Robinson, Wolverhampton. Eng. Pat. 19,554, June 29, 1889. 4d.

See under X., page 949.

*Improvements in or Appertaining to the Manufacture of Peroxide of Lead, or Alkaline Plumbate and By-Products.* P. Naef, Northwich. Eng. Pat. 11,962, July 27, 1889. 6d.

THE principal object is to effect the oxidation of oxide of lead to peroxide more economically than hitherto. Nitrate of sodium or other nitrates are employed for this oxidation, nitrites being obtained as a valuable by-product. The

oxidation may be effected also by atmospheric oxygen directly. In either case, as intermediate product, a compound of peroxide of lead with an alkali or alkaline earth is obtained, from which subsequently peroxide of lead is separated. Metallic lead may also be used for making the peroxide. The lead or lead oxide is oxidised in presence of caustic soda, by means of nitre or a blast of air, at an elevated temperature, and the sodium plumbate so obtained is first washed and then decomposed with boiling water into lead peroxide and caustic soda. The sodium nitrate is thereby converted into sodium nitrite, which is found in the wash-waters of the sodium plumbate melt, along with caustic soda. These wash-waters are boiled down to concentrate them, and returned to the process after addition of caustic soda. When rich enough in nitrite, this is separated either by boiling down to a suitable strength and allowing the nitrite to crystallise, or else by carbonating and evaporating the liquor and separating the sodium carbonate by fishing out the monohydrated salt during evaporation or crystallising out by cooling, the sodium nitrite remaining in solution.

For the manufacture of voluminous and finely divided peroxide, the dense peroxide obtained from any source is heated with highly concentrated caustic soda till the mass has become very consistent. The product is washed and decomposed in the usual way.—H. A.

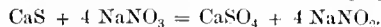
*Improvements in and connected with Apparatus for Producing Gaseous Hydrochloric Acid.* W. L. Wise, London. From Solvay and Co., Brussels, Belgium. Eng. Pat. 15,531, October 3, 1889. 8d.

The apparatus consists of two main parts: a horizontal rectangular distilling vessel provided with a partial partition, and containing sulphuric acid of about 140° Tw. or a strong solution of calcium chloride, and in connexion with this, and on the same level, an evaporating vessel with partition, for evaporating the dehydrating agent to its original degree of concentration.

A fine stream of hydrochloric acid is introduced in one end of the distilling vessel, where it mixes with the hot vitriol or calcium chloride propelled forward from the concentrating vessel, by means of a puddle-wheel or helix, or an air injector. While the mixture circulates along the partition, gaseous hydrochloric acid is given off, which may be condensed. The dilute sulphuric acid leaves at the other end of the distiller, and entering the evaporating vessel, takes the course formed by the partition wall, and returns to the distiller with the desired degree of concentration.—H. A.

*Improvements in the Manufacture of Nitrite of Soda.* J. Grossmann, Manchester. Eng. Pat. 15,655, October 5, 1889. 4d.

Sodium nitrate and calcium sulphide (or alkali waste which has previously been freed from cinders and coal) are heated together in quantities represented by the following equation:—



In lixiviating the resulting mass, boiling down and cooling, the impurities are separated from the mother-liquor, which will contain the sodium nitrite.—H. A.

*Improvements in the Method of Producing Chloride of Lead.* W. Shapleigh, Camden, U.S.A. Eng. Pat. 16,212, October 15, 1889. 8d.

The improvement consists in a better utilisation of the lead, nitric acid, and hydrochloric acid, as compared with the old process.

Finely-divided lead is introduced into dilute nitric acid, while a blast of air is forced through the vessel, to agitate and cool its contents and to oxidise the nitrous compounds, which will dissolve in the liquor. Part of the strong solution of lead nitrate is drawn off, decomposed with hydrochloric acid, the lead chloride collected on a filter, and the remaining part of the lead nitrate solution added to the

mother-liquor consisting of nitric acid and excess of hydrochloric acid. An additional quantity of lead chloride is obtained, and the nitric acid, being completely freed from hydrochloric acid, is fit to return to the process.—H. A.

*Process for obtaining Salts of Thorium, Zirconium, and like Metals from their Ores or from Waste Residues of Hoods used for Incandescent Gas Lighting.* O. Imray, London. From W. MacKean, Paris, France. Eng. Pat. 16,555, October 19, 1889. 6d.

THORIUM salts are obtained from the finely-ground ore by repeatedly digesting with hydrochloric acid, and treatment with an alkaline sulphide. Zirconium salts are obtained by fusing the ore with caustic alkali, baryta, or strontia, digesting the powdered melt with acid and water and treating the acid solution with an alkaline sulphide, precipitating the oxide with an alkali, and redissolving in hydrochloric acid.

The chlorides of zirconium and thorium are purified by repeatedly precipitating with sodium hyposulphite, and redissolving in hydrochloric acid, and finally, by precipitating with ammonia and dissolving in hydrochloric acid.

The separation of zirconium, thorium, lanthanum, and cerium, which may be contained in the waste residues of hoods, is based on the insolubility of the hyposulphites of zirconium and thorium in weak acid solutions, and on the solubility of thorium oxalate. Both lanthanum and cerium oxalate are insoluble, but their oxides can be separated by means of dilute nitric acid, which dissolves that of lanthanum.

—H. A.

*Improvements in obtaining Caustic Soda or Caustic Potash by means of Boracic Acid.* W. Garraway, Glasgow. Eng. Pat. 17,395, November 2, 1889. 4d.

A MIXTURE of boracic acid (3 to 4 mols. proportions) and common salt (2 mols.), when fused together, gives off hydrochloric acid, with formation of sodium metaborate or sodium pyroborate. The furnace mass is ground, dissolved in water, and decomposed with lime. Caustic soda remains in solution, whilst calcium metaborate or calcium pyroborate is precipitated. The boracic acid is regenerated therefrom by decomposition with the hydrochloric acid previously formed, or by any other suitable acid.

An admixture of nitre or salt cake, also injection of steam, facilitates the decomposition in the furnace.—H. A.

## VIII.—GLASS, POTTERY, AND EARTHENWARE.

*Recent Advances in the Pottery Trade.* Dingl. Polyt. J. 276, 580—596.

CERTAIN ornamental tiles of French make with a thick rose-coloured enamel, often showing a good many cracks, are glazed, according to a note in *Sprechsaal*, 1889, 597, as follows:—100 parts by weight of pure quartz sand are fused with 200 of red lead free from iron, and 5 of borate of lime, the melt quenched, ground up, and mixed with 10—30 per cent. of ruby glass having gold as its colouring material.

The yellow-green or bright red-brown earthenware vessels of Seinde are glazed with a lead silicate coloured with ochre or copper ashes (*Sprechsaal*, 1889, 183).

*Porcelain and Earthenware Glazes.*—The substitution of a leadless glaze for those in common use containing that metal is in many ways desirable, but the facility of production and the low cost due to the fusibility of lead glazes and the consequent small consumption of fuel in their production, lead to their retention.

G. Bücher, in the Jahresb. k.k. Fachschule f. Thonind. in Znaim defines the conditions which govern the successful production of a leadless glaze. A good refractory clay "fat" enough to take up one-third its weight of quartz sand, or a clay containing sand, but free from much lime, should be used. The simplest and cheapest glaze of the kind is made by dissolving 8 parts by weight of pearl-ash in hot water and stirring in 100 parts of dry ferruginous well-washed loam, mixed with 20 of yellow ochre. The goods are fired at an incipient white heat, and allowed to cool with the furnace closed and all cracks luted up; while thus cooling the copper-brown colour proper to the glaze is developed. A better class of pottery is made by using the same clay for the body of the ware, and the same glaze for the outside, but lining the inside with a mixture of 40 parts of white clay, 15 of fine sand, 15 of ground porcelain, and 20 of felspar, which is afterwards glazed with a composition consisting of 11 parts of felspar, 15 of calcined sand, 9 of ground porcelain, 9 of chalk, and  $3\frac{1}{2}$  of calcined kaolin. The furnace is hermetically closed while cooling as before. Instead of making the body of the ware of clay, a composition approaching that of porcelain is attained by using 48 parts of white clay, 6 of ground porcelain, 20 of calcined white sand, and 6 of felspar; in this case the outside is glazed with the brown mixture before firing, then the white glaze given above applied to the inside, and the firing repeated.

Seger, in continuation of his earlier work (Thonind. 1884, 46) has recently published some more valuable matter (Thonind. 1889, 524, 538, 553, 566) (for an abstract of a précis of this work see this Journal, 1890, 68). A more detailed account giving some of his recipes is as follows:—In pursuance of the attempt to prepare fusible glazes free from lead, various mixtures containing boric acid as a characteristic ingredient were tried in place of those containing baryta mentioned in his earlier paper. Attention was paid only to those glazes, the fusing points of which were below that of gold. The plan of investigation adopted was to prepare a mixture with the highest permissible amount of alkali and smallest quantity of lime and pass gradually to the other extreme. For each of these, mixtures were made in which the proportion of the other ingredients (silica, alumina, and boric acid) varied, and their behaviour examined.

#### I.—Glazes in which the bases have the relation—



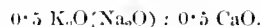
A glaze of the composition  $0.6 K_2O(Na_2O)$ ,  $0.4 CaO$ ,  $0.1 Al_2O_3$ ,  $2.5 SiO_2$ ,  $0.5 B_2O_3$ , fused at a temperature between the melting point of silver and that of the alloy 80 parts of silver to 20 of gold, and gave a smooth surface, moreover, attacking the colours beneath it less than most lead glazes; it was, however, necessary to use a highly siliceous well-burnt material for the body of the ware, e.g., 35 parts of clay, 5 of felspar, and 60 of quartz, burnt at a temperature corresponding to the cone known as Nos. 9 or 10; otherwise crackling was apt to occur. Such a glaze results from the prescription:—

	Parts by Weight.
Carbonate of soda .....	31.80
Marble .....	20.00
Ground quartz .....	75.00
Boric acid .....	31.00
Kaolin .....	12.95

On raising the proportion of alumina, silica, and boric acid until the formula  $0.6 K_2O(Na_2O)$ ,  $0.4 CaO$ ,  $0.6 Al_2O_3$ ,  $5.0 SiO_2$ ,  $1.0 B_2O_3$  was reached, a glaze was prepared which was white and opaque on slow cooling; a convenient mixture would be:—

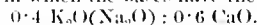
Nitre .....	30.80
Borax .....	47.75
Carbonate of soda .....	2.65
Marble .....	20.00
Kaolin .....	77.70
Quartz .....	114.00

#### II.—Glazes in which the bases have the relation—



A glaze from materials similar to those just mentioned and having the composition expressed by the formula  $0.5 K_2O$ ,  $0.5 CaO$ ,  $0.1 Al_2O_3$ ,  $2.7 SiO_2$ ,  $0.5 B_2O_3$  gave a smooth clear film at a temperature about that of the melting points of the alloys 20 gold to 80 silver, and 50 gold to 50 silver; very siliceous material was however necessary for the body of the ware. One of the composition  $0.3 K_2O$ ,  $0.2 Na_2O$ ,  $0.5 CaO$ ,  $0.6 Al_2O_3$ ,  $5 SiO_2$ ,  $1 B_2O_3$ , proved better adapted for a clay richer in alumina.

#### III.—Glazes in which the bases have the relation—



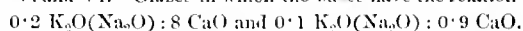
A glaze of the composition  $0.2 K_2O$ ,  $0.2 Na_2O$ ,  $0.6 CaO$ ,  $0.6 Al_2O_3$ ,  $5 SiO_2$ ,  $1 B_2O_3$ , and similar glazes richer in alumina, gave good results, and are of commercial value; they melt somewhat below the fusing point of gold. Those poorer in silica and alumina are of little use from their tendency to become turbid.

#### IV.—Glazes in which the bases have the relation—



No glaze of practical importance was produced, but a series of three with gradually increasing proportions of boric acid showed the influence of that substance very clearly, that containing  $0.5 B_2O_3$  giving a clear glass, while that with  $1.0 B_2O_3$  was opalescent, and that with  $2.0 B_2O_3$  distinctly milky.

#### V. and VI.—Glazes in which the bases have the relation—



A persistent and increasing tendency to become opalescent or milky characterised these and made them of little value.

The deductions from these experiments are that the conditions governing the successful application of such leadless glazes are more rigid than those for lead glazes; nevertheless the former could replace the latter for many purposes. In general those glazes containing much alkali are best for pottery made of highly siliceous clay, while those with more lime suit a material richer in alumina.

**Copper Red Glazes.**—Lauth and Dutailly have experimented in this direction; an abstract of their results may be found in this Journal, 1890, 68.

**Pink Mixture.**—Some recipes for a pink glaze on stoneware and majolica appear in Sprechsal, 1889, 706. One part of potassium bichromate is rubbed down with water and alcohol, with the gradual addition of 8 parts of powdered marble and "16 cbm. 5" of pure zinc oxide, and the almost dry mixture thoroughly ground together, then fired at the temperature usual for stoneware, washed with dilute hydrochloric acid until the washings are colourless, heated again and rewashed. The following mixture after ignition can be used as a diluent to give a suitable shade of colour: 2 parts stannic oxide, two of silica, one of powdered marble. Three different tints can then be prepared thus:—

Dark red...	{ 5 parts dried "pink mixture."
	{ 7 parts ignited "diluent."
Purple.....	{ 8 parts "dark red."
	{ 1 part "CaO <sub>3</sub> " (2 CaCO <sub>3</sub> ) (finely ground)
Cardinal...	{ 12 parts "dark red."
	{ 1 part calcium carbonate.

These colouring matters are mixed with 8 parts of one or other of the following mixtures:—

—	For Majolica Ware.	For Stone Ware.
Ground quartz.....	22.5	35
Borax (crystallised).....	18.5	10
Red lead.....	30.5	25
Kaolin (dried and sifted)	5.0	12
Chalk (levigated).....	7.0	18
Powdered felspar .....	16.5	..

A colour for porcelain or stoneware called *coralline red* may be prepared according to the *Deutsche Töpfer-oder Ziegler-Zeitung* 26, 796, thus: 20·40 parts of lead chromate are mixed with 67·33 of red lead and 12·27 of quartz, and the mixture fused to a clear melt, which is then powdered and mixed in about the proportion of 3:1 with either of the two following compositions:—

	I.	II.
Red lead.....	80	80
Quartz .....	100	20
Boric acid .....	20	..

No. I. is used for high and II. for low temperatures.

*Protection from Lead Poisoning in Potteries.*—N. A. Ruskitt recommends the adoption of the following regulations:—(1.) The conversion of metallic lead into lead oxide must only be effected after admixture of quartz sand or lead glass. Red lead must be mixed with quartz before melting. (2.) The furnaces in which such fusion is effected must be shut off from the workrooms so that no fumes can reach them. (3.) The flue must be so constructed that no injurious vapours escape. (4.) Grinding and stirring the glazing mixtures should not be done by hand. (5.) Children under 15 years of age should not be employed. (6.) The consumption of food and drink should be prohibited in the workrooms. (7.) The same regulations should apply to sleeping. (8.) The workshops must be provided with a good water supply and adequate sanitary arrangements. (9.) All workrooms must be roomy, bright, clean, well ventilated, and kept at a temperature of 16° C. (10.) Drunken or sickly persons should not be employed. (11.) All work in connexion with lead before its conversion into silicate should be carried on in a separate part of the works.—B. B.

*Direct Employment of Ceramic Colours with or without Glaze.* Deville, Pailette, and Co. *Monit. Céram. Verr.* 1890, 110.

SILICIC acid, which has been precipitated from a soluble silicate with hydrochloric acid is applied with a brush. The silicic acid combines with the metal and the colour. By an admixture of tribasic borate of lead the colour may be made lustrous while a dull appearance is best given by using silica powder. When the painting is to be glazed the article is burnt in a muffle-stove at a bright red heat, and then sprinkled with the following enamel mixture: 20 grms. of silica, 60 grms. of red lead, 50 grms. of borax, 10 grms. of saltpetre, 3 grms. of soda, and 1—1·2 grms. of zinc oxide. —A. R.

*New Porcelain and Glass Colours and their Fixation without Fire.* F. Babterrosses. *Deutsche Töpfer-Zeit.* 1890, 14, 187.

ORDINARY water colours are used, which are rubbed down with a particular mixture consisting of two solutions A and B. Solution A consists of 100 parts of a syrup of potassium water glass, to which has been added 10 parts of a lead acetate solution (15 parts lead acetate in 100 parts water) and stirred by an air stream until well mixed. Solution B consists of 50 parts of borax dissolved in 100 parts warm water and 20 parts of glycerol. Sixty parts of solution A and 40 parts of solution B are used in rubbing down the colours and for thinning, water and solution B in equal parts. After the articles have been painted they are placed in a bath made as follows: 1 part of borax is dissolved in 12 parts water and mixed in a gutta-percha flask with 50 parts hydrochloric acid, 10 parts sulphuric acid and 3 parts hydrofluoric acid. After the articles have been immersed 10 minutes in the bath they are taken out and washed with clean water. The colour appears as if burnt in.

—A. L. S.

*Decoration of Glass and Porcelain by Aluminium.* Gehring. *Monit. Céram. et Verr.* 1890, 21, 88.

ALUMINIUM is rubbed down with gum or oil and applied to the article to be decorated with a brush. The firing is done in a muffle and the heat slowly increased up to a red heat; the aluminium becomes first yellow, then brown, and finally white. Gold or enamel may be applied to the aluminium, or it may be allowed to remain matt or polished with a steel brush or an agate.—A. L. S.

#### PATENT.

*Improvements in Gas-fired Kilns for Burning or Heating Pottery and for other Purposes.* J. Holroyd, Leeds, and H. Wilson, Stafford. Eng. Pat. 3583, March 8, 1888. (Second Edition.) 8d.

See this Journal, 1889, 285.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

*Recent Improvements in Building and Paving Materials.* *Dingl. Polyt. J.* 276, 578—580.

E. RYAN has described in *The Michigan Engineers Annual*, a new kind of paving in use at Nashville, Tenn., which consists of ordinary stones soaked in coal-tar pitch set edgewise on a solid foundation of macadam and sand, and well rammed down; after four years' hard wear but little deterioration has taken place. The cost of preparing and laying the stones thus treated on a ready prepared surface is 1·8 dollars per square yard. A patented modification of this plan consists in covering the levelled surface of the pavement with 3—4 inches of sand, levelling this and covering it with tarred boarding, then spreading another even layer of sand about 1·5 in. in thickness, and placing on this the prepared bricks mentioned above, set edgewise and zigzag, and filling up all joints and crevices with another layer of sand. Besides this method two others are in use: in the first two layers of tiles are used, one set flat, the other on edge, and the whole filled in with sand; the other is the ordinary paving with broken stone or concrete; of these neither is as good as that previously described, and their cost is greater. In any case it is essential that good clay be used for making the tiles, it being necessary for it to be reducible to an impalpable powder when dry; a coarse clay of the right composition may be utilised by calcining and grinding. G. J. Snelus has analysed the ganister bricks of the firm *Grayson, Lowood, & Co.*, and finds them to have the composition  $\text{SiO}_2 = 95\cdot40$ ,  $\text{Al}_2\text{O}_3 = 3\cdot10$ ,  $\text{CaO} = 1\cdot68$  parts per cent. respectively. The lime is added as a binding material; the bricks do not expand at high temperatures.


—B. B.

*Magnesia in Portland Cement.* R. Dyckerhoff. *Chem. Zeit.* 14, 906.

THE author has already pointed out (this Journal, 1889, 781) the dangerous character of cements rich in magnesia, their tendency being to expand slowly after setting to the serious detriment of concrete or mortar in which they may be used. Particularly objectionable are they, inasmuch as the ordinary tests such as the "Darrprobe" (firing test) (this Journal, 1889, 781) fail to detect them, careful measurement of their expansion giving also only doubtful indications unless at least half a year has elapsed since gauging. He is disposed to fix the maximum permissible amount of magnesia in Portland cement at 4 per cent., and the Association of German Cement Manufacturers has formed a committee to report on the subject; such makers as are using dolomitic material being specially interested.—B. B.

## PATENTS.

*Improvements in the Method of and Apparatus for Building up the Inside of Kilns for Burning Enamelled Bricks and Tiles.* A. Taylor, Hipperholme. Eng. Pat. 13,036, August 19, 1889. 6d.

In burning enamelled bricks or tiles it has hitherto been the custom to support them in the kiln on large fireclay slabs resting on firebricks at each end; such slabs crack after once or twice using, and the object of the patentee is to avoid this inconvenience. The cracking being largely due to the weight of the bricks, the plan is adopted of substituting a number of slabs of the following shape  F, which are recessed so as to fit into each other and form a series of rectangular chambers in each of which a single brick is burnt.—B. B.

*Improvements in the Manufacture of Glazed Bricks and Tiles.* A. J. Clay, Stoke-on-Trent. Eng. Pat. 14,653, September 17, 1889. 6d.

ATTEMPTS have been made to manufacture glazed bricks from London clay, which have been unsuccessful owing to the non-refractory character of that material causing it to sinter or fuse below the melting point of the glaze. The patentee's process consists in adding 15–30 per cent. of silica in the form of sand or burnt and pulverised flints to the clay, the right proportion being ascertained by exposing trial mixtures in a kiln by the side of the glaze proposed to be used, the proper mixture agglomerating at a temperature above that at which the glaze melts. The mixture is pugged in the usual way, the bricks moulded, dried, and given a preliminary coating of a mixture consisting of 3 parts by weight of white lead, 2 of ground Cornish stone, and 1 of "Devonshire prepared potters best ball clay," and 10 parts of water. A suitable glazing mixture is then applied and the bricks fired.—B. B.

*Improvements in the Method of Burning Lime, and in Kilns therefor.* A. Bishop, Brockham. Eng. Pat. 14,997, September 23, 1889. 8d.

The patentee endeavours to combine to some extent the good qualities of the old fashioned "flare kiln," which produces lime of good colour, and those of modern continuous processes which are economical of fuel. The kiln is preferably built in the ordinary way so that the upper part just emerges from the ground. From the top of the kiln proper rises a cylindrical shaft, into which the charge of limestone together with a little fuel is tipped, and round the base of the main shaft and leading into the body of the kiln (which is roughly a reversed truncated cone) are smaller shafts (about eight in number), provided with covers, through which small charges of house coal siftings are from time to time introduced. The burning takes place in the ordinary way, save that each charge before reaching the zone of combustion is dried by the burnt gases, and that the flame given by the bituminous fuel put in at the side is drawn into the centre of the kiln and burns the lime much as in a flare kiln.

Air inlets formed in the walls of the base of the kiln are brought into use when a specially vigorous combustion is desired.—B. B.

*Improvements in the Manufacture of Plaster of Paris and in the Utilisation of Waste or Cheap Material therefor.* W. J. Oswald, Stoke-on-Trent; S. R. Ashwell and J. A. Cooke, Nottingham. Eng. Pat. 15,406, October 1, 1889. 4d.

Old plaster of Paris moulds, casts, or other waste, or artificial gypsum, are broken up in small pieces or formed into nuts and "burned" in kilns at a temperature between 95°–300° C. The kilns are provided with mechanical stirrers. The product so obtained possesses the property of setting with water.—H. A.

*An Improved Marine Cement.* F. C. Goodall, Richmond. Eng. Pat. 6711, May 1, 1890. 4d.

Forty parts of (melted) hard asphaltum, 40 parts of liquid asphaltum, 8 parts of boiled oil and 12 parts of finely ground corkwood intimately mixed constitute the cement.—K. M.

*Improvements in Bricks for Building Purposes.* D. Hart, Aberdare. Eng. Pat. 9239, June 11, 1890. 6d.

The patentee claims the manufacture of bricks with corresponding projections and depressions, interlocking in use and adding to the stability of the building of which they form part.—B. B.

## X.—METALLURGY.

*On Certain Ternary Alloys.* Parts I. and II. C. R. Alder-Wright and C. Thompson. Proc. Roy. Soc. 45, 461, and 48, 25–45.

THE authors, having had occasion to examine technically various mixtures of lead, zinc, and tin, noticed that whilst in certain cases these three metals could be fused together and intermixed so as to form a homogeneous alloy exhibiting no alteration on standing molten for some hours, this was by no means universally the case, especially with mixtures where the proportion of tin was materially below some 38 per cent. of the total mass; in such cases the fused mass separated into two dissimilar alloys, the heavier one mainly consisting of lead with some tin and zinc, and the lighter one chiefly containing zinc with some tin and lead. On preparing several series of mixtures where the proportion of tin gradually increased from 0 upwards, it was found that a variation of temperature of from below 600° C. to about 700° produced but little effect on the way in which a given mass divided itself into two alloys; but that at higher temperatures approximating to 800°, a measurable difference was discernible. On plotting curves with the percentages of tin in the heavier alloys as abscissæ, and those of zinc as ordinates, it was found that the solubility of zinc in lead-tin increased regularly as the proportion of tin present increased, the solubility curve at 800° distinctly overlying that at 650°. Similarly the solubility curves of lead in zinc-tin obtained in the same way with the lighter alloys exhibited analogous characters, the amount of lead dissolved by zinc increasing regularly as the quantity of tin present increased, and the solubility being uniformly greater at 800° than at 650°. A curious behaviour was noticed as regards the way in which the tin distributed itself between the two alloys; with small proportions of tin the lighter alloy contained most, the difference rising to a maximum when the entire mass contained 14 to 16 per cent. of tin; after which the difference diminished again until the entire mass contained 25 to 28 per cent., when both alloys contained the same percentage of tin. With still greater proportions the lighter alloy contained less tin than the heavier one; so that on plotting curves with the tin percentages in one alloy as abscissæ and the differences (+ or –) as ordinates, the curves rose from the origin to a maximum elevation, then fell again, and finally crossed the base line, and sank below it. It was found that the values of the ordinates were slightly variable with the composition of the total mass employed, although in all cases the character of the curve was the same; when the ratio of lead to zinc in the entire mass was 2:1, the curve underlay that obtained with mixtures where the ratio was 1:1; which again underlay that obtained with mixtures where the ratio was 1:2. On the other hand, whatever the ratio between lead and zinc might be, in every case the two alloys found corresponded with two points (*conjugate points*) lying respectively on the solubility curves of lead in zinc-tin, and of zinc in lead-tin, pertaining to the particular temperature employed.



Results in some respects similar, in others quite different, were obtained with other ternary alloys, the examination of some of which is still in progress. When lead and zinc are fused along with silver, or with cadmium, or with antimony, the masses separate into two different alloys when the three metals used are outside of certain limiting ratios with respect to one another; and the same remark applies to mixtures of bismuth and zinc, of lead and aluminium, and of bismuth and aluminium, with either tin or silver as third metal; but with some of these mixtures the solubility curves differ considerably from those observed with lead, zinc, and tin. Thus, in presence of tin, a given quantity of lead will take up considerably more zinc than it will if an equal weight of silver be substituted for the tin; and, similarly, a given weight of zinc will dissolve more lead in presence of a certain quantity of tin than it will if an equal amount of silver be substituted for the tin. Again, according to experiments still in progress, the way in which the tin divides itself between the heavier and lighter alloys is quite different with mixtures of bismuth, zinc, and tin from that above described with mixtures of lead, zinc, and tin; instead of the curve representing the results rising above the base line to a maximum and then descending again, it regularly sinks away from the base line: i.e., the heavier alloy always contains the most tin, the more so the greater the amount of tin in the total mass.

With mixtures of lead, zinc, and silver some remarkable numerical results were obtained, leading to the conclusion that two definite compounds,  $\text{AgZn}_5$  and  $\text{Ag}_2\text{Zn}_5$ , exist, the formation of which modifies the character of the solubility curves deduced. The first of these dissolves lead, and is itself dissolved by lead, to a greater extent than either pure zinc or the alloy  $\text{AgZn}_5$ ; so that when the lighter alloy formed contains silver and zinc in just the right proportions to form  $\text{AgZn}_5$ , more lead is dissolved therein than is the case with alloys either richer or poorer in silver. Moreover, this alloy is of an unstable character, so that if a saturated solution of lead therein be kept molten for a long time under favourable conditions, the mixture (at first perfectly homogeneous) gradually divides itself into two alloys, owing apparently to the breaking up of  $\text{AgZn}_5$  into free zinc and  $\text{Ag}_2\text{Zn}_5$ , the result of which is that more or less lead is thrown out of solution, sinking to the bottom and carrying with it a little zinc and silver. In the same way lead containing  $\text{AgZn}_5$  in solution, when kept molten for a long time, becomes changed; the solution, at first homogeneous, by-and-by forms two different alloys, because the breaking up of  $\text{AgZn}_5$  into free zinc and  $\text{Ag}_2\text{Zn}_5$  causes the former to be more or less thrown out of solution, so that it floats up carrying with it some silver and lead.

The other alloy,  $\text{Ag}_2\text{Zn}_5$ , is characterised by being even less soluble in lead than pure zinc (relatively to the zinc present); so that when lead is saturated with silver and as much zinc as will form  $\text{Ag}_2\text{Zn}_5$ , there is less zinc present than the lead could take up were the silver absent. Moreover, the solubility of lead in  $\text{Ag}_2\text{Zn}_5$  is less than in mixtures of this alloy with either  $\text{AgZn}_5$  or free silver. Further, the alloy  $\text{Ag}_2\text{Zn}_5$ , when exposed to the air for a short time after freshly scraping or filing, assumes a peculiar coppery hue; the presence of small quantities of lead or bismuth does not prevent this action taking place, nor does the tint become much modified if a little surplus zinc or silver be present, although larger proportions greatly reduce the shade.

Identically the same conclusions as to the existence of the two compounds,  $\text{AgZn}_5$  and  $\text{Ag}_2\text{Zn}_5$ , also appear to result from the examination of the alloys formed on intermixing bismuth, zinc, and silver, in some respects in an even more marked way. The detailed description of these results, like that of alloys containing aluminium and other ternary mixtures, is reserved for another occasion.

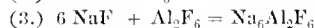
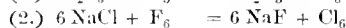
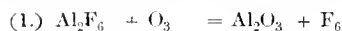
In the case of alloys of lead, zinc, and silver, the curve representing the distribution of the silver between the heavier and lighter alloys formed resembles in general character that similarly obtained in the case of lead, zinc, and tin; at first, with small proportions of silver, the lighter alloy contains by far the larger quantity of silver; so much so, indeed, that only inconsiderable amounts are retained by the lead, practically the whole floating up with the zinc, whence, obviously, the utility of the zinc process for

desilvering argentiferous lead. But with mixtures richer in silver this behaviour is less marked; as the silver in the total mass increases, the difference between the silver percentage in the two alloys increases less and less rapidly, until finally a point is reached when the difference attains a maximum, after which the silver in the heavier alloy increases more rapidly than that in the lighter alloy, causing the difference to diminish again. In other words, the curve first rises from the origin extremely steeply, then at a less rapid rate, until a turning point is arrived at, after which the curve descends again towards the base line. With tin as the third metal the curve finally descends below the base line; but with silver the descent is not carried so far as this.

Precisely analogous results appear to be obtained with mixtures of bismuth, tin, and silver.—C. R. A. W.

#### Artificial Cryolite and the Dissociation of Aluminium Fluoride. A. V. Asbóth. Chem. Zeit. 14, 868—869.

THE chemical analogies of magnesium and zinc suggest the utilisation of the cheaper metal as a reducing agent for such purposes as the production of aluminium from the double chloride of aluminium and sodium, and from aluminium fluoride. Experimenting in this direction, the author fused together common salt, aluminium fluoride, and zinc in a porcelain crucible, exposing the mixture to a blow-pipe flame for 15 minutes. After cooling, the melt was extracted with hot water, leaving some metallic particles and a rose-coloured powder. In the aqueous solution were found the chlorides of sodium, aluminium, and zinc. In the metallic particles no aluminium could be detected, and the rose-coloured powder appeared to be cryolite. Starting a fresh experiment, and endeavouring to gain more intimate admixture by first fusing the sodium chloride and dropping in small portions of aluminium fluoride, the author observed an evolution of gas of chlorous odour to take place with each addition, and abandoning the attempt to prepare aluminium, confined his attention to this reaction. On extracting the melt with water as before, the same rose-coloured powder remained undissolved, and on analysis proved to be impure cryolite. The following equations represent the mode of its formation:—



The method of analysis of the crude artificial cryolite was as follows:—0.3—0.5 gm. was weighed into a platinum dish and heated to dryness with strong sulphuric acid, the mass was dissolved in dilute sulphuric acid, the aluminium precipitated with ammonia, and the filtrate evaporated to dryness, ignited, dissolved in water, filtered, evaporated, and weighed as sodium sulphate. The fluorine was determined as  $\text{CaF}_2$  by the method due to Fresenius. Sulphuric acid was present in the substance as an impurity, probably as basic aluminium sulphate. The results of two analyses, with all the sodium calculated to cryolite, are—

	I.	II.
	Parts per Cent.	Parts per Cent.
Cryolite ( $\text{Na}_6\text{Al}_2\text{F}_{12}$ ).....	42.52	42.51
$\text{Al}_2\text{F}_6$ .....	8.42	16.14
$\text{Al}_2\text{O}_3$ .....	38.21	39.84
$\text{Fe}_2\text{O}_3$ .....	7.38	7.38
$\text{SO}_3$ .....	4.03	4.15
	100.56	101.02*

\* 101.03 in original.

The analyses are of different specimens prepared differently, and in both cases without weighing the ingredients before fusion. More cryolite appears to be formed when zinc is present. The presence of alumina as shown

above may either be due to the oxidation of aluminium momentarily reduced by the zinc, or to the decomposition of aluminium fluoride by the oxygen of the air, in which case free fluorine must result. The impure artificial cryolite is amorphous, partially fuses into granules at a high temperature, and becomes white.—B. B.

*Aluminium in Iron.* H. M. Howe. Eng. and Min. Jour. 50, 213.

THE writer points out that the remarkable effect produced by the addition of a minute quantity of aluminium in enabling extremely soft ingot iron, very low in carbon, to be run into very thin and difficult castings, is not due to the lowering of the melting point. F. Osmond has shown that an ingot iron containing 0.10 per cent. of carbon and 5 per cent. of aluminium begins to melt at 1465°, only 25° lower than that of soft steel. All that aluminium can do, Osmond believes, "is to reduce the oxide of iron, make a thin slag, and so enable the purified metal to run more fluid." A sample of spiegeleisen, to which 3 per cent. of aluminium had been added, underwent a remarkable change, the characteristic broad tabular fracture gave place to a fine granular fracture resembling No. 3 pig iron. The analysis of the sample before and after the addition of the aluminium showed that the combined carbon had been reduced from 4.8 to 0.93 per cent. and that the graphitic carbon had increased from nil to 3.45 per cent. The effect of the aluminium in this case is therefore similar to that of silicon in driving the carbon out of combination, and is opposed to the action of manganese, which, like chromium, increases the solvent power of iron for carbon. The great change in its affinities, which aluminium seems to undergo rather rapidly at high temperatures, leads to the question whether, like iron, nickel, and cobalt, aluminium may not be found to possess a distinct critical point in which the specific heat changes abruptly, with accompanying marked thermal phenomenon.

Accumulating facts seem to indicate that the broad tabular fracture of spiegeleisen is not due directly to the presence of manganese, but to the large proportion of combined carbon which that manganese permits the iron to retain. For ferro-chrome, whose composition resembles that of spiegeleisen only in its high proportion of combined carbon, has a fracture almost identical with that of spiegeleisen. The high proportion of combined carbon in the cast iron for the basic Bessemer process whose fracture more closely resembles spiegeleisen the more carbon it contains, is probably chiefly due to the low proportion of silicon present; and as the lower the silicon the better the iron for the basic process, so it may be graded by its fracture, the best iron resembling spiegeleisen the most closely. A sample of pig with the characteristic spiegeleisen fracture contained 4 per cent. of combined carbon, 0.02—0.05 per cent. of silicon, and 0.20 per cent. of manganese, 2.7 per cent. of phosphorus, 0.03 of sulphur. A fact which may bear on the explanation of the Mitis process is that aluminium added to molten steel quieted the metal completely and caused it to pipe deeply. Little white specks were found on the surfaces, which proved to be alumina. The aluminium appears to reduce any iron oxide present.

—J. B. C.

*The Imperial Silver and Lead Works at Příbram.* A. Zdráhl. Jahrb. derk. k. Bergakademien, 1890, 38, 1; Chem. Zeit. Rep. 14, 185.

THE fire bridges of the furnaces used in roasting the ore have, until recently, been cooled by water. This is now done by a stream of air which is conducted through a cast-iron conduit in the fire bridge. The air emerges from this at a high temperature and is passed into the fire, thus producing a higher temperature than if cold air were used. If the fire bridge is coated with magnesia, less repairs are needed. Care is taken to allow none of the fumes to escape, they being condensed in long flues. Flue dust, ash, and rich marl is used as a flux in roasting the ore. The sulphur is reduced on an average from 12 per cent. to 0.9 per cent. The lead ore is roasted in open kilns built of slag. As the

slag if untreated would fly on heating, slag bricks are prepared by melting the slag and mixing this with angular pieces of old slag and pressing into shape. The furnace charge is usually 10 t. roasted ore, 12 t. of slag, 1 t. of tuff, 1 t. of roasted lead metal, 2.9 t. of dross (litharge and such like), 1.4 t. of lime, 0.4 t. of cinder, 0.3 t. of cast iron, 1.5 t. of finery slag. 1,300 kilos. of this charge requires 150 kilos. coke, and 4 h.l. of charcoal, or 120 kilos. coke and 6 h.l. of charcoal, and usually 24 charges are made every 24 hours. The slag contains 28—30 per cent. of silica, 36—37 per cent. of ferrous oxide, 9—11 per cent. of lime, 4—5.5 per cent. of alumina, and 9—10 per cent. of zinc oxide, and usually not less than 2 per cent. of lead, and 0.025 per cent. silver. The lead metal contains 41 per cent. iron, 11 per cent. of lead, and 11 per cent. of zinc. In the cupellation furnace a marly bottom is made over the ordinary bottom and only the first is replaced for a fresh cupellation.

The loss of metal is given in the following table on 100 parts ore (a) and on 100 parts finished metal (b).

Lost in	Silver.		Lead.	
	a.	b.	a.	b.
Roasting and melting.....	0.878	0.73	9.151	4.85
Liquation .....	0.155	0.14	0.658	0.41
Pattinsonising .....	0.144	0.31	0.749	0.99
Refining and remelting .....	..	0.60	0.199	0.63
Capellation and refining...)	0.819	0.71	5.919	4.33
Melting the litharge .....			0.580	3.18
Hard lead melting .....	0.002	100.00	0.400	5.51
In the litharge sold .....	0.300	..	..	..
In the lead sold .....	0.063	..	..	..
	2.361		17.656	

—A. L. S.

*On the Amount of Phosphorus in Wrought Iron.* A. Ledchur. Stahl. u. Eisen, 1890, 10, 513.

AN equal amount of phosphorus is injurious to a different degree in different irons, being much more so to ingot iron, which cannot stand more than 0.2 per cent., than to wrought iron, which may contain up to 0.4 per cent. This is probably partly because the latter contains 0.1 per cent. in the slag which exists in the metal. In the case of steel rich in carbon, which is in itself often *short*, the presence of phosphorus must be particularly injurious.

On the other hand, there are many irons, such as that prepared by the Clapp-Griffiths process, which are proportionally unaffected by phosphorus, so that it cannot be said that with our present knowledge we are able to correctly estimate its effect.—A. L. S.

*The Action of Zinc on Dilute Sulphuric Acid.* F. Pullinger. J. Chem. Soc. 1890, 815—827.

THE results proved are as follows:—

1. Pure zinc with a perfectly smooth surface is not acted upon by dilute sulphuric acid which has been submitted to prolonged boiling.
2. Pure zinc with a rough surface is readily acted on, but in a less degree, by acids which have been boiled than by those which have not.
3. Oxidising agents, such as electrolysed sulphuric acid, hydrogen peroxide, and nitric acid, increase the rate of dissolution.
4. A reducing agent, such as hydriodic acid, almost entirely prevents solution, but those containing sulphur, such as sulphur dioxide, are without effect.

5. It is not improbable that when zinc with a rough surface dissolves in dilute sulphuric acid, persulphuric acid, acting catalytically, is the cause of the dissolution.

6. In all probability, pure dilute sulphuric acid would, at ordinary temperatures, be entirely without action upon metallic zinc, whether the surface of the latter were rough or smooth.

*On the Action of Sulphurous Acid on Metals.* J. Uhl. Ber. **23**, 2151—2154.

See under XXIII., page 970.

*The Metallurgical Treatment of Cupriferous Pyrites in the District of Huelva.* L. de Launay. Annales des Mines, 1889, XVI. 427; Proc. Inst. Civil Eng. **101**, 374.

THE author, after describing the geology and structure of the great pyrites mines of San Domingos, Tharsis, Rio Tinto, and others, proceeds to consider the methods adopted for exhausting the copper. These are classified as follow:—

1. Natural cementation.
2. Artificial cementation.
3. Artificial cementation with chlorides.

The first of these methods, which is essentially one of atmospheric oxidation, is practised at San Domingos, in Portugal, where the burning of pyrites in open heaps is forbidden by law. The ore, a cupriferous iron pyrites containing from 2.5 to 3.0 per cent. of copper, is broken into lumps of the size of the fist, and spread out on floors in heaps varying from 15 to 40 ft. in thickness. Deep trenches are cut through the heap at regular intervals for the escape of drainage water, and a series of holes lined with earthenware pipes are sunk into it at the top, for the purpose of inserting thermometers when it is desired to ascertain the temperature in the interior. Water is distributed over the surface by a rectangular network of pipes with jets, and the operation is continued for several years, about 3,000,000 tons being under treatment at one time. The sulphates formed by the joint action of air and water dissolve in the latter, and are collected in channels leading to the cementation tanks. The surface of the heap is broken up by a pick from time to time to prevent the crust of sulphates from becoming compact and impermeable to water. The operation, though extremely simple in principle, requires somewhat delicate management, especially in regard to temperature. If the heap is too cold the sulphide of copper is not attacked, while if too hot, an excessive production of steam prevents the access of air and the consequent oxidation of copper, while a larger proportion of iron is dissolved. The best results are obtained at 100° F., a heat that is maintained by varying the amount of water. It is also essential that the solution shall be as rich in copper sulphate as possible, and if the liquors are too poor, the watering of the heap is sometimes suspended from 15 days to a month.

When the proportion of copper in the heap is reduced from 2.8 to 0.25 per cent., the heap is broken up and exported for use as a sulphur ore, the process having the advantage of saving the bulk of the sulphur. The quantity of water required being very large, two storage reservoirs have been built for holding it, one being of 5,000,000 and the other of 1,500,000 tons capacity, the latter serving also for irrigation and domestic supply.

The liquors obtained from watering the heaps are led into the cementation tanks, which are asphalted basins 13 ft. square, arranged in 10 series of 24. These are filled with pig iron piled ehequewise, and the coppery liquors are run through them in such a manner as to obtain the most complete precipitation with a minimum consumption of pig iron. The precipitated copper or *casera* deposits on the surface of the pigs, and is cleaned off at intervals of a month. The deposit, consisting of a mixture of copper with graphite, and iron rust, containing about 65 per cent. of copper, is dried upon a heated surface and packed in bags of 100 lb. for export.

The consumption of pig iron, which theoretically should be only 0.83, is actually 2.0 per unit of copper obtained.

This is due to the formation of ferric sulphate in the liquors, which must first be reduced to ferrous sulphate by the iron, before any copper can be separated.

The exhausted liquor, essentially a ferrous sulphate solution, is stored in tanks, which, to avoid permanent poisoning of the river, are only emptied during the period of spring floods.

*Artificial Cementation.*—This term is applied by the author to the method of burning the pyrites in heaps as a preliminary to extraction by water and precipitation, which is in general use in Spain, although at present attempts are being made to abolish it on account of the damage done by the sulphur gases. At Lagunazo, near Tharsis, where the ore contains from 2 to 3 per cent. of copper, with about 3 per cent. of arsenic, 2.3 oz. of silver, and about 1½ dwt. of gold per ton, it is burnt in heaps called *teleras*, in a similar manner to that subsequently described as in use at Rio Tinto, for about two months.

The solution of the copper is effected in two stages, it having been found that ferric sulphate liquors will remove from one-eighth to one-fifth of the copper in the first 10 days. The burnt ore is subjected to a first washing in tanks for 12 hours, followed by a second of 24 hours, and a third of 48 hours, after which it is piled into heaps called *terrerros*, where the residual copper is extracted by watering, an operation which, like that in the natural cementation process, lasts for several years. The cementation is effected in tanks, through which a very rapid current is maintained, the iron being turned out every 10 days; the amount of iron consumed is 1.7 per unit of copper obtained. The *casera* when dried and roasted in a reverberatory furnace, an operation which requires about 4 per cent. of its weight of coal, is packed in bags for export.

The deposit at Lagunazo was only opened in 1882. It now produces about 120,000 tons of pyrites annually. About 1,000 tons of copper are obtained from 80,000 tons of burnt ore under treatment.

*Artificial Cementation with Chlorides.*—In this method, which is practised at Rio Tinto, the copper is dissolved, not as sulphate, but as chloride. This has the advantage of reducing the waste of iron in the precipitation tanks, by avoiding the formation of ferrous and ferric sulphates, as the solution of the sulphide of copper is effected by means of ferric chloride, while iron pyrites is but slightly acted on. By this means a ton of copper precipitate may be obtained with an expenditure of an equal weight of pig iron.

The produce of the mine is divided into five different classes, each having its own method of treatment. These are:—

1. Smalls, obtained by sifting the larger sizes. These are subjected to a modification of Doetsch's process of washing by ferric chloride, as they pack too close to be converted on sulphates by atmospheric oxidation.

2. Ordinary ores with less than 2.75 of copper. These are burnt, watered, lixiviated, and precipitated.

3. Ores of 3.75 and 4 per cent. average, obtained when necessary by the mixture of other kinds. These are sent by rail to the pier at Huelva and shipped, those of 3.75 per cent. going to England, and those of 4 per cent. to Germany.

4. Rich ores above 6 per cent. These are unfit for calcination, and are therefore smelted for regulus in water-jacket cupolas.

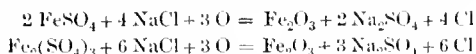
5. Minerals containing copper and lead. These are put aside as giving trouble in the ordinary working, and have not as yet been utilised.

*Treatment of the Smalls by the modified Doetsch Process.*—This, which like the method of natural cementation at San Domingos, does away with roasting, but reduces the loss of time and iron by adding salt, is applied in practice with the following modifications:—

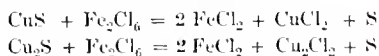
On the side of a hill below the *teleras*, or roasting heaps, there are five series of tanks, each series consisting of seven tanks 15 metres long, 4 metres wide, and 1½ metres deep, which are divided into eight compartments by walls of perforated bricks, placed edgewise and covered by planks. At a lower level is placed another series of four larger tanks, 100 metres long, 2 metres broad, and 1½ metres deep.

The whole are used indifferently for the treatment of the smalls, and for the first washing of the burnt ores, which extracts about one-fifth of their contents of copper, before sending them to the *terreras* at Naya. The raw smalls are mixed with a proportion of burnt ore in lumps of the size of a walnut, and washed with liquors brought from the large *terreras* containing a certain quantity of common salt. This operation lasts about eight days; the wash-water going to the precipitation tanks, while the partially exhausted mineral is taken to the *terreras* at Naya. The reactions in this method appear to be as follows:—

The burnt ore having been subjected, at a high temperature, to the action of oxygen and chloride of sodium, gives rise to—



The chlorine liberated, acting on the sulphides of iron and copper, produces ferric and cupric chlorides. There are therefore present  $\text{FeSO}_4$ ,  $\text{Fe}_2(\text{SO}_4)_3$ ,  $\text{CuSO}_4$ ,  $\text{Fe}_2\text{Cl}_6$ , and  $\text{CuCl}_2$ , with an excess of salt which changes the ferric sulphate into chloride. The copper in the ore, existing partly as cuprous sulphide  $\text{Cu}_2\text{S}$ , and partly as cupric sulphide  $\text{CuS}$ , the reactions become—



the ferric chloride attacking and dissolving the copper, while the iron pyrites is almost untouched. The final separation of the copper is effected by cementation in the manner previously described.

#### *Treatment of Ores with less than 2.75 per Cent. of Copper.*

—This, the method of artificial cementation with chlorides, is applied to the largest part of the output of the mine. It includes the four operations of roasting, washing, precipitation, and cleaning of the precipitate, or *cascara*.

The roasting is performed in *teleras*, which are heaps of a truncated pyramidal form, 3 metres high, on a base of 6 by 8 metres for those of 800 tons, and 9 by 8 metres when containing 1,200 tons. At the bottom, one transverse and three longitudinal flues, about 20 in. square, are formed by the larger blocks of mineral. These are the firing passages, and communicate with vertical chimneys, of which there are two in the 800 ton, and three in the 1,200 ton heaps. The mass of the heap is made up of lumps a little above nut size, salt being added in the proportion of 14 tons to 800 tons of pyrites. When the fire has been started with a little wood, it is kept up by the heat of the burning sulphur. It is essential that no rich ore shall be included, as, on account of the action of "kernel roasting," lumps of rich sulphide of copper are formed, which cannot be subsequently dissolved; and as the mass of material is so large that hand-picking is not easy, great loss of copper is likely to ensue. It need scarcely be said that the operation causes an enormous nuisance, as, since the introduction of the use of salt, the volatile products contain chlorine as well as sulphurous acid, and, especially during the prevalence of easterly winds, the village of Rio Tinto is almost uninhabitable from the sulphur smoke, while all vegetation is destroyed to a distance of six leagues around. This is now sought to be mitigated by removing the roasting heaps to a greater distance—at Planes, where large lead chambers are under construction for the purpose of saving from the sulphur gases the sulphuric acid required for cleaning the copper precipitate, and which has hitherto been purchased abroad. A more important application has, however, been found in using sulphurous acid gas for promoting the formation of sulphates in the ore, which is now carried out on a very extensive scale at Naya.

In this method, the *teleras* made in the ordinary way, as soon as they begin to give off sulphurous acid, are covered up with a fresh quantity of mineral, partly raw and partly calcined, to which 2 or 3 per cent. of salt, and a similar proportion of peroxide of manganese have been added. The whole is formed into a heap 8 in. high with a flat top, which is divided by gutters into squares of 8 metres in the side. The remaining operations are effected in the usual way, the *teleras* being watered at intervals for months and years, the

copper being slowly dissolved, and collected at the bottom of the heap. It is necessary to break up the surface with a pick at intervals, to prevent its becoming impermeable to water.

The sulphurous acid gas in this modification of the process, in the presence of steam formed by the heat developed in the heap, produces sulphuric acid, which acts upon the oxides in the crust of the roasted minerals. The salt and manganese ore may, jointly with the sulphuric acid, evolve chlorine, forming ferric chloride, which decomposes the sulphides of copper and silver. It is also possible that, under the action of heat and sulphuric acid, oxygen is evolved, which acts directly upon the pyrites.

The precipitation of the copper is effected at La Cerda, near Rio Tinto, in a series of tanks 100 metres long and 30 metres broad, divided into 10 parallel series, receiving a uniform supply. The total length is about 400 metres, with a difference of level of 4 metres, giving a sufficiently rapid flow. Pig iron as run from the furnace and scrap are used, the latter being put into baskets. Every 10 days the iron is removed, and, after scraping to collect the deposited copper, is returned to the tanks. The consumption is reduced to a little more than 1 ton of pig iron per ton of precipitate produced.

The *cascara* precipitate as collected is very impure, containing only 65 to 70 per cent. of copper, the remainder being ferric oxide, more or less arsenical, graphite from the pig iron, silica, &c. After treatment with water acidulated with sulphuric acid, which dissolves the basic ferric arseniates without touching the copper, it is passed over sieves of  $\frac{1}{16}$  in. mesh to separate any adherent masses of cast iron, which are hand-picked, and any copper separated is subjected to another washing with acid. The *cascara* passing through the sieves is washed in a current of water, where it separates the order of density into *cascara*, or copper; *grafita*, or particles of coal and graphite; and *pucha*, a fine black sand. The *cascara* is subjected to a fresh washing upon a fine wire-gauze screen, which separates a further quantity of graphite, and this passes to a series of plunger jiggling machines, where it is finally cleaned. The graphite is collected and returned to the rich ore smelting furnaces.

The *pucha* is washed upon convex round buddles 15 ft. in diameter, by which a certain quantity of fine copper is saved. The waste is made into balls, which are dried in heaps and also sent to the smelting furnaces.

The silver in the pyrites usually remains in the *terreras*, a small proportion only passing into the copper precipitate.

The rich ores containing more than 4 per cent. of copper are, after burning, smelted in cupolas or water jacket furnaces of an American pattern, having a large exterior fore-hearth or receiver in which the separation of slag and regulus takes place. The charge consists of burnt ores, old slags, a certain proportion of raw pyrites, quartz, and graphite. The regulus, which is exported to England, contains from 38 to 40 per cent. of copper; the slags with 0.4 per cent. are thrown away.

#### PATENTS.

*Improvements in Apparatus for Deoxidizing, Dephosphorizing, Desulphurizing, and Treating Iron and other Ores, and for Collecting and Burning the Fumes and Gases therefrom.* M. R. Conley, Brooklyn, and J. H. Lancaster, New York, U. S. A. Eng. Pat. 8678, May 24, 1889. 8d.

The process is carried out in a series of cylindrical retorts placed in a furnace. The retorts are filled through vertical pipes arranged in a line, so that they may be charged successively from a suitable hopper travelling on rails. Each of the retorts is provided with a discharge chute, which extends through the rear wall of the combustion chamber, and their lower ends are approximately upon a level. The contents of the retorts may be discharged directly into an open-hearth furnace or into an air-tight box, and, when sufficiently cooled, mixed with pitch, so as to withstand exposure to atmospheric air. To desulphurise, &c. the ore, air, hydrogen, or other gas may be admitted by suitable means, and the fumes conveyed to a gasometer, and

thence to the furnace for heating the retorts. Each of the retorts is provided with a rotary stirrer, extending longitudinally. The shafts of these stirrers have bearings at their rear ends in sockets. Outside the front ends of the retorts the stirrers have secured upon them sprocket wheels. Upon this sprocket wheel moves a sprocket chain extending about the sprocket wheels and also about a suitable pulley. The sprocket wheels are so arranged that the sprocket chain, as it passes to and fro, will engage with the teeth on the wheels, and will cause all the stirrers to rotate in unison. In order to discharge the sprocket chain from any one or more of the wheels, levers are fulcrumed on the front of the combustion chamber upon the bent arm of which are attached friction rollers. The chain passes between these rollers, so that by raising the lever the chain is raised above the wheel, which is thrown out of gear. The shafts of the stirrers have teeth curved longitudinally, and are wider at their free ends. They are also provided with longitudinal strengthening ribs. At the ends of the teeth there are formed on them horizontally-extending flanges, through which extend transverse apertures. The teeth extend radially from the shaft in three directions, certain of the teeth being in line with each other, and the others at right angles, so that in discharging, the teeth may be turned so as not to obstruct the bottom of the retort. Each retort is provided with a scraper, which, when not in use, is braced against the end of the retort. It is released from outside, and can be moved freely inside the retort, without admission of air.—J. B. C.

*Improvements in and connected with the Utilisation of Sulphate of Iron Galvanising Pickle.* T. Parker and A. E. Robinson, Wolverhampton. Eng. Pat. 10,554, June 29, 1889. 4d.

The waste pickling solution is evaporated in contact with iron in order to neutralise any free or uncombined acid to a specific gravity of about 1.4 or 1.5. "The evaporation is preferably conducted in iron vessels in which scrap iron or iron borings or turnings are contained, and by means of artificial heat. To the concentrated neutral pickle thus obtained, is then added a sufficiency of strong sulphuric acid to precipitate the iron. The supernatant solution may be drawn off or otherwise separated, as is well understood, and employed in the preparation of a second pickling solution, or be otherwise utilised. The precipitate consists of anhydrous sulphate of iron, and this is distilled and manufactured into fuming or Nordhausen sulphuric acid in the ordinary manner."—J. B. C.

*Improvements in Roasting certain Ores in order to obtain Metals therefrom, and in Means or Apparatus employed therein.* S. Trivick, London. Eng. Pat. 11,882, July 26, 1889. 8d.

THIS invention relates to the construction of a furnace to effect the roasting of ores sufficiently rich in sulphur to support combustion without additional fuel. This furnace contains a series of hearths superposed one above another upon a bed. These hearths are formed of firebrick, communicating by openings with the one above and the one below. The top hearth is arched over, the arch containing an opening for the admission of the ore. At the front of each chamber is a door, adjustable in its position so as to admit more or less air, and for the introduction of rakes for moving the ore from floor to floor. "At each of the sides of the chamber is an opening, preferably towards the front, through which heated air enters and aids combustion of the sulphur;" and "this air is conducted and heated in its conduction by means of passages carried along the side and up over the fire chamber, joining other passages leading down behind, and then in front under the bed or through it to the front of the furnace. The products of combustion of the sulphides pass through the series of chambers and away from the uppermost by openings leading to a middle flue passage below a heating chamber," and then to the

stack. "The heating chamber provided rearward of each furnace and over the flue chamber, serves, when packed with sand, to contain tubes suitable for heating leaching or other fluids used in the subsequent treatment of the ores."

—J. B. C.

*Improvements in the Production of Aluminium and in Apparatus employed for that purpose.* H. Bessemer, jun., Westend. Eng. Pat. 12,033, July 29, 1889. 8d.

THIS invention consists in the use of combustion under pressure in order to secure a temperature sufficiently high to effect the reduction of aluminium oxide by carbon or gaseous hydrocarbons, and in a method of rapidly cooling the aluminium vapour produced, by allowing the gaseous products to issue through a small aperture into a second chamber where they expand and become cooled. A mixture of aluminium oxide and carbon is made into briquettes, and is charged into a strong iron vessel having a refractory lining and cooled externally by means of water. Air, previously heated by a regenerator, and gaseous fuel are forced in at a pressure of 3 or 4 atmospheres with the result that a very high temperature is produced attended with the reduction of the alumina. The aluminium vapour and products of combustion escape at the further end of the vessel through a small fireclay tube, whose size is so adjusted that the pressure in the combustion chamber is not impaired by an undue escape of the gases. The rate of flow of the latter is further controlled by an adjustable plug which partially closes the end of the tube. The gases escape into a cooling chamber communicating with the atmosphere. The aluminium condenses and the products of combustion pass onwards.—H. K. T.

*An Improved Process of and Apparatus for Galvanising Metal Strips or Ribbons, Wire, and the like.* N. Vilart, London. Eng. Pat. 14,297, September 10, 1889. 8d.

THE ribbons or wires are drawn successively through a solution of potash, dilute acid, molten zinc, and water, being in each instance made to dip into the bath by passing over rollers and under drums. After leaving each bath they are passed through two cushions or bolsters of linen or asbestos, which clean them from the material of the one bath before passing into the next, and which, in the case of the zinc, regulate the thickness of the adherent metal. "As pure zinc is not sufficiently fusible it is generally mixed with about 5 per cent. of tin." The galvanised strips are, by this process, rendered capable of corrugation, or of manufacture into flexible metallic pipes, without the zinc stripping off. Seven drawings are given in the specification showing the arrangement of the appliances.—A. W.

*Apparatus and Process for Condensing the Metallic Vapours produced in Zinc Furnaces, and Furnace applicable in Combination with the said Condensing Apparatus.* E. Grützner, Loslau, and O. Koehler, Czernitz, Germany. Eng. Pat. 15,245, September 27, 1889. 8d.

THIS apparatus consists of a sheet-iron condensing chamber and a zig-zag settling chamber, open at the bottom, both submerged in water. The retorts are hermetically connected with the condensing chamber, in which a partial vacuum is produced by pumping out a portion of the water with which it is at first filled. The vacuum is afterwards maintained by connecting the exit end of the settling chamber with the furnace or its chimney. A furnace is described containing a battery of four or six vertical retorts surrounded by spaces for the circulation of furnace gases. The retorts are charged from the top, the residue after distillation being removed by detaching the bottoms of the retorts, which are held in position by clamps.—H. K. T.

*Improvements in the Manufacture of Alloys of Nickel with other Metals.* H. Marbeau, Paris, France. Eng. Pat. 16,318, October 16, 1889. 6d.

THE purpose of this specification is the production of an alloy of nickel, iron, and manganese, containing carbon, which is therein called "nickelo-spiegel," to be used to convert iron into ferro-nickel or steel-nickel. It is prepared by reducing together nickel, iron, and manganese ores, the following proportions being given as an example:—

Nickel ores (10 per cent. of Ni).....	2 tons.
Iron ores (50 per cent. of Fe).....	12 cwt.
Manganiferous iron ores (10 per cent. of Mn and 40 per cent. of Fe) .....	1 ton.

—A. W.

*Process for obtaining Salts of Thorium, Zirconium, and like Metals from their Ores or from Waste Residues of Hoods used for Incandescent Gas Lighting.* O. Imray, London. From W. MacKean, Paris, France. Eng. Pat. 16,555, October 19, 1889. 6d.

See under VII., page 941.

*A New or Improved Universal Furnace for the Fusion and Refining of Metals, Alloys, Dross, by the Dry Process or otherwise.* J. B. Alzugaray, Porto, Portugal. Eng. Pat. 16,776A, October 24, 1889. 8d.

THIS is a vertical furnace of octagonal shape having a crucible at its centre. The latter is provided with a discharge pipe and with a hopper passing through the top of the furnace.—H. K. T.

*Improvements in the Treatment of Waste Pickle from Galvanising Works and other such like Acid Liquors, and in the Apparatus employed therefor.* T. Turner, Birmingham. Eng. Pat. 17,074, October 29, 1889. 8d.

THE acid liquor is evaporated in an open roaster, the bed of which is made in the form of a shallow basin. The liquor is fed in this as fast as it is decomposed. After the liquor has attained a pasty condition, it is conveyed to the hottest (dull red) part of the furnace, where the iron chloride in contact with air and the acid vapours yields iron oxide and hydrochloric acid; this is condensed and used over again for galvanising purposes.

In order to render the furnace liquor-tight, a layer of sulphur, puddle, asphalt, &c. is inserted a few courses above the ground-line. In starting the furnace the liquor will permeate in the brickwork and cement it, and in the course of evaporation a protecting layer of iron chloride will be formed on the furnace bed. (See also this Journal, 1890, 809.)—H. A.

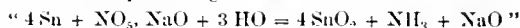
*Improvements in Apparatus for Separating Precious Metals by Amalgamation from Ores or Materials containing them.* R. E. Shill, London. Eng. Pat. 20,017, December 12, 1889. 8d.

THE apparatus consists of an inverted funnel-shaped vessel, whose base is perforated with holes, into each of which a short tube is fixed. These tubes pass under the surface of mercury contained in the base of another but larger conical vessel which surrounds the smaller one and at the top of which there is an outlet pipe. The pulp enters the tube of the inner vessel, passes downwards through the small tubes, through the mercury upwards into the annular space between the two vessels, and out at the exit at the top of the outer one. It is stated that the materials being thus forced under the surface of and through the mercury the float gold will be caught. The sulphides do not accompany the tailings, but fall on to the surface of the mercury, whence they are gradually worked by the agitation of the same into an annular space around the base of the outer vessel provided for the reception of them. The tailings, if they contain

gold, are passed through another similar vessel, or through another contrivance based on the same principle of forcing the materials through liquid mercury.—A. W.

*Improved Process for Separating Tin from other Metals more particularly applicable for the Treatment of Tin Plate Scrap and old Tinned Cases.* I. Bang, and A. Rudlin, Paris, France. Eng. Pat. 70, January 2, 1890. 6d.

THE tin is dissolved by means of caustic soda or potash lye, in presence of sodium nitrate or a suitable oxidising agent, the object of the latter being to hasten the solution of the tin by removing the film of hydrogen, formed by the reaction, from the surface of the material treated. The lye should be of a strength of 15° B., and should contain a proportion of nitrate largely in excess of the quantity needed to dissolve the tin and required by the following equation, given by the patentees as representing the reaction—



Caustic soda and ammonia are thus obtained while the tin is in solution as sodium meta-stannate. By the addition of bulk of lime, calcium meta-stannate is precipitated, and the clear alkaline solution is ready for use again.

Pure metallic tin is easily obtained by fusing the calcium meta-stannate with carbon and a flux.—O. H.

*Improvements in and connected with Furnaces for Recovering Tin from Tin Slag.* J. Letcher, Devonan. Eng. Pat. 5608, April 14, 1890. 6d.

ACCORDING to this invention the furnace in which the tin slag is smelted consists of a vertical chamber constructed of fireclay, bricks, or other suitable material. Air is admitted to the furnace through apertures provided in the walls at a convenient and suitable height from the base. Below the level of the air apertures and between them and the base is an aperture which, while the melting process is in progress, is blocked by fireclay. At intervals, the fireclay blocking the aperture is pierced and the metal run out through the hole made therein. At the top, the furnace communicates by a throat with a long horizontal flue, which communicates with a chimney stack. "By the employment of a long horizontal flue communicating with a stack, a draught is maintained sufficiently powerful to melt the metal in the furnace without the waste of coke, employed as fuel, obtaining in furnaces, where a blast or jet of steam is directed into the furnace." The long flue also serves as a means for receiving particles of metal deposited from the fumes passing through the flue on the way to the stack.—J. B. C.

*Improvements in Converters.* A. J. Boulton, London. From J. W. Bookwalter, Cologne, Germany. Eng. Pat. 5894, April 18, 1890. 8d.

IN order to reach the highest results in the rapid mode of conversion, the air blasts should be so applied to the metal in the bath that the returning currents of metal shall not be upon the surface towards the tuyeres, for the scoria floating upon the surface of the metal would thus be drawn with these return currents into the area of violent agitation, and become remixed with the metal. The returning currents of metal must be drawn from a comparatively quiescent body of metal, and from such a depth below the surface that there shall be no tendency to carry with the metal any of the impurities formed in the process.

This is attained by applying a series of tuyeres at one side only of the metal in the bath, arranged in a straight line and parallel with each other, so as to act upon the metal, near the surface or, instead of a series of tuyeres, one continuous sheet of the air blast corresponding to the width of the metal in the bath may be applied.

By this arrangement, surface currents in the metal are avoided, which tend to carry the floating scoria back to the mouth of the tuyeres and to mix together metal and scoria



In order to renew the tuyeres and lining, which are rapidly corroded, the converter is constructed of two parts, the upper portion being fixed or attached to trunnions, and the lower being detachable and containing the metal bath and tuyeres. The arrangement is shown in drawings attached to the specification.—J. B. C.

*An Improved Process for Treating Copper and its Alloys to prevent Oxidation and Decoloration thereof during Heating or Annealing.* H. H. Lake, London. From G. W. Cummins, Vienna, U.S.A. Eng. Pat. 8989, June 10, 1890. 8d.

This process consists in heating or annealing copper in a closed vessel filled with inert gas. The vessel consists of a horizontal tube closed with a plug at one end and communicating at the other end by means of an elbow-piece with a vessel of water. The vessel is first filled with steam or other inert gas, the copper articles are then introduced, and when sufficiently heated are raked into the cooling liquid. By this means oxidation is prevented, a matter of importance when dealing with copper wire required for electrical purposes. The use of this apparatus also prevents the reduction of oxides of arsenic and antimony which are generally present in copper. Those oxides frequently become reduced when copper is heated to a high temperature in contact with products of combustion, and render the metal brittle.—H. K. T.

*Improvements relating to the Manufacture and Treatment of Iron and Steel, and to Compounds for use therein.* H. H. Lake, London. From J. D. Brown, New York, U.S.A. Eng. Pat. 8991, June 10, 1890. 4d.

This is a compound for welding, toughening, or refining steel, and consists of "borax 33 parts, pumice-stone 8 parts, salt 1 part, sal-ammoniac 3 parts, dolomite 1 part, and spathic iron ore 1 part, proportions by weight." It may also be used for producing a high-grade steel by adding it to the molten metal. Venetian red or carbonate of iron may be used instead of spathic iron ore, ordinary limestone instead of dolomite, and silica sand in lieu of pumice-stone. The essential ingredients are borax, sal-ammoniac, pumice-stone and dolomite.—H. K. T.

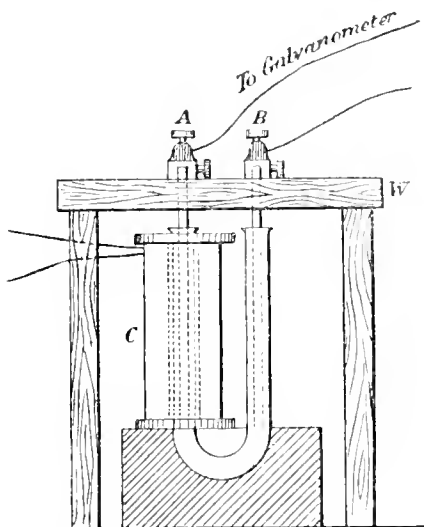
*Improvements in the Reduction of Iron Ores.* H. W. Lash and J. Johnson, Pittsburgh, U.S.A. Eng. Pat. 8992, June 10, 1890. 6d.

The improvement relates to the direct reduction process, by which the loss of iron is reduced to less than 4 per cent. of the amount present. The ore and coke are separately pulverised and passed through a quarter-inch screen. The former is then mixed with 20 to 30 per cent. of its weight of the latter, and the whole is thoroughly ground with a sufficient quantity of water, oil, or other suitable fluid to form a pasty mass. This grinding is the chief point of the process. The finely-ground pasty mixture is spread on the hot hearth of the furnace and covered with a layer of pulverised carbon. As reduction takes place the mass swells, and it is maintained that as a result the fused slag escapes through the pores of the sponge on to the furnace bottom, whence it is tapped off, thus giving a purer metal than is otherwise obtained. The reduced iron is balled up and treated in any suitable manner.—A. W.

## XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

*The Passive State of Iron and Steel.* Part I. T. Andrews. Proc. Roy. Soc. 48, 116—126.

The author has studied the effect of magnetism on passive steel in respect to its behaviour with strong nitric acid, both cold and hot, by measuring the E.M.F. produced by two bars of the same steel, one magnetic and the other not so, immersed in the acid. The apparatus was arranged as in the following sketch:—



THE PASSIVE STATE OF IRON AND STEEL.

The polished bars were  $8\frac{1}{2}$  in. long and 0.261 in diameter, and each was placed in one limb of the U-tube containing  $1\frac{1}{2}$  fluid ounce of nitric acid of sp. gr. 1.42. The bar A was sometimes magnetised beforehand and sometimes by the coil C, in which latter case any results obtained were due to residual permanent magnetism. Results are given of the measured E.M.F. at the time of immersion, and at intervals extending in some instances over seven days. In some cases at the moment of immersion an instantaneous but steady deflection of the galvanometer took place, indicating an electro-positive position for the magnetised bar, but this subsided in a few seconds, and did not interfere with the results due to electro-chemical action. These results show that magnetism of low intensity and even of high intensity, whilst modifying to a limited extent, does not entirely overcome the passivity of steel in cold nitric acid.

The effect, however, is quicker when the acid is warm. A somewhat similar apparatus was placed in a beaker of water which was gradually heated, and the observations were taken at different temperatures up to the transition point from the passive to the active state, which averaged  $195^{\circ}$  F. The acid remained colourless until a temperature of  $100^{\circ}$  to  $120^{\circ}$  F. was reached, when a very pale yellow tint was perceptible in both tubes. Very little solvent action took place, however, until the critical point was reached, indicated by the explosive evolution of red fumes. This happened first in the limb containing the magnetised bar, the first intimation of the passivity of the steel having been overcome being a sudden and extensive deviation of the galvanometer indicating electro-positive for the magnetic bar. The E.M.F. at this point was very high, being from  $\frac{1}{10}$  to  $\frac{1}{5}$  of a volt. After this the steel became partially passive again for a short time. Unless the critical point was reached the steel never fully lost its passivity but was only very slowly and partially acted on by the acid.—A. W.



*The Oxidation of Metallic Sulphides by the Electric Current.* E. F. Smith. Ber. **23**, 2276—2283.

See under XXIII., page 971.

#### PATENTS.

*Improvements in Galvanic Batteries and Electro-Chemical Accumulators.* R. E. B. Crompton and D. G. Fitzgerald, London. Eng. Pat. 5159, November 25, 1881. (Third Edition.) 6d.

In order to obtain a porous electrode presenting great surface to the action of the electrolyte an alloy of lead and some suitable metal such as sodium, potassium or zinc, or a mixture of lead with a material such as chalk, manganese dioxide, &c. is formed into the desired shape, and then subjected to the action of water, acid, or other suitable liquid which is capable of dissolving out all the material with which the lead is mixed, leaving a porous lead plate. Electrolysis may also be used for the removal of the foreign matter.—B. T.

*Improvements in Effecting the Separation of Gold from Antimony.* T. C. Sanderson, Oporto, Portugal. Eng. Pat. 6882, April 24, 1889. 6d.

This invention consists in separating by electrolysis, gold from antimony obtained in smelting auriferous antimony ores. For this purpose the antimony containing the gold is placed in a bath of a suitable salt of antimony and connected with one pole of an electric generator to serve as a positive electrode. To the other pole is connected a plate of suitable conducting material such as carbon or antimony to serve as negative electrode, and a current of electricity is passed through the bath, the antimony plate being dissolved leaving a mud containing most of the impurities originally present in the plate and practically the whole of the gold present. Metallic antimony will at the same time be deposited upon the negative electrode from the bath or electrolyte. The auriferous antimony ore is smelted and pickled in hydrochloric acid or sulphuric acid to get rid of sulphide of iron if present. The electrolysis bath is made up of chloride of antimony formed by dissolving the powdered ore in commercial hydrochloric acid. This is diluted with strong brine in the proportion of 1 measure of chloride of antimony containing 35 per cent. of the salt to 3 measures of saturated brine. The plates of auriferous antimony are placed in this solution and connected with the electric generator. The other pole is also connected and a current of 5 volt passed through the solution. To collect the auriferous mud and keep up free circulation of the bath, the negative electrodes are enclosed in rectangular wooden frames on both sides of which porous material such as calico, flannel, &c. is stretched. The muddy liquid resulting from the solution of the plates attached to the positive electrode is drawn off from the bottom of the bath, passed through a filter, and again introduced into the vessel through the top of the frames containing the negative electrodes. Instead of making a bath by adding chloride of antimony, brine acidified with hydrochloric acid may be used. On the passage of the current, the antimony on the positive electrode will dissolve and form chloride of antimony.

—J. B. C.

*Improvements in Lead Tanks and in the Method of Manufacturing the same.* P. J. Davies, London. Eng. Pat. 12,157A, July 31, 1889. 8d.

This describes an arrangement of cores and moulds for casting lead tanks or cisterns suitable for storage batteries and other purposes; an arrangement of wooden stays and runners may be used for strengthening purposes.—B. T.

*Improvements connected with or Applicable to Baths or Vessels containing Liquids used in Electro-plating or Dyeing, or the Electrolyte of Storage Batteries, and the like.* E. Hopkinson and D. Appleton, Manchester. Eng. Pat. 14,871, September 20, 1889. 6d.

To obtain uniform density of the liquid used in dye-works, electro-depositing baths, electric accumulators, &c., perforated pipes are laid along the bottoms of the vessels, and air or other suitable gas is pumped through these perforations to produce circulation of the liquid.—B. T.

*Improvements in Methods of and Apparatus for Melting, Refining and Casting Metals and other Substances by Electricity.* H. H. Lake, London. From E. A. Colby, Newark, U.S.A. Eng. Pat. 7876, May 20, 1890. 8d.

To obtain a perfectly homogeneous mass of a refractory metal, such as platinum, the metal is heated electrically to the point of liquefaction in a rarefied atmosphere, or in the presence of any desired gas. Alternating currents of electricity are passed round a primary coil of wire insulated with asbestos and arranged in annular form around the substance to be melted, a core of laminated iron being also employed to intensify the currents. Induced currents of great quantity are thus obtained in the substance if it be a conductor of electricity, and, if it be not, in the metallic vessel containing the substance to be treated, the heat thus produced reducing the substance to a state of fusion. Provision is made for pouring the molten material into a mould without opening the furnace.—B. T.

## XII.—FATS, OILS, AND SOAP MANUFACTURE.

*Notes on the Manufacture of Mineral Lubricating Oil.* A. Veith. Chem. Zeit. **14**, 901—902 and 942—943.

See under III., page 926.

*On the Detection of Fish Oil in Linsced Oil.* A. Ruffin. Chem. Zeit. Rep. **14**, 205.

See under XXIII., page 974.

#### PATENTS.

*Improvements in the Composition of Soap.* T. Anyon, Manchester. Eng. Pat. 16,518, October 19, 1889. 4d.

FINELY powdered dry neutral soap is incorporated with a powerful disinfectant such as permanganate of sodium or potassium. This powder is either used as such in sprinklers, or it is subjected to pressure to solidify it into a tablet soap.—K. M.

*An Improvement in Lubricants.* W. Brinek, Linden near Hanover, Germany. Eng. Pat. 17,163, October 30, 1889. 4d.

To impart the requisite degree of viscosity to liquid mineral oils a small quantity of caoutchouc is dissolved therein, and the solution filtered. To solid lubricants caoutchouc may also be added with advantage of economy in the course of manufacture, the lubricant not melting so easily in the bearings. Caoutchouc in an oil prevents to a great extent the liability of "gumming," to which bearings are subject with ordinary lubricants.—K. M.

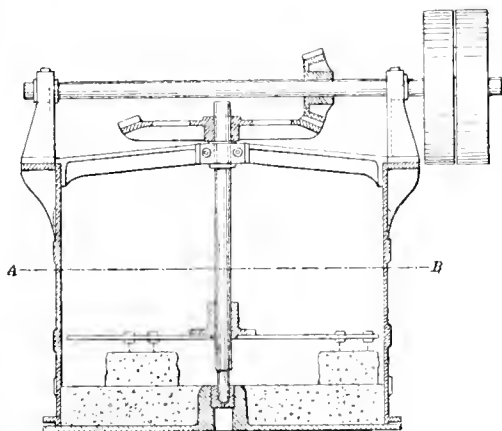
### XIII.—PAINTS, PIGMENTS, VARNISHES, AND RESINS.

*Description of the Cleator Iron Ore Company's Barytes and Umber Mines and Refining Mills.* P. L. Addison. Proc. Inst. Civil Eng. 102.

On a property known as the Caldbeck Fells, in Cumberland, umber and barytes were discovered in workable quantity about five years ago.

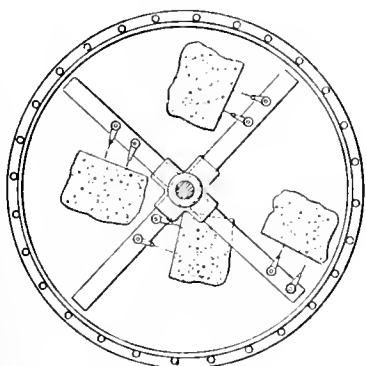
The vein of umber contains crystals of quartz, and lies in a granitic rock largely decomposed. The method of working is as follows:—The umber is brought down by an overhead tramway and passed through a hopper into a wash barrel consisting of a cylinder formed of parallel bars one-eighth of an inch apart, having a perforated pipe conveying water, for its axis. By this means the umber is washed through, the quartz being retained; the former then passes to an edge-runner, the casing of which is of sufficient depth to allow of the submersion of the rollers. The rate of revolution is about 14 to the minute, and the finer floating particles flow into the drag mill shown in the figure. The bed of this mill is a single block of granite and over it the four burrstone blocks shown in the plan are dragged; the

Fig. 1.



Section.

Fig. 2.



Section below A B.

DRAG MILL.

finer "floating" particles of umber pass to a second mill of the same kind, then through a brass wire sieve (to remove particles of peat and heather that have been floating throughout the process) to settling tanks, composed of brickwork lined with cement (7' x 7' x 5'). After settling

for four hours four-fifths of the water are drawn off, and the umber, now of the consistency of slurry, filter-pressed and dried. It has the following composition:—

#### *Analysis of Umber.*

	Parts per Cent.
Ferric oxide .....	47.14
Manganese dioxide .....	11.17
Cupric oxide .....	3.23
Alumina .....	7.66
Lime .....	Trace.
Magnesia .....	Trace.
Silica .....	21.70
Combined water .....	6.18
	<hr/> 100.08

In this condition it may be put on the market, serving for colouring coarse brown paper (that being the chief use to which umber is put), or it may be re-ground in a conical burrstone mill and sold to paint and oil-cloth manufacturers and the makers of the finer kinds of brown paper. The fine state of division to which it is reduced may be judged from the facts that the workman in charge of the mill is compelled to wear a respirator, and the stain is not easily removed from the hands.

The power necessary is obtained from a light overshot water wheel 30 x 2 feet.

The process for preparing barytes needs considerably greater power, a water wheel 36 x 5 being employed. Some difficulty arises from the presence of a ferruginous clay with the barytes, which necessitates a washing with acid to attain a good colour. The rough barytes is washed fairly free from clay, broken into half-inch cubes in a crusher, and ground in a flat burrstone mill, the upper stone of which weighs 35 cwt., and is driven at 120 revolutions per minute, a jet of water being allowed to play between the stones. From this the sludge flows to drag mills similar to those illustrated above in the description of the umber works. Thence it is carried to a receiving tank provided with an agitator to prevent the heavy substance sinking to the bottom and forming a caked unmanageable mass. "Bleaching" is now effected by treating the barytes, holding but little water, with sulphuric acid in the proportion of 70—80 lb. to 25—30 cwt. of the mineral. The operation is conducted in red sandstone tanks provided with agitators made of pitch-pine, experience showing that material to resist the action of the acid better than oak or other ordinary wood. After adding the acid steam is turned on, and the mixture boiled for one hour, after which cold water is run in and the contents of the tank passed into the washing vat, where repeated agitation with water removes the acid until none can be detected by litmus paper. Ultramarine blue in the proportion of 6—8 tablespoonfuls to 30 cwt. barytes is added "to give clearness to the white," excess being avoided as an objectionable "pearly tint" is produced thereby. The washed barytes is filter-pressed (on very strong cloths needed on account of its great weight), dried in a steam room, and packed into clean paraffin casks holding about 10 cwt. apiece. It has the following composition:—

#### *Analysis of Barytes.*

	Parts per Cent.
Barium sulphate .....	96.29
Silica .....	2.60
Lime and alumina .....	0.10
Ferric oxide .....	0.01
	<hr/> 99.00

Large quantities of water being needed, the mountain streams are utilised, and freed from sand and peat by the following device: The supply for grinding, bleaching, and washing is taken from the trough supplying the water wheel, the outlet being 3 in. above the floor of the trough. It flows thence to the bottom of a column of 12 in. cast-iron pipes, the upper parts of which are only 9 in. in diameter. Between the two portions is a diaphragm of brass gauze (1,600 meshes per square inch), and above this the purified

supply is taken. A sludge cock at the bottom allows of an occasional blow-off, cleaning at the same time the wire gauze. The water being always drawn from the top of what is in effect a very deep settling tank, is free from sand and other impurities.—B. B.

*The Analysis of Celluloid.* H. Zauschirm. Chem. Zeit. 14, 905.

See under XXIII., page 979.

## PATENTS.

*Improvements in Elastic Sheets or Tubing.* L. W. Leeds and P. S. Justice, London. Eng. Pat. 13,053, August 19, 1889. 4d

This invention consists in the application of an elastic coating to the tube or sheet while the latter is in an expanded condition. The rubber or other elastic tubes are expanded by air under considerable pressure, and soluble rubber, elastic varnish, or other suitable material is then applied to the internal or external surface, or the coating material may be forced into the tube under sufficient pressure to expand it to the desired degree, the pressure being subsequently removed and the excess of material run from the tube, which may then be internally polished by forcing through it one or more shuttles or marbles, and finally dried by heated air or other means.—E. G. C.

*Improvements in Translucent Plastic Compositions.* F. Eckstein, Vienna, Austria. Eng. Pat. 14,569, September 16, 1889. 4d.

The inventor describes his composition, "hyaline," as a "horny, translucent, plastic composition of great tensile strength and considerable elasticity, which may be used as a cheap and inodorous substitute for celluloid, and can be worked, dyed, pressed, denitrated, and rendered incombustible or fireproof." "Hyaline" is a mixture of about equal parts of gum-cotton and colophony, or shellac, copal, dammar, turpentine, or of any mixture of these resins.

—E. G. C.

*Improvements in Paints or Compositions for Covering Surfaces.* R. Barton, Hull. Eng. Pat. 14,797, September 19, 1889. 4d.

KIESELGUHR (12 parts) is mixed with linseed or colza oil (3 parts) and carbonate or oxide of lead, baryta, or the like (5 parts), any suitable colouring matter, such as lamp black, ochre, ultramarine, &c. being added.—E. G. C.

*An Improved Polish for Cleansing and Reviving Furniture and Analogous Articles.* J. Simpkins, Winchester. Eng. Pat. 15,180, September 26, 1889. 4d.

The composition is as follows:—"Beer, waste beer, cold tea, or vinegar, 48 parts;" methylated spirit or finish, 32 parts; dragon's blood, 1 part; gum benzoin, 3 parts; linseed oil, 16 parts.—E. G. C.

*A New Pigment or Paint Base and Processes of producing the same.* W. L. Wise, London. From J. P. Perkins, New York, U.S.A. Eng. Pat. 15,805, October 8, 1889. 6d.

A VARIETY of bright red pigments consisting of a mixture of ferric oxide and silica are produced from silicious ferruginous slag. The pulverised slag is either treated with sulphuric acid, and the resulting mass gradually heated to a light red heat (never above 1800°) till all sulphuric acid is driven off, or it is heated in presence of air and then treated with sulphuric acid with subsequent heating. The latter method requires more fuel but less sulphuric acid than the former, the product being substantially the same. The resulting

pigment can easily be ground to an impalpable powder, and consists of ferric oxide and silica in the proportion of about 25 parts of silica to 75 parts of the oxide. The shade of colour may be varied by varying the amount of sulphuric acid used, the more acid used, the brighter the red. Owing to the extremely fine condition and opacity of the silica set free by the action of the acid on the silicates in the slag, these pigments are brighter and more pronounced in colour than those pigments produced by mixing ordinary Venetian reds with ground silica.—K. M.

*Improvements in and relating to the Manufacture of Vulcanised Plastic Compounds.* A. J. Boulton, London. From W. Kiel, Butler, U.S.A. Eng. Pat. 9822, June 24, 1890. 6d.

This invention relates to the manufacture of vulcanised rubber, having several advantages over the material as ordinarily prepared. The product may be made either hard and rigid, in which case it takes a higher polish, is not so brittle, and can be more readily turned in a lathe; or it may be made so flexible that a rod or tube may be twisted to any shape.

The ingredients for the manufacture are crude rubber, sulphur, and mineral oil, in the proportions of about equal parts of rubber and sulphur, the oil varying from  $\frac{1}{16}$  to  $\frac{1}{2}$  of the sulphur, according to the degree of hardness required. The compound is vulcanised at a steam pressure of not less than 60 lb., the operation lasting about three hours; or by increasing the pressure to 100 lb. the vulcanisation may be completed in one hour (see this Journal, 1889, 204, and 1890, 398).—F. H. L.

*Improvements in making White Pigments of Lead.* J. Gordon, Bridge of Allan. Eng. Pat. 9897, June 26, 1890. 6d.

GALENA, in the state of fine powder, is injected, by means of a current of compressed air, into the upper part of a eupola furnace fed with coke. The oxidised product, consisting of sulphite, sulphate and oxide of lead in varying proportions, is drawn, by means of an exhausting apparatus, into a wet fume condenser containing water acidulated with hydrochloric acid to remove impurities.

Drawings of a suitable furnace are given.—F. H. L.

*A New or Improved Process for producing Minium.* M. Alsborg, New York, U.S.A. Eng. Pat. 10,130, July 1, 1890. 4d.

A MIXTURE of litharge or white lead and lead nitrate, prepared either by mixing together 100 parts of lead oxide, 18.6 parts of lead nitrate, and 15 parts of water, or by adding 7.1 parts of dilute nitric acid to 100 parts of litharge, is exposed to a temperature of from 700° to 900° F., care being taken not to reach a red heat.—F. H. L.

## XIV.—TANNING, LEATHER, GLUE, AND SIZE.

*Wattles and Wattle Barks, being Hints on the Conservation and Cultivation of Wattles, together with Particulars of their Value.* J. H. Maiden, Department of Public Instruction, Sydney, N.S.W. 1890.

AT present only the following wattles are recommended to be planted:—

The South Australia broad-leaved wattle, *Acacia pycnantha*, the Sydney black wattle, *Acacia decurrens*, and the Tasmanian and Victorian black wattle, *Acacia mollissima*. Throughout Australia the species of wattle richest in tannin

are becoming seriously diminished, and tanners will have to fall back on second-grade barks, unless the cultivation of good barks is actively entered on. For bark which in 1872 cost from 2*l.* 10*s.* to 3*l.* 15*s.* per ton, 8*l.* or 9*l.* was paid in 1887. The best Sydney bark has fetched 10*l.* this season and this appears to be the top average price.

**CULTIVATION OF WATTLES.**—Wattles will grow on the poorest soils where not even grass grows. Sandy soil is best, lying upon a clay subsoil. A moderate amount of moisture, say from 18 to 20 inches per annum, is best; an unlimited supply of moisture is prejudicial.

The outer covering of the seed is of great hardness and the seed requires to be heated, either by moist or dry heat, to ensure early germination.

Wattle trees are sometimes recommended to be pruned, but this is to be deprecated in warm northern exposures as too much sun would be admitted to the stem of the tree.

Under the section *Profits to be Derived from Wattle Cultivation*, detailed estimates are given in which it is shown that the "profit balance" exclusive of improvements or supplementary sowings, on a wattle plantation of 100 acres during eight years, amounted to a total of 2,637*l.* Other estimates are given showing similar results.

**WATTLE BARKS IN GENERAL.**—(a.) *Time of Year for Stripping.*—Wattles should only be stripped for three or four months in the year, as when stripped out of season there is usually a depreciation of the tannin. September, October, November, and December are recommended, but no fixed time applicable to all parts of the country and to varying seasons, can be given. Bark should be purchased in the stick or bundle, and tanners are warned against receiving it in a damp state as it engenders mould "of a most virulent description and is liable to spontaneous combustion."

(b.) *Age and Size of Trees.*—Bark may be stripped at the end of five years, and no stems under five inches in diameter should be cut.

(c.) *Export, Packing, &c.*—Barks are sent into commerce in one or more of four forms:—(1.) In the bundle. (2.) Chopped, i.e., into pieces a few inches in length. (3.) Ground, forming a substance something like "tow." (4.) Powdered. Extract has been made from the wattle "trash" which yields 12 to 16 per cent. of tannage. This extract, however, contains a large amount of gum and mucilage, and some cheap process of getting rid of this mucilage is a desideratum.

**THE VARIOUS KINDS OF WATTLE BARKS.**—This section contains a description (illustrated with seven full page drawings of wattles) of the various wattle barks, with observations and analyses partly given before in a paper by the author which is published in *Proc. R.S., N.S.W.*, 1887, 30; this Journal, 1888, 38 and 392. The following is synopsis of wattle bark analysis performed by the author. The percentage of tannin in each case was determined on the bark dried at 100° C. by the improved Löwenthal method. The local name, species name, percentage of tannin, and where grown are given in the order named.

**Valuable Wattle Barks.**—Black wattle, *Binervata*, 19·3 to 30·4, N.S.W.; broom wattle, *Calamifolia*, 36·06, S.A.; black wattle, *Decurrens*, 24·13 to 36·3, N.S.W.; Mountain hickory, *Elata*, 20·1 to 31·1, N.S.W.; Tasmanian, black or green wattle, *Mollissima*, 24·63 to 35·3, N.S.W.; Hickory, *Penninervis*, 14·49 to 31·0, Q.; broad-leaved wattle, *Pycnantha*, 16·24 to 46·47, S.A.; weeping wattle, *Vestita*, 27·96 to 33·2, N.S.W.

**Wattle Barks of Secondary Value.**—, *Amara* 23·5, N.S.W.; silver wattle, *Dealbata*, 17·1 to 25·9, N.S.W.; golden wattle, *Longifolia*, 5·0 to 18·93, N.S.W.; Sally, *Longifolia* var. *Floribunda*, 2·54 to 6·09, N.S.W.; Blackwood, *Melanoxylon*, 11·12, N.S.W.; A black wattle, *Nerifolia*; —, Q.; a silver wattle, *Podalyriaefolia*, —, Q.; grey wattle, *Promincens*, 14·42 to 18·03, N.S.W.; — *Pruinos*, 24·25, N.S.W.; Cooba, *Salicina*, 13·21 to 15·1, N.S.W.

**WORTHLESS WATTLE BARKS.**—Mulga, *Aucura*, 2·32 to 8·62, N.S.W.; Wait-a-while, *colletoides*, 4·4, N.S.W.;

a black wattle, *Cunninghamii*, —, Q.; —, *Flavescens*, —, Q.; Myall or boree, *Glaucescens*, 8·10, N.S.W.; Curly yarran, *Homalophylla*, 9·06, N.S.W.; —, *Impleta*, 7·82, N.S.W.; —, *Leptocarpa*, —, Q.; Miljee, *Oswaldi*, 9·72, N.S.W.; Bastard gidgee or myall, *Pendula*, 3·25, N.S.W.; Yarran, *Pendula*, var. *glabrata*, 7·15, N.S.W.; —, *Polystachya*, —, Q.; —, *Pravissima*, 10·66, N.S.W.; Nealie or needle-bush, *Rigens*, 6·26, N.S.W.; —, *Sentis*, 6·32 to 10·26, N.S.W.; —, *Siculiformis*, 7·87, N.S.W.; —, *Stenophylla*, 9·49, N.S.W.; River wattle, *Subporosa*, 6·6, N.S.W.; Dead finish, *Tetragonophylla*, 5·59; —, *Verniciflua*, 3·16, N.S.W.

A small chapter is also devoted to a discussion of the similarities and dissimilarities of *Acacia decurrens*, *Mollissima* and *Dealbata*. A very complete index is given at the end of the pamphlet.—B. H.

#### *On the Action of Phenylhydrazine on Tannin Extracts.* C. Böttinger. *Annalen*, 259, 125—132.

PURE tannins yield with phenylhydrazine different kinds of derivatives which do not crystallise and do not allow of satisfactory separation. The amorphous form of the compounds is evidently a consequence of their high molecular weight. The tannin materials experimented with were extracts of sumac, divi-divi, oakwood, oak-bark, and pine-bark. The diluted extract was heated with phenylhydrazine for six to eight hours on a water-bath, whereby a precipitate was obtained which was filtered off, washed with water and air-dried. During the operation a considerable evolution of carbonic acid and apparently nitrogen took place, at the same time a part of the phenylhydrazine was decomposed into ammonia and azobenzene. 100 cc. phenylhydrazine yielded 248 grms. of air-dried precipitate from oak-bark extract, and 175 grms. from pine-bark extract. The precipitate contained phenylhydrazine derivatives of tannic and gallic acids and osazones of dextrose and levulose, together with amorphous substances similar to caramel.—G. H. B.

#### *On Gallic, Tannic, and Gallotannic Acids.* C. Böttinger. *Annalen*, 259, 132—136.

THE author has endeavoured to effect the union of hydrocyanic acid with the acids of the tannin group, and for this purpose the acetyl and benzoyl derivatives of the tannins were boiled for several hours with an aqueous solution of potassium cyanide in excess. No cyanogen compound was obtained, but ammonium formate was produced and the tannin was changed to gallic acid. Well dried tannin heated with anhydrous hydrocyanic acid in a sealed tube in boiling water yielded only an amide of gallic acid. Victor Meyer's employment of hydroxylamine chloride as a reagent for ketones and aldehydes is of such utility that the same investigator entertained the idea that the reaction might play a part in the actual growth of plants, but he was unable to trace it in nature. The author thinks that he has found a reason for this in the fact that none of the tannin acids could be made to react with hydroxylamine.—G. H. B.

## XV.—AGRICULTURE, MANURES, Etc.

*Wattles and Wattle Barks, being Hints on the Conservation and Cultivation of Wattles, together with Particulars of their Value.* J. H. Maiden, Department of Public Instruction, Sydney, N.S.W. 1890.

See under XIV., page 954.

*Loss of Nitrogen in the Decay of Nitrogenous Organic Substances.* H. v. Krause. *Jour. f. Landw.* 38, 1.

THE experiments were made in glass flasks on one litre of cow's urine, and may be divided into two groups. (1.) Experiments on the influence of various substances, with free access of air and with limited access of air, upon

the decay of nitrogenous organic substances, as well as their influence on loss of nitrogen generally. (2.) Experiments on the loss of free nitrogen in the presence of different preservatives.

The substances added were superphosphate, superphosphate and gypsum, gypsum, kainit, potassium chloride, salt, Thomas-slag (85 per cent. fine), peat and distilled water.

Superphosphate is the best preservative of nitrogen, surpassing in this respect the much recommended superphosphate and gypsum, smaller quantities of the pure superphosphate being required; the richest superphosphates are the best.

Gypsum has a tendency to promote rather than hinder fermentation; it can retain an amount of ammonia equivalent to its sulphuric acid. Rapid fermentation, however, means a greater loss of free nitrogen, so that gypsum is not a good preservative.

Kainit was found to be good for a short time, but after fermentation had set in, the loss, especially of free nitrogen, was very great; the practical conclusion therefore is that kainit should be used only when the dung is to be a short time in the heap.

Peat promotes fermentation, and loss of nitrogen, especially free nitrogen, is considerable. At first the peat prevents loss because its humic acid combines with the ammonia; also because it retains water. Practically peat may be used when the dung heap requires solidifying.

Thomas-slag is useless as a preservative; it promotes fermentation and cannot absorb ammonia.—A. G. B.

#### *The Distribution of Boracic Acid in Plants.* J. S. Callison. Jour. Anal. Chem. 1890, 191.

CERTAIN plants seem to possess a peculiar assimilating power for boracic acid, others are found perfectly free from it, although growing with the former plants in the same soil. Of the 92 plants examined by the author, eight contained no boracic acid. It must be assumed that the plants obtain the acid from the soil although in no case was it found either in the soil or in the spring water. Analyses of certain manures such as bone manure and Pern guano, as well as of potassium chloride and kainit from Stassfurt, indicated the presence of considerable quantities of boracic acid. (See also this Journal, 1889, 569.)—A. R.

#### PATENT.

##### *Improvements in the Manufacture of Manures and Fertilisers.* M. C. Gunster, Birmingham. Eng. Pat. 575, January 13, 1890. 4d.

THE object of this invention is to produce, by chemical means and without the employment of machinery, a rich fertiliser possessing a high percentage of ammonia and phosphates from blood or blood and fish combined, or other organic matter. The blood is collected in suitable vessels, then passed through a screen into a tank. 35—40 lb. of oil of vitriol to the ton of blood are then added, and the mixture is stirred for 10—15 minutes. The mixture is allowed to stand 10—12 hours, and then transferred to a press and the liquid drained off. The solid substance is broken up and dried in the sun or in a kiln at a temperature not exceeding 100° F. This manure contains about 14 per cent. nitrogen. The press liquor is said to form a good manure if mixed with some dry substance such as bone or wool dust.

The fish are treated in a similar manner to the above. They are first finely chopped, however, before being put into the tank. 35—40 lb. of acid to the ton are used, the mixture stirred for 15 minutes, and allowed to stand 6—8 hours. The liquid is then drained off and treated similarly to the blood liquid. The solid substance is sun- or kiln-dried at a temperature not exceeding 80° F. This manure contains a high though variable percentage of phosphate and ammonia.—F. W. T. K.

## XVI.—SUGAR, STARCH, GUM, Etc.

### *On the Estimation of Raffinose and Invert Sugar in the Products of the German Beet Sugar Manufacturers.* Chem. Zeit. 14, 854—855.

See under XXIII., page 978.

### *"Fucusaldehyde," a Mixture of Furfuraldehyde and Methylfurfuraldehyde.* K. Bieler and B. Tollens. Annalen, 258, 110—128.

THE compound termed "Fucusol," obtained by Stenhouse by distilling seaweed with sulphuric acid, is generally regarded as an isomeride of furfuraldehyde, but the very slight differences between these substances made it seem probable to the authors that it is only a mixture of that substance with some difficultly separable homologue. A revision of Stenhouse's work was undertaken to decide this point, and to determine the identity of the oil resembling furfuraldehyde, obtained by one of the authors and Stone and Wheeler (Ann. 254, 304) from arabinose. During the progress of the experiments Hill (Ber. 22, 607; this Journal, 1889, 973) isolated from the furfuraldehyde obtained by the distillation of wood, a higher homologue, which he regarded as "Methylfurfuraldehyde." Maquenne (Compt. Rend. 109, 571 and 603; this Journal, 1889, 974) examined fucusaldehyde, and obtained results agreeing with those of the authors, and Oliveri and Peratoner (Gazz. 19, 633) separated fucusaldehyde into furfuraldehyde and a compound of a higher boiling point. The authors draw the following conclusion from their experiments: (1.) Fucusaldehyde from seaweed is merely a mixture of furfuraldehyde and methylfurfuraldehyde. (2.) The furfuraldehyde from seaweed and its derivatives are identical with the compounds obtained from bran. (3.) Methylfurfuraldehyde boils at 182°—184°, gives an orange-red colouration with aniline acetate, yields a hydramide melting at 87°, and a methylpyromucic acid melting at 108°; it also exhibits a fine green colouration when treated with sulphuric acid and isatin. (4.) A sugar may be separated from seaweed which yields furfuraldehyde or methylfurfuraldehyde, and a hydrazone which resembles rhamnose hydrazone, but has a higher melting point. It has not yet been crystallised.—S. B. A. A.

### *Carbohydrates of the Sweet Potato (Batatas Edulis).* W. E. Stone. Ber. 23, 1406—1408.

SWEET potatoes of the varieties "American Red" and "Southern Queen" are found to contain  $\frac{1}{4}$  to 2 per cent. of cane sugar, a large proportion of starch, but no reducing sugar. Baking develops dextroses and glucoses from these constituents.—D. A. L.

### *A New Crystalline Carbohydrate.* A. v. Planta and E. Schulze. Ber. 23, 1692—1699.

THE extract obtained from the tubers of *Stachys tuberosa* by pressing and washing with water is precipitated firstly with lead acetate, then with mercuric nitrate, treated with hydrogen sulphide, filtered, neutralised with ammonia, evaporated to a syrup and poured into alcohol. The syrupy precipitate is mixed with phosphotungstic acid, filtered, treated first with barium hydroxide to remove the excess of phosphotungstic acid, then with carbonic anhydride to remove the barium, and the final filtrate is evaporated to a small volume and poured into absolute alcohol. The precipitate obtained in this manner is dissolved in water and re-precipitated with alcohol two or three times, and is finally crystallised from 91 per cent. (by volume) alcohol. The crystals assume two forms, of one of which, tablets, the measurements are given:—

$$\alpha = 88^\circ 41\frac{1}{2}', \beta = 92^\circ 32\frac{1}{2}', \gamma = 153^\circ 43\frac{1}{2}'. \quad a:b:c = 0.7848:1:2; \quad c = 0 \text{ P } (001), \quad b = \alpha \text{ P } \underline{x} \quad (010), \quad p = \alpha \text{ P}_1(110), \quad p' = \alpha'_1 \text{ P } (110), \quad p'_2 = \alpha'_1 \text{ P } \frac{8}{9} \quad (890).$$

They are colourless, hard and glistening, lose 9.67 per cent. of water of crystallisation at  $103^{\circ}$ — $104^{\circ}$ , but above that temperature undergo decomposition. Various analyses point to the composition  $C_{36}H_{62}O_{31} + 7 H_2O$ ; the water of crystallisation is very slowly removed by exposure over strong sulphuric acid, being incomplete after many weeks. The aqueous solution is dextro rotatory, a 9.0 per cent. solution in a Soleil-Ventzke apparatus showing  $[\alpha]_D = +148.1^{\circ}$ , and is without action on Fehling's solution. With nitric acid it yields mucic acid, and gives a red colouration and precipitate with resorcinol and hydrochloric acid, but not with phloroglucinol. Lead acetate produces a precipitate in the presence of alcohol or ammonia, but not in the ordinary aqueous solutions. Barium and strontium hydroxides do not precipitate this carbohydrate. It is readily inverted, half the product being galactose; the specific rotation after inversion =  $[\alpha]_D = +75^{\circ}$ . This carbohydrate has a very slightly sweet taste, of known compounds of this description it most resembles lactosin, but is not considered identical with it, and has been named by the discoverers stachyose.—D. A. L.

*Contributions to a Knowledge of the Carbohydrates.* A. Wold. Ber. 23, 2084—2110.

AFTER giving a short account of the investigations previously published concerning the action of dilute acids on different carbohydrates, and especially on cane sugar, the author proceeds to give an account of his own experiments.

In a research which he has made working with Kolbrepp, on the inversion of cane sugar by hydrofluoric acid, it was found that with concentrated solutions the less the quantity of acid used, the greater was the maximum observed levo-rotation. Further experiments have shown that this held good with other mineral acids. By heating on the water-bath 80 parts of pure cane sugar with 20 parts of water and 0.004 parts hydrochloric acid for one hour, a pure colourless invert sugar was obtained, which on standing crystallised.

**I. Action of Hydrochloric Acid on Concentrated Solution of Cane Sugar.**—The following experiments were made, the solutions being digested for 30 minutes in a closed flask in boiling water:—

40 per cent. solution with 0.06 per cent. HCl (on the sugar) gave  $-15.4^{\circ}$  and — mgrms. Cu.

60 per cent. solution with 0.025 per cent. HCl (on the sugar) gave  $-14.1^{\circ}$  and 306 mgrms. Cu.

76.6 per cent. solution with 0.01 per cent. HCl (on the sugar) gave  $-13.15^{\circ}$  and 302 mgrms. Cu.

80 per cent. solution with 0.01 per cent. HCl (on the sugar) gave  $-12.3^{\circ}$  and 296 mgrms. Cu.

88 per cent. solution with 0.01 per cent. HCl (on the sugar) gave  $-16^{\circ}$  and 260 mgrms. Cu.

92.6 per cent. solution with 0.01 per cent. HCl (on the sugar) gave  $+0.5^{\circ}$  and — mgrms. Cu.

Calculated for complete conversion  $-16.1^{\circ}$  and 313 mgrms. of Cu.

Further heating of one of these solutions containing 13.024 grms. sugar and 3.3 cc. dilute hydrochloric acid (0.01 per cent. HCl on the sugar) diminished the observed angle and cupric reducing power. In the most concentrated solution the boiling point of water was not hot enough to properly fuse the mixture, and so inversions with this concentration were made at  $105^{\circ}$ — $110^{\circ}$ .

0.01 per cent. HCl (on the sugar) gave  $-12.3^{\circ}$  and 296 mgrms. Cu. Solution colourless.

0.02 per cent. HCl (on the sugar) gave  $-10.0^{\circ}$  and 286 mgrms. Cu. Solution light yellow.

0.03 per cent. HCl (on the sugar) gave  $-6.5^{\circ}$ . Solution yellow.

0.05 per cent. HCl (on the sugar) gave  $-4.6^{\circ}$ . Solution deep yellow.

These experiments may be explained by supposing that either the whole of the cane sugar is not converted or that in these concentrated solutions the invert sugar is not in the same state as it is in weak solutions. Experiments with much stronger acid show that it is not due to the destruction of the levulose. To enlighten this point the second series of experiments were made.

**II. Action of Hydrochloric Acid on Concentrated Invert Sugar Solution.**—The invert sugar was prepared by mixing solutions of pure levulose and dextrose in equal proportions. It had all the properties of invert sugar. The following comparative experiments were made:—

I. 3.425 grms. dextrose and levulose were heated in a 50 cc. flask with 1 cc. dilute hydrochloric acid (0.04 per cent. HCl) and 0.35 cc. dilute hydrochloric acid (0.08 per cent. HCl) for 17 minutes in boiling water. The concentration of the solution corresponded to a 80 per cent. solution of cane sugar completely inverted and containing 0.01 per cent. HCl on the sugar before inversion. The polarisation was  $-12.7^{\circ}$  instead of  $15.8^{\circ}$ , the angle for pure invert sugar.

II. 6.512 grms. of cane sugar with 1.7 cc. dilute hydrochloric acid (0.04 per cent. HCl) was treated in exactly the same way. The polarisation was  $-12.3^{\circ}$ .

III. Experiment I., with 30 minutes heating, gave  $-12.05^{\circ}$  and 295 mgrms. of Cu.

IV. Experiment II., treated in the same way, gave  $-12.25^{\circ}$  and 296 mgrms. of Cu.

V. Experiment I., with 60 minutes heating, gave  $-10.6^{\circ}$  and 287 mgrms. of Cu.

VI. Experiment II., in the same way, gave  $-10.5^{\circ}$  and 288 mgrms. of Cu.

If the acid be omitted in the above experiments, the heating will not alter the properties of the invert sugar. It is thus evident that in concentrated solutions, under the above conditions, a condensation of invert sugar is effected.

**III. Action of Hydrochloric Acid on Concentrated Levulose Solution.**—6.85 grms. of levulose treated in the same way gave  $-63.5^{\circ}$  and 264 mgrms. of Cu. The numbers for pure levulose are  $-73.1^{\circ}$  and 300 mgrms. of Cu.

Other experiments gave similar results. It is, therefore, evident that the condensation observed is not a reproduction of cane sugar, but merely a condensation of the levulose.

**IV. Levulosin.**—This is the product obtained by the above condensation. It was prepared by heating levulose in concentrated solution with 0.01 per cent. of HCl in boiling water for one hour.

The polarisation fell from  $-36.15^{\circ}$  to  $-18.8^{\circ}$ , and the reduction from 153 mgrms. to 81 mgrms. of Cu. The product was dissolved in hot absolute alcohol, and on cooling a dextrinous precipitate separated. It was purified by washing and reprecipitation from its solution in hot alcohol, and dried over sulphuric acid. It has an optical activity about one-half, and a cupric reducing power about one-third, of that of levulose. On digestion with dilute acids these numbers increase, and approach those of levulose, so that in all probability levulose is produced.

**V. Action of Hydrochloric Acid on Dextrose.**—Concentrated dextrose solutions, 80 per cent., were acted on at  $105^{\circ}$  with 0.05—1.0 per cent. HCl for half an hour, one hour, and two hours. The optical activity was found to increase and the reducing power to diminish, these changes being proportional to the strength of acid and duration of heating.

**VI. Reversion and Inversion.**—The author considers that Tollen's oxy-formulae for dextrose and levulose best explain their condensation as above described under the influence of dilute acids.

**VII. On the Saccharification of Starch.**—It has been noted by various observers that in the saccharification of starch by dilute acid, the theoretical numbers for dextrose are never obtained. The author has acted on dextrose under the same conditions as the conversion of starch usually takes place, and finds that the optical activity increases and the cupric reducing power diminishes with the duration of the reaction. He gives a formula for this.

**VIII. The Preparation of Dextrin by the Action of Small Quantities of Nitric Acid and Water on Starch.**—The author considers that part of the dextrin produced is due to reversion of the dextrose. In an experiment he found that a concentrated solution of dextrose heated with a small

quantity of nitric acid for half an hour, showed an increase in the optical activity and diminution in the cupric reducing power.

**IX. Saccharification of Inulin.**—Owing to the rapidity in which levulose is acted upon by dilute acids, it is important, when saccharifying inulin, to work under the most favourable conditions. The author finds that with 80 per cent. solutions and 0.11 per cent. of HCl on the inulin, the maximum of rotation, equivalent to 86 per cent. of the theoretical yield of sugar, is reached in 25–30 minutes at 100°. Honig and Schubert, with a 17 per cent. solution, obtained 91 per cent., and with a 4.62 per cent. solution 93.8 per cent. of the theoretical yield.

**X. Preparation of Levulose.**—In the preparation of levulose, if much acid is used, or if the syrup has to be exposed to high temperature (evaporation) for any length of time, much colour is developed, and it is very difficult to get the levulose to crystallise. The following method is stated to yield a pure product: 50 cc. of water, 200 grms. of inulin, and 0.02 gm. HCl (more hydrochloric acid if the inulin contains ash) are placed in a half-litre flask, and digested for half an hour in the water-bath; sufficient chalk is then added to neutralise the acid. The concentrated solution is dissolved in 1 litre absolute alcohol, decolourised by a small quantity of blood charcoal, and, after standing 12 hours, poured off the residue. If a few crystals of pure levulose are introduced into the solution, a crop of pure crystalline levulose will separate. It is, however, better to evaporate the alcoholic solution in vacuo to a thick syrup, which, when placed over sulphuric acid, soon solidifies to a crystalline mass of levulose. This is dissolved in 3–4 times its weight of absolute alcohol, and if a crystal of levulose be added to the clear solution, a crop of pure levulose soon separates.—A. L. S.

#### *Synthetical Researches in the Sugar Group.* E. Fischer. Ber. 23, 2114.

A LECTURE delivered before the German Chemical Society, giving a complete account of our present knowledge concerning the constitution of the sugars and their synthesis. All the work has been published and is contained in papers by Fischer, Kiliani, Scheibler, Maquenne, Löwe, Tollens, and others, the greater portion of which has been abstracted in the last few volumes of this Journal and that of the Chemical Society.—A. L. S.

#### *On Sugars richer in Carbon from d-Mannose.* Fischer and Passmore. Ber. 23, 2226–2239.

THE authors have proceeded, according to the method they have already indicated (this Journal, 1890, 638), to build up from mannose sugars containing 7, 8 and 9 atoms of carbon and which they have called mannoheptose, mannoctose, and mannnonose, and the corresponding acids heptonic, octonic, and nononic acids, and the corresponding alcohols heptitol, nonitol, from their synthesis it follows that all these bodies contain a normal carbon chain and the sugars are aldehydes.

**d-Mannoheptonic acid** (mannose-carboxylic acid), described in their first communication (this Journal, 1890, 638), they have obtained in quantity from vegetable ivory shavings, by hydrolysing with sulphuric acid, acting on the solution with hydrocyanic acid, and decomposing the amide with baryta, when the barium salt of *d*-mannoheptonic acid is obtained, which is purified by crystallisation; the barium removed by sulphuric acid and the pure acid or its lactone crystallised. The acid melts at 175° C., and by heating is converted into its lactone which melts at 148°–150° C.; its aqueous solution is levo-rotatory  $[\alpha]_D = -74.2$  at 20°.

**d-Mannoheptose** is prepared by reducing the *d*-mannoheptonic lactone with sodium amalgam, the solution being kept in a freezing mixture, and just acid with sulphuric acid. When the reduction is complete the solution is made alkaline with soda, filtered, neutralised with sulphuric acid,

evaporated to a syrup on the water-bath, and added to 10 times its bulk of boiling absolute alcohol. The alcoholic mother-liquor is evaporated to a syrup, and in one or two days the sugar crystallises out; this cannot be easily purified from heptitol, the corresponding alcohol, except by preparing the hydrazone and reconvertng this into the sugar. It crystallises in fine needles having a composition corresponding to the formula  $C_7H_{14}O_7$ , melting point 134°–135°; it has a sweet taste, is easily soluble in water, difficultly in absolute alcohol. Its aqueous solutions are dextro-rotatory and show bi-rotation, the constant value is  $[\alpha]_D = 68.64$ . It is not fermentable with yeast. It yields a hydrazone,  $C_7H_{14}O_6 \cdot N_2H \cdot C_6H_5$ , which is very difficultly soluble in water, melting point 197°–200°, and an osazone,  $C_7H_{12}O_6 \cdot (N_2HC_6H_5)_2$ , melting point 200°.

**Mannoheptitol** is produced by reducing mannoheptose by sodium amalgam in acid solution, the product is purified by crystallisation from water and alcohol, it melts at 188° C. It forms a pentacetyl derivative, melting point 119°. A careful comparison of it with the naturally produced body *perseitol* (Maquenne, Ann. Chim. Phys. [6], 19, 1) showed that they were identical. The authors have converted the natural perseitol by oxidation into the above-described mannoheptose.

**d-Mannoctonic acid** is prepared by the action of hydrocyanic acid on mannoheptose just as mannoheptonic acid is obtained from mannose. Its lactone tastes sweet, melting point 167°–170° C. with decomposition, its solution is levo-rotatory  $[\alpha]_D = -43.58$  at 20° C.; it forms a hydrazone, melting point 243°,  $C_8H_{16}O_8 \cdot N_2H \cdot C_6H_5$ .

**d-Mannoctose**,  $C_8H_{16}O_8$ , is obtained by reduction of the above acid just as *d*-mannoheptose is obtained from its corresponding acid. The sugar is very easily soluble in water and difficultly in absolute alcohol. It tastes sweet and does not ferment with beer yeast. Its aqueous solutions are levo-rotatory  $[\alpha]_D = -3.3$  at 20° C. It forms a difficultly soluble hydrazone,  $C_8H_{16}O_7 \cdot N_2H \cdot C_6H_5$ , melting point 212° with decomposition, and an osazone,  $C_8H_{14}O_6 \cdot (N_2HC_6H_5)_2$ , melting point 223° with decomposition.

**d-Mannoctitol** is obtained by the reduction of the above sugar; it is only slightly soluble in water. It has a composition expressed by the formula  $C_8H_{18}O_8$ , melting point 258° C.

**d-Mannnononic acid** is obtained from *d*-mannoctose in the same way as the previously described acids were obtained from the corresponding sugars. Its lactone,  $C_9H_{18}O_9$ , melting point 175°–177° C. is easily soluble in water and levo-rotatory  $[\alpha]_D = -41.0$ . It forms a hydrazone difficultly soluble in water, melting point 254° C. with decomposition.

**d-Mannnonose**,  $C_9H_{18}O_9$ , is obtained by reducing the last described lactone. It melts at about 130° and is dextro-rotatory, having  $[\alpha]_D$  about 50°. It forms a hydrazone,  $C_9H_{18}O_8 \cdot N_2H \cdot C_6H_5$ , difficultly soluble in cold water, melting point 223° with decomposition, and an osazone, melting point 217°. It ferments with beer yeast as easily as dextrose, which it in many ways resembles.—A. L. S.

## XVII.—BREWING, WINES, SPIRITS, Etc.

*On the Purification of Raw Spirit and Brandy by the Process of Traube and Bodländer; a Report on Experiments performed to test the Value of the Process.* Zeits. f. Spiritusindustrie, 13, 75, 81, 91, 97.

For an account of the process, see this Journal, 1888, 762; 1889, 559 and 995.

The experiments consisted in the determination of the alcohol, fusel oil, and potassium carbonate in the various layers formed in the apparatus, and also in the determination of the alcohol and fusel oil in the raw spirit and distillates. The alcohol was determined by the specific gravity,



the fusel oil by the chloroform extraction method, and the potassium carbonate by titration. The distillate was usually divided into 11 fractions of about 10 litres each. One litre of each was put on one side for analysis, and the remainder of the first nine fractions mixed to form an average sample such as would be obtained commercially. As the distillation was always carried on until the whole of the alcohol had passed over, the last two fractions, being very poor in alcohol, were not added to the average sample.

A distillation carried on in this way gave the following result:—

	Per Cent. Vol. of Alcohol.	Per Cent. Vol. of Fusel Oil.
Raw spirit.....	80.42	0.303
Average sample.....	75.95	0.171
1st 10-litre portion.....	..	0.322
4th 10-litre portion.....	..	0.240
7th 10-litre portion .....	..	0.085
8th, 9th, 10th, and 11th 10-litre portion	..	0

37 per cent. of the fusel oil has thus been removed.

As in the Traube process, pure spirit and potassium carbonate are only used to form the layers in the last stages, but in the first stages raw spirit and spirit from the next vessel in the series is used, so a certain quantity of fusel oil must be introduced at each operation. The difference between the amount contained in the alcohol added and that skimmed off previously shows the net gain or loss of fusel oil. One experiment gave the following:—

Formation of Layer.	Alcohol.	Fusel Oil.
	Litres.	cc.
1st	+ 1.130	+ 7.6
2nd	+ 0.822	+ 8.2
3rd	+ 1.338	+ 7.9
4th	- 0.482	- 10.8
5th	+ 0.029	- 11.4
6th	+ 0.030	- 10.3
7th	- 0.019	- 12.0
8th	- 1.355	- 43.5
9th	- 0.782	- 24.0
Total .....	+ 0.692	- 88.3

That is, although in the first three operations there was a gain of fusel oil, on the whole the total loss was 88.3 cc.; in the raw spirit there was 0.303 per cent. vol. of fusel oil, i.e., in the quantity used 257.5 cc. fusel oil, so that 34.3 per cent. vol. fusel oil had been removed. Other experiments gave similar results.

Experiments were then made with the improved process, whereby a sharper separation of the layers was obtained, and the distillation was made with and without a column. First runnings and after runnings were also separated, and the average sample taken only of the middle fractions.

*Experiment without a Column.*—Thirteen layer formations. A first running and 10 fractions obtained by distillation. The average sample was taken of the first six and half of the seventh fractions; the other half of the seventh and eighth was used in forming a layer in a succeeding operation, the ninth and the first runnings was put on one side with other impure portions, and the 10th was thrown away.

The raw spirit contained....	0.114 per cent. vol. of fusel oil.
The first runnings contained	0.157 " "
The average sample (as above) contained .....	0.033 " "

and contained 20 per cent. of the fusel oil and 76 per cent. of the alcohol present in the raw spirit.

If the whole of the distillation fractions are taken for the average sample, the amount of fusel oil contained by it is 55 per cent., and the amount of alcohol nearly 100 per cent. of that contained in the raw spirit.

*Experiment with a Column.*—Thirteen layer formations. The first runnings were 3 litres (the amount of raw spirit was 95 litres). The next seven 10-litre fractions formed the average sample, and the rest the last runnings.

The raw spirit contained.....	0.127 per cent. vol. of fusel oil.
The first runnings contained..	0.037 " "
The average sample contained	0 " "
The last runnings contained..	1.314 " "

The composition of the whole of the distillate showed that there had been a loss of only 7 per cent. of fusel oil and 5 per cent. of alcohol, but that by the use of the column the whole of the fusel oil was retained in the first and last portions of the distillate, and there was a loss of 20 per cent. of alcohol. The result of all the experiments, then, appear to be that the process of Traube does not fulfil its purpose.

—A. L. S.

*A New Vegetable Ferment.* W. Sigmund. *Monatsh.* 11, 272—276.

A PAPER has recently been read before the Akademie der Wissenschaften at Vienna in which the author states that he has isolated an albuminous substance from the aqueous extract of rape seed, which has a specific action on fats, especially olive oil, decomposing them and liberating fatty acids. When its action was compared with that of ordinary egg albumen, it was found that 0.52 grm. liberated 9.6 mgrms. of oleic acid as against 1.1 mgrms. given by the latter. From this it appears to be a true ferment capable of decomposing fats.—B. B.

*The Examination and Valuation of Spirits.* W. Fresenius. *Zeits. Anal. Chem.* 29, 283—317.

See under XXIII., page 975.

*On some Essences used for Strengthening Spirituous Liquors and Ripening Artificial Brandies.* E. Poleske. *Proceedings of the Imperial Board of Health (Russia)*, 1890, 294.

*Examination of the Essences.*—Taste and smell, the latter especially when the essence is evaporated over the water-bath, will detect many ethers, also fusel oil, acetic acid, vanillin, &c. If the essence contains much alcohol dilution with an equal bulk of water will make it turbid, from separation of oils or resins if they be present. Dilution of the essence with water till it contains 10 per cent. alcohol and subsequent shaking with ether or chloroform will extract many substances, and these may be detected by evaporating the ether by a stream of cold air.

100—300 cc. of the essence (diluted, if necessary, to contain about 30 per cent. of alcohol) is distilled over wire gauze till about 80 per cent. of distillate (I.) is obtained, and a residue (II.) left. This distillate will contain the alcohol, volatile ethers, ethereal oils, fusel oil, part of the ananthic ether and volatile acids; when it reacts acid it is neutralised with decinormal potash and phenolphthalein, redistilled, and the residue mixed with residue II.

The neutral distillate is heated in a reflux apparatus with 2—4 grms. of potash for 2½ hours and distilled (distillate a, residue b). This distillate is made up to the original volume with water and serves for determining the alcohol and the fusel oil by Rose's chloroform method. The chloroform extract will contain any unsaponifiable ethereal oils which may be present, as well as the fusel oil; it is washed with water and quickly evaporated in a stream of cold air; oily drops will be left on the surface of the water, and these, if fusel oil be present, will smell strongly thereof and will give Uffelmann's methyl-violet reaction. A very

small quantity of fusel oil will, however, evaporate with the chloroform and the smell of the ethereal oil will become evident.

Residue *b* will contain the potassium salts derived from the ethereal salts; the corresponding acids may be liberated by distilling with phosphoric acid and steam; if ananthic ether was present some capric and caprylic acids will be obtained here, but most of them will be in residue II. To determine the ananthic ether the essence must be directly saponified, distilled, and the residue distilled with phosphoric acid. The solid (capric and caprylic) acids are filtered from the distillate, dissolved in alcohol, and titrated; again liberated, dissolved in ether, evaporated, and weighed. The lower acids in the filtrate are titrated with baryta, evaporated to dryness, and weighed; if the barium salts are not less than 0.2 grm. they may be separated by absolute alcohol after Luck (*Zeits. Anal. Chem.* 10, 185), and the formic acid determined by mercuric chloride or Menair's bichromate method (*Zeits. Anal. Chem.* 12, 398).

The cinnamic and benzoic acids are extracted together with the capric and caprylic acids from the distillate containing the latter, by ether. All are converted into the potassium salts and the fatty acids thrown out by calcium chloride; the filtrate is evaporated when the sparingly soluble calcium cinnamate separates.

The acids found in residue *b* will serve for the estimation of the rest of the ethereal salts.

Residue II. will smell of vanillin if this be present; shaking with ether and treatment with sodium bisulphite, after Tiemann's and Haarmann's method, will extract the vanillin. The residue from this ether extract will contain resins, such as capsicum, balsam of Peru, and spices. Sugar and caramel can be detected by tasting residue II. A tenacious froth produced by shaking, and an astringent irritating after-taste indicate saponin.

The following table shows the results given in the paper:—

	Rhenish Cognac Essence.	Fine Champagne Cognac Essence with Bouquet.	"Cognac Grund- stoff."	"Brannt- wem- scharfe."
	Grms. per Litre.	Grms. per Litre.	Grms. per Litre.	Grms. per Litre.
Citron oil .....	0.54	..	..	..
Onanthic ether.	9.65	2.60	1.30	..
Peru balsam ...	21.80	..	..	..
Vanillin .....	0.29	0.03	0.20	..
Ethyl acetate ...	30.00	..	3.83	..
Ethyl butyrate .	Traces.	2.50	2.60	..
Ethyl formate...	Traces.	7.50	0.96	..
Acetic acid.....	..	..	0.29	..
Butyric acid ....	..	1.10	Trace.	..
Formic acid.....	..	2.60	..	..
Resin .....	5.50	..	..	..
Extract.....	..	1.40	47.31*	4.00
Ash.....	1.10	0.04	..	0.08
	Per Cent. by Volume.	Per Cent. by Volume.	Per Cent. by Volume.	Per Cent. by Volume.
Alcohol .....	77.00	87.09	59.84	98.00
Fusel oil.....	0.24	0.37	0.21	..

\* Containing grape sugar, 7.16; cane sugar, 9.00; ash, 0.36.

—A. G. B.

## PATENTS.

*Improvements in or Relating to a Process for the Saccharification and Fermentation of Amylaceous Matter.* W. P. Thompson, Liverpool. From La Société Générale de Maltosa, Brussels, Belgium. Eng. Pat. 16,093, October 12, 1889. 4d.

THE use of ammonium or sodium fluorides or the double potassium sodium fluoride is recommended in saccharification or fermentation as an antiseptic. A quantity about 20 grms. per hectolitre is suitable. Almost any other fluoride may be used for the same purpose.—A. L. S.

*An Improved Process for Manufacturing Non-alcoholic Wine, and for Utilising a By-product.* E. Dean, Hastings. Eng. Pat. 16,105, October 12, 1889. 4d.

GRAPES are placed in a boiler and heated to 212° F. The juice is separated from the skin and stones, boiled, a little gum arabic added, put into hot bottles, and hermetically sealed up.

The stones are separated from the skin, which is chopped up and made into jam in the usual way.—A. L. S.

*Novel Construction or Arrangement of Condensing Cylinders used for Aërating Malt and other Liquors.* D. Wickham, Ware. Eng. Pat. 16,556, October 19, 1889. 6d.

THE apparatus consists of a cylinder with moveable ends, each having a packing joint which are kept in gas-tight contact with the cylinder by long bolts arranged on the outside of the cylinder from one head to the other. This arrangement is very easy to clean. The agitator shaft passes through the ends and is turned by a handle outside. The interior is tinned or silvered. To use the apparatus carbonic acid and the liquor to be carbonated is pumped into the cylinder until the necessary pressure is reached, the liquor stirred by the agitator and bottled by any suitable machine. —A. L. S.

*An Apparatus for Cooling and Aërating Hot Worts for the Production of Malt Liquors.* M. Hanford, Boston, U.S.A. Eng. Pat. 4982, March 31, 1890. 8d.

By converting hot wort into a fine spray and allowing the spray to fall a short distance through the air of the cooling room on to the cooler, it is quickly aërated and cooled. An apparatus for the purpose consists of a vertical tube with a conical-shaped deflector fitted to its end, leaving a small annular space between the end of the tube and the deflector through which the wort flows being converted into a fine spray thereby. This annular opening, if regulated to be of the correct size at the commencement, soon ceases to act properly owing to an accumulation of gummy matter, &c. For the purpose of preventing this, the conical deflector is fastened to one end of a lever pivoted in the centre, and to the other end of the lever a weight is fixed so that at the commencement of the operation the aperture is of the correct size. If the aperture become too small by an accumulation of gummy matter, the pressure exerted on the deflector by the liquid will be greater, and the consequence will be that the deflector will be forced further away from the end of the tube and the annular space be enlarged to the correct dimensions. In order that the deflector shall not stick, there is a stop which prevents it coming too close to the end of the tube. A cord is attached to the lever so that by pulling it the annular aperture may be temporarily enlarged for flushing.

—A. L. S.

*A New or Improved Process for Preventing and Arresting the Development of Acids during the Process of Malting Barley or other Malted Cereals, such as Rye, Oats, or Wheat, and the Storage of the same after having been Malted, and in the Infusion whilst Macerating or Infusing Malted Barley, for the Manufacture of Concentrated Extract of Malt, or for Ale Brewing Purposes, or for the Manufacture of Malt Vinegar.* W. Paterson, Glasgow, Eng. Pat. 6337, April 25, 1890. 6d.

THE finest barley is chosen and malted as usual, except that all water used is made alkaline by potassium or sodium carbonate, or kept neutral by finely-divided calcium carbonate. The malt is stored in air-tight iron tanks which are filled with carbonic acid gas. The malt when required for use is ground in the usual way and mashed in a tub from which either the air is exhausted by a vacuum pump or displaced by carbonic acid. The mash, if required for the manufacture of malt extract, is neutralised with sodium carbonate or bicarbonate, the wort filtered and evaporated in vacuo, the access of air to the wort at any stage being prevented.—A. L. S.

## XVIII.—CHEMISTRY OF FOODS, SANITARY CHEMISTRY, AND DISINFECTANTS.

### (A).—CHEMISTRY OF FOOD.

#### PATENT.

*Improvements in Apparatus for Sterilising Milk and other Liquids, and for Drawing off or Decanting the same.* G. T. Redfern, London. From J. F. H. Gronwald, E. H. C. Oehlmann, and G. H. Nenhaus, Berlin, Germany. Eng. Pat. 11,554, July 23, 1890. 6d.

Two or three forms of the apparatus are described, but the following are the essential points common to all of them. The milk or other liquid is run into a number of chambers or vessels, which by their peculiar shape or arrangement offer a very large heating surface, so that the milk can be rapidly heated to the desired temperature, and the risk of its browning avoided. These vessels are enclosed in a large heating chamber, into which steam can be admitted, the pressure of the latter being regulated by a suitable valve. The vessel which is eventually to receive the milk is situated outside the chamber, and is connected with the milk vessels by means of a pipe. Air can enter or escape from it only by means of an air-filter. Before admitting the sterilised milk, this vessel is sterilised by blowing steam through it. Connexion with the external air is then cut off by closing various taps, the steam condenses, a vacuum is formed, and the milk is then admitted without coming in contact with the air.—F. W. T. K.

### (B).—SANITARY CHEMISTRY.

*The Pollution and Spontaneous Purification of Rivers.* Chem. Zeit. 14, 855.

At the annual meeting of the German Society of Gas and Water Engineers, Professor v. Pettenkofer read a paper with the above title.

No general statements can be made with regard to the spontaneous purification of rivers, but each case must be considered on its own merits. The great bulk of the sewage of Munich is conducted direct into the Isar. This is not considered to have any prejudicial effects on the inhabitants living on the banks of the river below Munich. The example of Rome, the sewage of which has run into the Tiber for more than 2,000 years, is quoted as upholding this opinion; it is also stated that the sewage of Hamburg has run into the Elbe for the last 40 years, and yet the water at Altona (40 miles below Hamburg) is

drinkable. The amount of organic matter introduced into the river by the Munich sewage amounts to 6 mgrms. per litre, and this is considered as insufficient to be hurtful. With respect to the danger which arises from the presence of pathogenic bacteria, it is known that for long the effluvia from hospitals and slaughter-houses has been allowed to pass direct into various rivers without ill effects, it is probable that these organisms do not find proper nourishment in the water, and are not able to hold their own in the struggle for existence, against the more numerous harmless bacteria. The spontaneous purification of rivers probably depends more on the velocity of the current than the volume of the water. It is probably unnecessary in many cases to pass the sewage through the land first, as under most circumstances it would do no harm if allowed to flow directly into the river.—A. L. S.

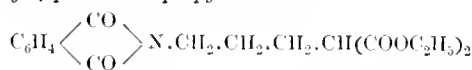
## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

*Synthesis of Homopiperidine Acid and Piperidine Acid* S. Gabriel. Ber. 23, 1767—1773.

By oxidising acid derivatives of piperidine and decomposing the oxidation products with hydrochloric acid, Schotten obtained homopiperidine acid,  $C_5H_{11}NO_2$ , which he proved to have the constitution of a  $\delta$ -amidovalerianic acid,  $NH_2 \cdot CH_2 \cdot CH_2 \cdot CH_2 \cdot CH_2 \cdot COOH$ , and piperidine acid,  $C_6H_9NO_2$ .

The author has succeeded in preparing homopiperidine acid synthetically, in the manner described below, and has also proved that piperidine acid is  $\gamma$ -amidobutyric acid,  $NH_2 \cdot CH_2 \cdot CH_2 \cdot CH_2 \cdot COOH$ .

*Ethyl- $\gamma$ -phthalimidopropylmalonate—*



can be obtained by heating the sodium derivative of ethyl malonate with bromopropylphthalimide in alcoholic solution. The mixture is boiled for an hour, the alcohol and the unchanged ethyl malonate distilled with steam, and the residual oil separated or extracted with ether; the product thus obtained is dissolved in boiling light petroleum, from which it separates after some time in compact crystals, melting at  $46^\circ$ — $48^\circ$ . When this compound (5 grms.) is heated at  $180^\circ$ — $190^\circ$  for three hours with hydrochloric acid of sp. gr. 1.13 (25 cc.) it is decomposed into ethylchloride, carbonic anhydride, phthalic acid and the hydrochloride of homopiperidine acid ( $\delta$ -amidovalerianic acid); the amido-acid thus obtained is identical with the compound prepared by Schotten from benzoylpiperidine.

When the hydrochloride of piperidine acid, prepared from piperidylurethane by Schotten's method (Ber. 16, 643), is converted into the free acid, and a crystal of  $\gamma$ -amidobutyric acid placed in its saturated solution in alcoholic ether, a colourless crystalline powder, which shows all the properties of  $\gamma$ -amidobutyric acid, is obtained; piperidine acid is, therefore, identical with  $\gamma$ -amidobutyric acid.

Schotten drew attention to the fact that although  $\delta$ -amidovalerianic acid is harmless, its anhydride, piperidone, is a powerful poison; he also showed that piperidine acid has no poisonous properties. The author has found that pyrrolidone, the anhydride of piperidine acid, has a poisonous action very similar to that of piperidine.

—F. S. K.

*Conversion of Tropidine into Tropine.* A. Ladenburg. Ber. 23, 1780—1781, and 2225.

WHEN tropidine is treated with hydrobromic acid in the cold it yields small quantities of a base which is not volatile with steam, and which forms a crystalline platino-chloride

containing the same percentage of platinum as the corresponding salt of tropine; only a very small quantity of this non-volatile base is produced, most of the tropidine remaining unchanged, but when the reaction is carried out at a higher temperature a better yield is obtained.

The product, prepared in this way, decomposes on distillation and seems to contain bromine; when freed from halogen by means of silver oxide it yields a strong base which, on treatment with tropic acid and hydrochloric acid, gives a solution showing all the properties of atropine, and possessing like the latter a mydriatic action.

Further experiments have shown (Ber. 23, 2225) that the base prepared in the cold is in fact identical with tropine; its platino-chloride resembles that of tropine very closely and has practically the same melting point; it also forms a sparingly soluble cadmio-iodide and a deliquescent hydrochloride.—F. S. K.

*Oil of Nutmeg and Oil of Mace.* F. W. Semmler.  
Ber. 23, 1803—1810.

OIL of mace, which consists essentially of terpenes, has been shown by Wallach (this Journal, 1889, 635) to contain pinene and dipentene. Oil of nutmeg has been investigated by Gladstone, Wright, and others, and has been found to contain two terpenes,  $C_{10}H_{16}$ , a very little cymene, and an oil (myristiccol), boiling at  $212-226^\circ$ , to which Gladstone assigned the composition  $C_{10}H_{14}O$ , and Wright the composition  $C_{10}H_{16}O$ ; the latter also obtained from the oil a liquid of the composition  $C_{10}H_{16}O_2$ , boiling at  $260-290^\circ$ , whilst a solid stearopten myristicin which was isolated by John was proved by Flückiger to be myristic acid. According to Wright, oil of nutmeg also contains a non-volatile resin of the composition  $C_{10}H_{16}O_3$ .

The results of previous investigators being in many cases either doubtful or self-contradictory, the author has commenced an examination of these two oils.

Oil of nutmeg, as obtained from Schimmel and Co., is colourless and transparent and has a specific gravity of 0.8611 at  $15^\circ$ ; it consists of a mixture of various terpenes and does not contain cymene, myristiccol, or any oils of high boiling point.

Oil of mace, from the same firm, has a specific gravity of 0.9309 at  $14^\circ$ , and gives a green colouration with an alcoholic solution of ferric chloride; on cooling it to  $-10^\circ$  no solid substance is deposited. On fractional distillation under a pressure of 10 mm., 53 per cent. passes over below  $71^\circ$ ; this fraction consists of terpenes and contains pinene and dipentene as was shown by Wallach (loc. cit.). The portion passing over between  $71^\circ$  and  $114^\circ$ , which forms 15 per cent. of the whole, should, according to Wright, contain the myristiccol  $C_{10}H_{16}O$ , but it has not yet been investigated. The remaining 31 per cent. (which has not been distilled) is practically identical with an oil that is sold as "High-boiling portions of oil of mace." This commercial article is yellow, gives a green colouration with alcoholic ferric chloride, and contains myristicin (see below), which is deposited in small quantities on cooling to  $-17^\circ$ ; the specific gravity of the oil is 1.1303 at  $14^\circ$ , and it dissolves in concentrated sulphuric acid, yielding a clear red solution. Under a pressure of 10 mm. it begins to boil at  $124^\circ$ , and on fractional distillation the distillate is resolved into at least two substances, but when carefully distilled over excess of sodium, under reduced pressure, a colourless highly refractive oil passes over at  $142-149^\circ$  (10 mm.) and solidifies on cooling to a colourless crystalline mass. This crystalline substance has the molecular formula  $C_{12}H_{14}O_3$ , and is named by the author *myristicin*. It has an odour of nutmeg, melts at  $30-35^\circ$ , and has a specific gravity of 1.1501 at  $25^\circ$ ; on distillation over zinc dust it yields benzene, showing that it is an aromatic compound.

*Dibromomyristicin*,  $C_{12}H_{14}Br_2O_3$ , is obtained when myristicin is treated with bromine in carbon tetrachloride solution until the bromine ceases to be rapidly absorbed. It crystallises from boiling light petroleum in colourless needles and melts at  $105^\circ$ .

Oil of mace contains about 22 per cent. of myristicin.

—F. S. K.

*Coniceines.* E. Lellmann. Ber. 23, 2141—2142.

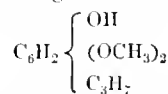
$\alpha$ -CONICEINE can be prepared, according to Hofmann, (1) by treating conydrine with hydrochloric acid; (2) by the elimination of hydriodic acid from the iodoconiine obtained by the action of hydriodic acid on conydrine; and (3) by the action of sulphuric acid on the bromoconiine in which the bromine atom is in combination with nitrogen.

The author's experiments have shown that these three methods all yield different coniceines, that prepared by the first method being probably the  $\alpha$ -compound. The bases obtained by the second and third methods, which are named  $\epsilon$ - and  $\delta$ -coniceine respectively, resemble the  $\alpha$ -compound in some respects, but differ from it in others.  $\alpha$ -Coniceine, for example, boils at  $158^\circ$ , and is hygroscopic;  $\delta$ - and  $\epsilon$ -coniceine boil at  $158^\circ$  and  $150-151^\circ$  respectively, and are not hygroscopic.  $\delta$ -Coniceine is levo-rotatory ( $-7^\circ 48'$ ), the  $\epsilon$ -base, on the other hand, is dextro-rotatory ( $42^\circ$ ). The hydrochloride of the  $\epsilon$ -base is very deliquescent, and that of the  $\alpha$ -compound moderately so, but the salt of the  $\delta$ -base is stable in the air. The picrate of  $\alpha$ -coniceine melts at  $224^\circ$ , that of the  $\delta$ -base at  $226^\circ$ , and that of the  $\epsilon$ -base at  $223-224^\circ$ ; the aurochlorides melt at  $196-198^\circ$ ,  $207^\circ$ , and  $178^\circ$  respectively, but the salt of the  $\epsilon$ -base does not decompose at its melting point, whilst that of the  $\delta$ -base undergoes slight, and that of the  $\alpha$ -base very considerable decomposition.—F. S. K.

*The Constitution of Apiol and of its Derivatives.* G. Ciamician and P. Silber. Ber. 23, 2283—2295.

PREVIOUS investigations of the authors have pointed out the analogy of apiol to safrol and methyl eugenol (this Journal, 1888, 399 and 583; and 1889, 1007). The present paper is a continuation of these researches. Isapiol, when oxidised by potassium permanganate in alkaline solution, behaves like all similarly constituted bodies, yielding, in addition to apioic acid, a ketonic acid. The latter is easily separated from apioic acid, owing to its ready solubility in water.

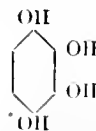
When reduced by sodium in alcoholic solution, isapiol behaves like isosafrol, two products resulting—a dihydro-isapiol,  $C_{12}H_{16}O_3$ , and a monovalent phenol,  $C_{11}H_{16}O_3$ . The dihydro-derivative is separated from the product of the reaction by treatment with water, when it rises to the surface and can be filtered off and recrystallised from alcohol, or be purified by distillation. The alkaline filtrate is extracted with ether after distilling off the alcohol, when the phenol is obtained as a thick yellow oil, boiling at  $277-278^\circ$ . It has the following constitution:—



Whilst isapiol and isosafrol are readily reduced as shown, apiol and safrol are practically unacted upon by sodium in alcoholic solution.

Ginsberg has shown (this Journal, 1888, 583) that isapiol when treated with bromine yields a tribromo-derivative, two of the bromine atoms of which are removed by treatment with zinc dust in alcoholic solution, whilst the third remains unchanged. A monobromo-derivative results. By treating isapiol in ethereal solution at  $-18^\circ$  with bromine, a dibromo-isapiol is formed, which melts at  $75^\circ$ . The dibromo-isapiol is decomposed by boiling water; treated with zinc dust in alcoholic solution, an oily product is formed which appears to differ from isapiol.

The authors have previously shown that apion is the dimethyl-methylene ether of a tetrahydroxyphenol apionol, and have succeeded in preparing apionol tetramethylether (this Journal, 1889, 1007). The four substituting groups in this ether, and therefore also in apionol, appear to have the relative positions 1, 2, 3, 4, whence apionol would have the constitution—



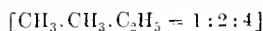
The two unsubstituted hydrogens are consequently in the 1:2 position. The former is obtained by treating apion in glacial acetic acid solution with nitric acid at  $-18^{\circ}$ , and forms yellow needles which melt at  $117^{\circ}$ — $118^{\circ}$ , and which are reduced to the latter by means of tin and hydrochloric acid. Diamido-apion forms prisms which melt at  $119^{\circ}$ , and yields azines with both di-acetyl and with di-benzil.

As to the constitutional formula of apion itself, it is evidently derived from 1, 2, 3, 4, tetrahydroxyphenol.

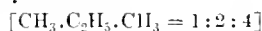
Eykmann (Ber. **23**, 857) has shown that the difference between apion and isapiol is due to the different structure of the  $C_3H_5$  group—the former containing the allyl group  $-CH_2 - CH = CH_2$ , and the latter the propenyl group  $-CH = CH - CH_3$ —C. A. K.

*Laurene.* E. Uhlhorn. Ber. **23**, 2346—2349.

THE author has studied the products obtained when Japan camphor is distilled with chloride of zinc, with a view of clearing up the conflicting statements as to the nature of the hydrocarbons formed. He finds that the fraction boiling between  $180^{\circ}$ — $195^{\circ}$  contains two ethylxylenes. The ortho body—



and the para body—



the fraction from  $185^{\circ}$ — $187^{\circ}$  probably contains the ethyl-meta-xylene—



Several derivatives of these hydrocarbons are described. —C. A. K.

*Propylxylenes.* E. Uhlhorn. Ber. **23**, 2349—2351.

*Propylortho-xylene* [ $CH_3 \cdot CH_3 \cdot C_3H_7 = 1:2:4$ ] prepared by Fittig's method from monobromo-*o*-xylene and propyl bromide, boils at  $209^{\circ}$  and remains liquid at  $-20^{\circ}$ . Its sulphonic acid forms thin needles and yields characteristic barium, magnesium and sodium salts. The sulphamide, sulphanilide, and tribromo-propyl-xylene are described. The hydrocarbon yields para-xylic acid (melting point  $162^{\circ}$ ) on oxidation.

*Propyl-meta-xylene* [ $CH_3 \cdot CH_3 \cdot C_3H_7 = 1:3:4$ ] boils at  $205^{\circ}$ — $208.5^{\circ}$ , and remains liquid at  $-20^{\circ}$ . Similar derivatives to the above are described, also the trinitro derivative of the hydrocarbon. On oxidation xylic acid (melting point  $126^{\circ}$ ) results.

*Propyl-para-xylene* [ $CH_3 \cdot C_3H_7 \cdot CH_3 = 1:2:4$ ] boils at  $206^{\circ}$ — $207^{\circ}$  and remains liquid at  $-20^{\circ}$ . Similar derivatives to the above are again described.

The isopropylxylenes could not be prepared directly by Fittig's method, but the meta product, isopropyl-meta-xylene [ $CH_3 \cdot CH_3 \cdot CH(CH_3)_2 = 1:3:4$ ] was obtained from isopropylbenzene by converting this into the mono-bromo-derivative and then substituting the bromine in the resulting monobromopara-isocymol by methyl. The hydrocarbon boils at  $194^{\circ}$ — $195^{\circ}$ ; its sulphonic acids and salts thereof are described, also the sulphamide, sulphanilide, tribromo- and trinitro derivatives.—C. A. K.

*Melting Points of Quinine, Cinchonidine, and their Isomerides.* O. Hesse. Annalen, **258**, 133—144.

THE author has repeated his determinations of the melting points of these substances (Ann. **135**, 327; **176**, 207) using Roth's apparatus (Ber. **19**, 1970; this Journal, 1886, 617). Anhydrous quinine obtained by drying the trihydrate over sulphuric acid and heating to  $115^{\circ}$  or  $120^{\circ}$ , melts at  $171.2^{\circ}$ — $172^{\circ}$ , when obtained by heating the benzene compound  $C_{20}H_{24}N_2O_3 \cdot C_6H_6$  to  $120^{\circ}$ , it melts at  $171.6^{\circ}$ — $172^{\circ}$ . The anhydrous isomeride formed by the prolonged heating of solutions of quinine in dilute alcohol, &c. at  $30^{\circ}$ , and which may be termed quinine anhydride or *Homo-quinine* melts at  $174.4^{\circ}$ — $175^{\circ}$ . It is reconverted into quinine by the prolonged action of dilute sulphuric acid.

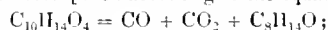
Cinchonidine melts at  $202^{\circ}$ — $202.8^{\circ}$ , and is readily converted into its isomeride homocinchonidine, which melts at  $207^{\circ}$ — $208.2^{\circ}$ . The latter may be reconverted into cinchonidine by repeated crystallisation from dilute sulphuric acid (Ann. **205**, 194; this Journal, 1888, 350).

The  $\beta$ -cinchonidine obtained by the author by the action of hydrochloric acid on cinchonidine (Ann. **205**, 327) melts at  $206^{\circ}$ — $207^{\circ}$  but acquires a brown colouration. In an acid solution it polarises more strongly than homocinchonidine; the sulphate resembles the homocinchonidine salt, and may be similarly converted into the cinchonidine compound, but it is more soluble in water. It may, therefore, be assumed that the first product of the action of hydrochloric acid (sp. gr. = 1.125) upon cinchonidine at  $140^{\circ}$  consists of homocinchonidine, which is further partially converted into apocinchonidine. In the presence of concentrated sulphuric acid only the first change takes place.

—S. B. A. A.

*Conversion of Compounds of the Terpene Group into Metaxylene.* O. Wallach. Annalen, **258**, 319—340.

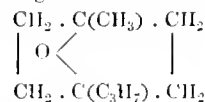
CINEOLIC acid,  $C_{10}H_{16}O_3$  (this Journal, 1888, 586), is a dibasic acid; its methyl salt,  $C_8H_{14}O(CO_2CH_3)_2$ , melts at  $31^{\circ}$ ; the anhydride,  $C_{10}H_{14}O_3$ , prepared by distilling a solution of the acid in acetic anhydride, and removing the excess of solvent in a vacuum at a low temperature, melts when pure at  $77^{\circ}$ — $78^{\circ}$ , boils at  $157^{\circ}$  under a pressure of 12—13 mm., dissolves in chloroform and benzene, separating on addition of light petroleum in long needles. It is reconverted into the acid by boiling with water. By dry distillation cineolic anhydride is decomposed according to the equation—



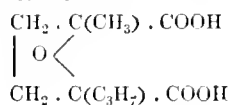
cineolic acid on the other hand is partially converted into the anhydride and partly decomposed into carbonic anhydride and a monobasic acid,  $C_8H_{14}O_3$ .

The compound  $C_8H_{14}O$  is a liquid with a pleasant penetrating odour, boiling at  $173^{\circ}$ — $174^{\circ}$ , V.D. = 4.19. Sp. gr. at  $20^{\circ}$  = 0.8530. Index of refraction  $n_D = 1.44003$ . It is an unsaturated compound which absorbs bromine and the halogen acids and is oxidised in the cold by potassium permanganate with evolution of carbonic anhydride; in contact with solid caustic potash it becomes warm and is partially resinified; with sodium bisulphite it forms a crystalline unstable addition compound. It thus appears to contain doubly linked C atoms and a ketonic (or aldehydic?) group. When it is heated with dry zinc chloride first at  $95^{\circ}$  and subsequently at  $145^{\circ}$ , the product may be separated by fractionation into two portions, boiling at  $132^{\circ}$ — $134^{\circ}$  and  $280^{\circ}$ — $285^{\circ}$  respectively. The fraction (132—134) has the formula  $C_8H_{12}$ , its odour recalls that of xylene, its sp. gr. at  $20^{\circ}$  = 0.8275, index of refraction at  $20^{\circ}$   $n_D = 1.4675$ , V.D. = 3.74. Like the terpenes it combines with the halogens and halogen acids, but it differs from them in its conversion into nitro-xylene by the action of nitric acid.

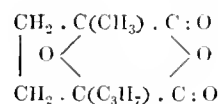
The fraction ( $280^{\circ}$ — $285^{\circ}$ ) contains a polymeride having the formula  $C_{16}H_{24}$ . The identity of this xylene is established by the coincidence of the melting points of a large series of nitro- amido- and acetyl- derivatives with the corresponding compounds prepared from metaxylene. The authors regard cineol as having the constitution—



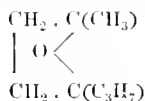
cineolic acid thus becomes—



and the anhydride—



the compound  $C_{10}H_{16}O$  which should accordingly have the constitution—



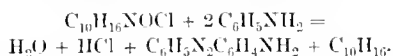
appears, however, to open out into the unsaturated ketone  $CH_3 \cdot CO \cdot CH_2 \cdot CH : CH \cdot CH(CH_3)_2$  in a manner analogous to the formation of pinacolone from pinacol. The closed xylene chain is re-formed when water is eliminated from the molecule by the combination of the oxygen in the CO with two hydrogen atoms from the  $(CH_3)_2$ .—S. B. A. A.

*The so-called "Massoyene."* O. Wallach. *Annalen*, **258**, 340—343.

Woy (*Archives der Pharm.* **228**, 22) obtained from Massoy bark a new terpene boiling at  $172-173^\circ$  having a specific gravity of 0.8581 at  $18^\circ$  and yielding a bromide melting at  $93^\circ$ . The author finds that by steam distilling and fractionation this terpene a considerable proportion passes over between  $160-165^\circ$ , this fraction forms like pinene a nitrosochloride, which yields a nitrobenzylamine melting at  $123^\circ$ ; it is converted by alcoholic potash into the nitrosopinene, melting at  $133^\circ$ . The other fraction boils between  $170^\circ$  and  $175^\circ$ , and yields on bromination in a glacial acetic acid solution dipentenetetra bromide melting at  $123^\circ$ , together with some impure by-products with inconstant melting points ( $97-104^\circ$ ) which probably consist of a mixture of limonene and dipentene tetra bromides; no bromide melting at  $93^\circ$  was obtained. The combinations with the halogen acids  $C_{10}H_{16} \cdot 2 HCl$ , &c. obtained by Woy render it probable that the second terpene in massoy-bark oil is limonene containing a small proportion of dipentene; the presence of limonene is also indicated by the melting point ( $93^\circ$ ) of the nitrolamine obtained from the fraction ( $170-175^\circ$ ). Woy's massoyene thus appears to be a mixture of limonene, pinene, and dipentene.—S. B. A. A.

*Pinene.* O. Wallach. *Annalen*, **258**, 343—347.

CHEMICALLY pure pinene may be prepared by treating pinenenitrosochloride obtained as described (*Ann.* **253**, 251; this *Journal*, 1889, 959) with aniline (*Ann.* **252**, 132), steam distilling the product and agitating the distillate in the cold with water and excess of acetic acid; the light oil which separates is thoroughly washed with water and acetic acid. The following reaction takes place:—



Pinene prepared in this way boils at  $155-156^\circ$ , sp. gr. = 0.858 at  $20^\circ$ , 0.854 at  $25^\circ$ . Index of refraction  $n_D = 1.46553$  at  $21^\circ$ . It is inactive to light and remains so after treatment with hydrogen chloride. It is not therefore like the so-called "inactive pinene," an accidental mixture of nearly equivalent quantities of the dextro- and levo-gyrate varieties in which the components are distinguishable by the action of reagents. Inactive pinene combines with nitrosyl chloride like the active modification, pinol also occurring as a by-product (*Ann.* **253**, 249).

By reducing a glacial acetic solution of nitrosopinene,  $C_{10}H_{15}NO$ , with a solution of hydriodic acid in the same solvent or with zinc dust an oily base  $C_{10}H_{15}NH_2$  is obtained. This compound smells like borneol, distils without decomposition at  $98-99^\circ$  under a pressure of 22—23 mm. and between  $207-208^\circ$  at the ordinary pressure. It differs greatly from the isomeric carvylamine (*Ber.* **20**, 486) and from amidoterebenthene (*Gazz.* **16**, 228, 341), the hydrochloride of the new base melting at  $221-222^\circ$  and the benzoyl derivative at  $116^\circ$ . The sulphate, unlike the amidoterebenthene salt, is a stable well-crystallised compound.—S. B. A. A.

*On the Physiological Action of Uranium.* J. Woroschilsky. *Chem. Zeit.* **14**, 1002—1003.

SINCE the discovery of uranium about 100 years ago by Klaproth, its action on the animal organism has been frequently investigated, the first to do this being Gmelin, in 1824. The author has recently studied the physiological action of this element, and has verified and extended the observations of previous experimenters. The action of it has been tried under the most varied conditions on mammals, birds, frogs, and worms; the results show that uranium, however administered, is a very poisonous metal. Uranium in fact has been included in the list of poisonous substances in the Russian pharmacopœia.—A. W.

*Artificial Musk.* A. Baur. *Chem. Zeit.* **14**, 1094—1095.

THIS new commercial product is chemically quite distinct from real musk, as it is a nitro-derivative of isobutyltoluene. The latter hydrocarbon, which is also found in the so-called essences of rosin, is best prepared by Friedel and Crafts' method. The chief product is a meta derivative. This is nitrified with a mixture of strong nitric and sulphuric acids first in the cold and finally by heating on the water-bath for 24 hours. The nitro compounds produced are separated in the usual manner, and again nitrified as above to ensure complete conversion into the trinitro-derivative. The latter is finally crystallised from alcohol, when yellowish white needles melting at  $96-97^\circ$  are obtained, insoluble in water and possessing a distinct smell of musk. By acting on a solution of isobutyltoluene in glacial acetic acid with nitric acid the mononitro-derivative is produced which has an unpleasant smell and may be converted by further nitration into the trinitro-derivative. The former can be distilled with aqueous vapour; it is an oil which distils in vacuo at  $160-162^\circ$ .

Hepp has examined the physiological action of artificial musk, and finds that large quantities even are without effect on rabbits.

Isobutylmetaxylene also gives a trinitro-derivative possessing a musk-like smell, but neither as strong nor as pleasant as the trinitro-derivative of the first-mentioned hydrocarbon. (See following abstract; also this *Journal*, 1889, 1004.)—A. R.

*Musk.* *Monit. Scient.* 1890, **4**, 718—721.

**I. NATURAL MUSK.**—Musk is a secretion of the male musk rat (*Moschus Moschiferus*). It occurs in a small pouch which is situated below the stomach, and furnished at the posterior end with a small duct through which the musk is discharged. The quantity of musk found in the pouch is very various; 28 grms. may be considered the average quantity in an adult animal. Other animals, such as the civet, &c., and some plants, as *Adora moschatellina*, &c., yield similar substances.

Musk is a brownish slimy solid, with a sharp, slightly bitter taste and a disagreeable odour when strong, which becomes pleasant on dilution. It is a very complex body, containing nearly half its weight of water. It occurs in commerce both in the pouches and in bulk. The price is subject to considerable variations, Nankin musk being the most valuable. It is often adulterated, frequently by introducing foreign bodies into the pouches to increase their weight, and sometimes by perforating the pouches and dissolving out some of the substance. These falsifications are, however, easily detected. All pouches which show any sign of having been tampered with may be considered as adulterated. Musk in the bulk is much more easily adulterated; to determine if this is so, a little of the substance is warmed with glycerin or turpentine, and on cooling observed under the microscope. Pure musk forms a mass of yellow granules easily distinguished from any foreign matters which may have been added. It contains 45—46 per cent. water, and yields on incineration 4—6 per cent. grey ash; any divergence from these numbers indicate adulteration. Blood and meat are often added; these may be detected by a microscopic examination.

**H. ARTIFICIAL MUSK.**—Schauffer and Hupfeld patented a process in 1888 (this Journal, 1890, 102) for preparing artificial musk. Three kilos. of dimethyl benzene, 2 kilos. of isobutyl alcohol, and 9 kilos. of zinc chloride are heated together in an enclosed vessel for 7—8 days. The product is distilled, the fraction passing over  $198^{\circ}$ — $230^{\circ}$  only being kept and nitrated by fuming nitric acid. The product is washed and dissolved in alcohol, and crystallised from the solution. Its formula is  $C_{12}H_{16}NO_2$ . Another method is to use toluene or xylene with isopropyl or isosamyl alcohol and zinc chloride, and proceed as above.

Baur's patent (this Journal, 1889, 1004) consists in the preparation of meta-methyl-isobutyl-benzene by acting on isobutyl chloride by toluene in the presence of aluminium chloride and nitrating the product. Artificial musk is not a very satisfactory preparation, and its use is only limited.

To distinguish between natural and artificial musk, use is made of the property which sulphate of quinine possesses of destroying completely the odour of artificial musk without affecting in any way the natural product. Other bodies, such as bitter-almond oil, and all bodies containing benzoic aldehyde or hydrocyanic acid, possess the same property.—A. L. S.

*On Orcine.* A. Donner. Pharm. Zeit, 1890, 418.

See under XXIII., page 974.

*On Strychnine.* H. Beckurts. Arch. Pharm. 1890, 313.

PURE strychnine melts at  $265^{\circ}$ . The ferrocyanide is gradually converted by the action of light and air into the ferricyanide. The monobromide is prepared by adding 1 mol. of bromine in aqueous solution to 1 mol. of strychnine hydrobromide. On adding ammonia it is precipitated and on recrystallising from alcohol melts at  $222^{\circ}$ . The hydrochloride, hydrobromide, and nitrate of strychnine crystallise without water, the sulphate with 7 mols. of water. Bromostychninemethylodide, the ammonium base, nitro-bromostychnine, amidobromostychnine, and bromostychninedibromide are also described.—A. R.

*On Brucine.* H. Beckurts. Arch. Pharm. 1890, 426.

On adding an aqueous solution of 1 mol. of bromine to 1 mol. of brucine hydrobromide a violet precipitate is first formed, which changes through brown to a pure yellow. After washing and drying over sulphuric acid it is found to have the composition of a brucine tribromide or hydrobromide of dibromobrucine. Strychnine under the above conditions (see previous abstract) gives a monobromide. By shaking brucine with chlorine water a dark red powder is obtained, dichlorobrucine.—A. R.

*Report on Volatile Oils and Essences.* Relazione nel Lab. Chim. Centr. delle Gabelle, 119—121.

THE Custom house tariff distinguishes between "an essence properly so called" (i.e., an essential oil), and "an alcoholic essence." The latter is classed as alcoholic perfumery, or aromatic alcohols, and pays the surcharge on the spirit. The following rapid and sufficiently exact method for the estimation of the alcohol is proposed. To 4—5 cc. of the essence to be examined an equal volume of distilled water and of a mixture of two-thirds glycerol with one-third water is added and the whole allowed to stand. If alcohol is present there is found a milky turbidity in the layer below the essential oil, while the diminution of volume of the oil is approximately equal to the volume of the alcohol added.

Analyses are given of five disputed samples, and allusion is made to certain samples of essences of geranium and rose destined for the State manufacture of tobacco.—V. H. V.

*A New Monobromocamphor.* J. E. Marsh. J. Chem. Soc. (Trans.), 1890, 828—833.

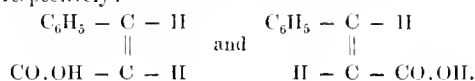
$\beta$ -MONOBROMOCAMPHOR is best prepared by the action of bromine on camphor in presence of alcohol. The ordinary or  $\alpha$ -monobromocamphor is also formed, the two being

separated by fractional crystallisation from alcohol in which the  $\beta$ -compound is more soluble. When bromine acts on camphor in presence of alcohol, the substitution appears to take place at once without formation of the additive compound  $C_{10}H_{16}OBr_2$ , whereas when bromine acts on camphor alone, the additive compound formed at first decomposes on raising the temperature with evolution of hydrobromic acid. By the latter method, on distillation, a liquid is obtained boiling at  $160^{\circ}$  under 20 mm. pressure, and containing an amount of bromine midway between mono- and dibromocamphor.

$\beta$ -monobromocamphor after distillation in vacuo forms, on solidifying, an almost transparent mass, easily breaking up into a soft granular powder, very different in appearance from the hard crystals of the ordinary modification. The rotatory power is for  $[\alpha]_D = +34.9^{\circ}$ , the ordinary modification giving  $[\alpha]_D = +139^{\circ}$ . When distilled under ordinary pressure the new isomer begins to boil at  $250^{\circ}$  and distils fairly constantly at  $265^{\circ}$ . The distillate crystallised from alcohol gives the hard crystals characteristic of the ordinary modification. It is thus evident that the  $\beta$ - is converted into the  $\alpha$ -modification by distilling under ordinary pressure, while no change is effected by distilling in vacuo.—J. B. C.

#### ERRATUM.

For the two constitutional formulæ given in the May number of this Journal, page 541, top of second column, read respectively:—



## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

*Etching on Glass by Photomechanical Processes.* A. Müller-Jacobs. Zeits. f. angew. Chem. 1890, 451—453.

THE photomechanical processes for etching on glass which are already known are either applicable only to fine subjects or present great practical difficulties when applied on a large scale. The author's process is based on the fact that when films of the resinat colours (this Journal, 1889, 770) are exposed to light they undergo change which, although almost invisible, is rendered evident by their behaviour towards various reagents. The dyestuff separates from the metallic resinate, and can be removed by means of alcohol or even hot water, in which it was previously insoluble, or can be bleached by reagents which had no action on it before exposure to light. After the action of the bleaching agent or solvent there is left a film of the metallic resinate which is insoluble in benzene, but can be removed by the action of oxidising agents.

When an exposed film is treated with benzene previously mixed with a large proportion of turpentine, the insolated portions remain undissolved whilst the unexposed parts pass into solution, the action of the solvent being inversely proportional to the action of the light. By this mode of development, as well as by treatment with oxidising agents, the gradations of the object which has been copied can be reproduced. Development by oxidation yields a positive image and development by benzene and turpentine a negative image. Development similar to that produced by oxidation is obtained when the plate is merely heated.

The exposed portions of the film which have not been affected by the developer offer great resistance to the action of acids, and are impermeable to them to an extent depending on the time of exposure. It follows that heliogravures on metal or glass can be produced by means of the resinat



colours, the resins themselves providing the necessary grain. The adhesion of the film of resinate to the glass is secured by mixing the benzene solution of the resins with india-rubber solution, a treatment which at the same time improves the grain. If too much india-rubber is applied, the sensitiveness is reduced; if too little, there is a tendency for the film to leave the glass.

*Preparation of the Sensitive Resinate.*—100 grms. of clear colophony are boiled with 10 grms. of sodium hydroxide, 33 grms. of crystallised sodium carbonate, and 1,000 cc. of water for two hours. 1,000 cc. of water is then added and the liquid is mixed with a filtered solution of 7.5 grms. of methyl-violet 3 B. (methyl-green, magenta, safranin, rhodamine, auramine, chrysoidine, Nile blue, Victoria blue, &c.) in about 500 cc. of boiling water. The resinate colour is precipitated by gradual addition of a solution of 60 grms. of magnesium sulphate, well washed, and dried at 60°–80°.

*Preparation of the India-rubber Solution.*—50 grms. of caoutchouc are digested with 4,000 grms. of carbon bisulphide for a long time, then heated in a water-bath until about half the bisulphide has distilled off, and pure benzene is added in sufficient quantity to make the total weight 3,333 grms.

*Preparation of the Sensitive Film.*—20 grms. of magnesium resinate violet, 8 grms. of the resinate green, 8 grms. of the resinate chrysoidine, and 4 grms. of the resinate magenta are dissolved in 130 cc. of pure benzene and 70 cc. of chloroform, and the solution is mixed with 120 cc. of the caoutchouc solution. After standing 8 days the mixture is filtered through cotton-wool and kept in the dark.

Well-cleaned glass or metal plates are coated with the mixture, dried for six hours, and exposed under a negative to direct sunlight, the time of exposure varying from 15 minutes to 3 hours. Red resinate colours increase the sensitiveness, blue and green colours reduce it. For deep etching thin films and long exposures are required.

The exposed plate is allowed to cool in the dark, and is then developed with a mixture of 1 part of benzene and 3 parts of turpentine (in summer, 1:4) at the ordinary temperature in the same way as a carbon print. When development is complete the plate is washed with light petroleum, the edges are coated with asphalt and the etching conducted in the usual manner. For matt etching on glass fuming hydrofluoric acid mixed with 10 per cent. of water may be used. If the action of the acid is continued too long the grain is destroyed and no matt surface is obtained, especially if the exposure has been short and the development has been carried too far. For deep etching an exposure of several hours is required.

When the plate is exposed under a negative, developed with benzene, and etched, and some colouring matter, such as white lead, is rubbed in the etched portions, an image is obtained, which, when placed against a black background, shows as a very clear diapositive. Instead of white lead, glass colours may be used and can afterwards be burnt in.

—C. H. B.

## PATENTS.

*New or Improved Lifters for Photographic Plates, Papers, and Films.* T. Stanway, Hauley. Eng. Pat. 16,528, October 19, 1889. 6d.

A PLAT or ribbon-like piece of ebonite, vulcanite, metal or other suitable material is bent into a loop of such form that it readily fits the corner of a photographic plate, and at the same time has a handle or finger-hole by which it can be lifted. Two such holders are used and they are applied to two diagonally opposite corners of the plate. Paper and films are placed on glass plates of suitable size, and at the corners to which the holders are not applied the paper or film is kept in contact with the glass by means of small india rubber bands.—C. H. B.

*An Improved Method of Preparing Photographic Plates of Mica.* E. Edwards, London. From O. Moh, Goerlitz, Germany. Eng. Pat. 11,104, July 16, 1890. 4d.

PLATES or sheets of mica are treated with a solution of chrome alum mixed with gelatin, in order "to remove mineral grease," and are afterwards coated with gelatinobromide emulsion in the usual way. The preliminary treatment with chrome alum and gelatin makes the emulsion adhere firmly to the mica.—C. H. B.

## XXII.—EXPLOSIVES, MATCHES, Etc.

*Report on the Circumstances attending an Explosion of Fireworks at Mr. James Pain's Firework Factory, near Mitcham, July 6, 1890.* Col. V. D. Majendie, C.B.

IN this report Colonel Majendie draws attention to the fact that the explosion did practically no damage beyond the building in which it occurred, even although more than three tons of fireworks of the most explosive character were contained in it, and the distance between the destroyed building and the other parts of the factory were moderate. This he considers shows the advantage of having such factories laid out on intelligent principles and with due regard to the risks inseparable from this class of manufacture.

There were present in the magazine three classes of fireworks more or less liable to spontaneous heating with a risk of ignition, viz., gerbes, blue stars, and asteroids and green lances. The blue stars and asteroids both contained "Chertier's copper," which is very liable to become acid, and although the samples of these fireworks examined were neutral this ingredient is liable to dangerous change during keeping, and ought not to be employed in firework mixtures. Chertier's copper is a mixture of potassium chlorate and copper sulphate, which has been moistened with ammonia and dried. When freshly made it is alkaline, but in time loses ammonia and becomes acid.

The gerbes also are liable to heating when damp, and although they are not likely to ignite spontaneously, the heat evolved might greatly endanger the stability of other fireworks in their neighbourhood. The gerbes contained iron filings, and although the filings have a protective coating of marine glue or similar composition, an elevation of temperature occurs when they are moistened. Although these fireworks just referred to are undoubtedly dangerous and might give rise to an explosion, in this particular instance the evidence goes to show that the accident may be referred to the spontaneous ignition of one of the green lances.

A few days after this accident a fire broke out in the same factory by the observed spontaneous ignition of one of the green lances, identical with those in the magazine. These lances contained "white" and "green fire" mixtures, the former consisting of potassium nitrate, flowers of sulphur, and antimony sulphide; the latter of barium and potassium chlorates, shellac, and charcoal. Dr. Dupré points out that "kept separate, neither of these mixtures would be liable to spontaneous decomposition, but when brought into contact they become dangerous. If these lances are filled with great care so that the two compositions are in contact only at the sectional area along which they touch, the danger is reduced to a minimum, though not perhaps wholly absent. But if, during the filling in of the two compositions, one upon the other, any intermixture takes place at the junction, a condition of things will be brought about which, under favourable conditions as to temperature, moisture, and retention of heat is certain, sooner or later, to lead to spontaneous ignition."

Several lances were cut open very carefully and in most cases the filling had been done with great care, but in one

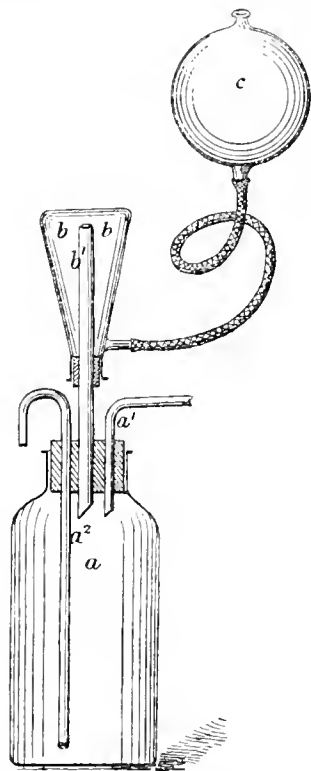
case decided intermixing had taken place, and thus formed (flowers of sulphur and barium chlorate) one of the most dangerous firework compositions. Col. Majendie trusts that the lessons which this accident are calculated to teach will induce firework makers to exclude from their factories those particular elements of risk of which this accident affords a further and striking illustration.—W. M.

## XXIII.—ANALYTICAL CHEMISTRY.

### APPARATUS.

*Sulphuretted Hydrogen Apparatus.* H. Wilde. Chem. Zeit. **14**, 905—906.

The apparatus shown in the figure resembles in principle that previously described (this Journal, 1888, 523) by the author in conjunction with Pollak.



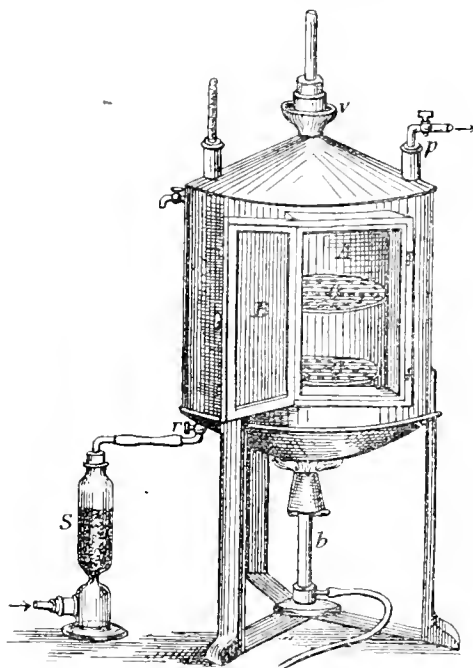
SULPHURETTED HYDROGEN APPARATUS.

The solid material, *e.g.*, ferrous sulphide, is placed in *a* and the acid in *b* and *c*. By opening a stopcock on *a1* (not shown) acid finds its way to the small hole at the top of the tube *b1* and trickles down causing a steady evolution of gas; *c* should be big enough to take any overflow from *b* consequent on closing *a1*. The spent liquor can be forced out at *a2* without dismounting the apparatus. To give a regular stream of gas the hole at the top of *b1* should be small, 1 mm. sufficing for a supply of 100 litres per hour; *b1* itself on the contrary must be wide to allow of the unhindered flow of acid downwards; 2 cm. is a suitable width.—B. B.

*Apparatus for Drying Substances in a Rarefied Atmosphere.* D. Sidersky. Zeits. Anal. Chem. **29**, 280—282.

*A* is a double-walled copper cylinder about 30 cm. high and 26 cm. diameter; the inside ends are flat but the outside

ends are arched. The door *B* is a thick glass plate and closes hermetically against an india-rubber washer. Water is poured in through the funnel opening *c*, which is then

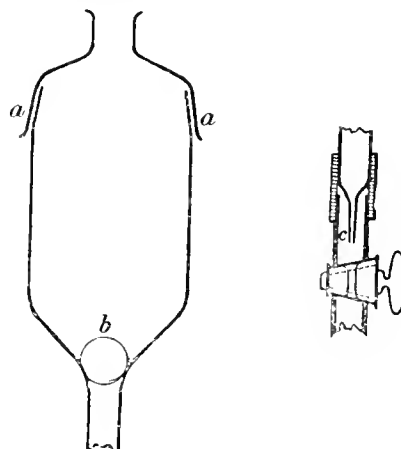


DESICCATION IN A RAREFIED ATMOSPHERE.

closed with a cork having a long glass tube running through it to act as a condenser, and when it has been brought to the boiling point by the burner *b*, the air in *A* is rarefied by connecting the stopcock *p* with an aspirator. Should it be desirable to use a current of air the stopcock *r* can be opened and air drawn through the purifier *S*.—A. G. B.

*An Improved Apparatus for Decompositions in the Cold and under Diminished Pressure.* J. W. Brühl. Ber. **23**, 1460.

This is an improvement on an apparatus devised by the author (Ber. **22**, 236) for diazotising, for preparing leuco-compounds, and for the formation and separation of crystalline substances at any temperature, and in an atmosphere of any gas.



It consists of a cylindrical glass vessel closed at *a* with a glass cap ground on, and communicating at the base with a long tube, the entrance to the tube being closed by a platinum cone, or in its latest form by a glass or earthenware marble *b*. At the base of the tube, which is considerably contracted, *c*, a glass stopcock, is attached with rubber tubing.

Marbles varying in size from a cherry upwards may often be used with ordinary funnels, with or without a pump to replace filter plates, and give excellent results even with finely divided crystals.—F. H. L.

### INORGANIC CHEMISTRY.— QUALITATIVE.

*Tables for the Rapid Determination of the Common Minerals.* A. J. Moses. *School of Mines Quarterly*, **11**, 334—355.

THE two general methods for the rapid determination of minerals are those of von Kobell (Munich, 1884), and Weisbach (Leipzig, 1886); in both there is first a division into minerals of metallic lustre and minerals of non-metallic lustre, but in the one they are then grouped by their behaviour before the blowpipe and with acids, whilst in the other the classifying characters are the external signs of hardness, colour, streak, tenacity, &c. The author now combines, in a series of tables both systems of examination.

—T. L. B.

*On the Presence of Boracic Acid in Caustic Alkalis.* F. P. Venable and J. S. Callison. *Journ. Anal. Chem.* 1890, 196.

SEVERAL samples of caustic alkalis from Schuehardt, Marquart, and Trommsdorf described as chemically pure, were found to contain boracic acid. Meissl's method for determining boracic acid in milk was employed, *i.e.*, the alkalis were just saturated with acid in a platinum dish, the salts moistened with a little dilute hydrochloric acid (1:100), a few drops of tincture of turmeric added and then evaporated to dryness. Traces of boric acid causes a cherry or cinnabarred colouration. The flame test was also used. From the degree and delicacy of the reaction, it may be assumed that the quantity of boracic acid in the alkalis exceeded 1 per cent. This should be taken into account in quantitative determinations of boracic acid as caustic potash is frequently employed in them.—A. R.

*The Action of Zinc on Dilute Sulphuric Acid.* F. Püllinger. *J. Chem. Soc. (Trans.)* 1890, 815—827.

See under X., page 946.

### INORGANIC CHEMISTRY.— QUANTITATIVE

*On a Source of Error in Determinations of Sulphuric Acid.* E. von Meyer. *J. prakt. Chem.* **42**, 1890, 270.

HOLTZWART found, in the author's laboratory, that during the evaporations of a large volume of liquid over a gas flame, appreciable quantities of sulphuric acid were derived from the gas and collected in the liquid. Two litres of pure water were evaporated on a high-standing water-bath, heated over the gas, to a volume of 50 cc., an operation that took six hours. It was then found to yield 0.0426 grm. of  $\text{BaSO}_4$ , equal to 0.01462 grm. of  $\text{SO}_3$ , or 0.00731 grm. per litre. When the same volume was evaporated by distillation, no trace of sulphuric acid was found.

A similar experiment has been repeated by Bechert, at the author's request. One litre of pure water was gradually evaporated in a platinum dish, always keeping about

40—50 cc. water in the dish, over a small Bunsen flame. The time required was 12 hours. The residual water yielded 0.0309 grms. of  $\text{BaSO}_4$ , equal to 0.0106 grm. of  $\text{SO}_3$ . This relatively larger amount than found by Holtzwardt, is explained by the longer time the evaporation required. These observations point to the necessity of precaution being taken in water analyses and in all sulphuric acid estimations in which a large volume of liquid has to be evaporated.—H. S. P.

*Valuation of Zinc Dust.* G. Klemp. *Zeits. Anal. Chem.* **29**, 253—266.

THE author has devised a process for the above purpose which depends on the power of zinc to reduce potassium iodate to potassium iodide in an alkaline solution; when the solution is afterwards acidified iodine is liberated by the interaction of the iodine and hydriodic acids, and is distilled off from the reaction mixture into potassium iodide solution, and titrated with thiosulphate. The details of the method are as follows:—

0.5—1 grm. of the well-mixed zinc dust is weighed in a tube and poured into a 200 cc. stoppered flask, the tube being again weighed. For every 0.1 grm. of zinc present, 10 cc. of a solution of alkali (containing 370 grms. of potassium hydroxide, or 300 grms. of sodium hydroxide per litre) and 3 cc. of a solution of potassium iodate (containing 15.25 grms. of potassium iodate per 300 cc.) are measured into a beaker and then poured into the flask. Some glass beads are added, and the flask stoppered and shaken for five minutes in the cold, no advantage being gained by heating it; its contents are then washed into a 250 or 500 cc. flask, and made up to the mark with water. 100 cc. are now pipetted into the retort of a Töpfer's apparatus (*Zeits. Anal. Chem.* **26**, 293), dilute sulphuric acid is added and the apparatus filled with carbonic anhydride. A solution of potassium iodide is placed in the receiver and the retort heated, at first gently and then more strongly till the contents are perfectly colourless; the stream of carbonic anhydride is continued throughout the distillation, which generally occupies about 20 minutes. The solution of iodine in potassium iodide is then transferred from the receiver into a flask, a standard solution of sodium thiosulphate added in slight excess, and the excess titrated back with weak standard iodine solution, starch being used as an indicator. After correcting for the excess, the quantity of zinc is calculated from the amount of thiosulphate used.

The author finds that the addition of powdered lead and iron to the zinc makes very little difference in the quantity of zinc found by this process, the results being a little lower in the presence of these metals.

The results by this method agree well with those obtained by Fresenius' method, but are generally higher than those obtained by Drewson's and lower than those obtained by Töpfer's method.—A. G. B.

*Estimation of Zinc in its Ores.* D. Coda. *Zeits. Anal. Chem.* **29**, 266—271.

THE usual method of determining zinc in its ores consists in dissolving the ore in aqua regia, evaporating, redissolving in dilute hydrochloric acid, precipitating lead, copper, and cadmium with hydrogen sulphide, oxidising the iron and precipitating with ammonia containing some ammonium carbonate; the iron is thus precipitated and the zinc remaining in solution is estimated with standard sodium sulphide solution. The objection to this method is that the iron precipitate invariably contains zinc, even in the presence of ammonium carbonate and when the precipitate is left in the liquid for 24 hours. Better results are obtained by dissolving the precipitate and reprecipitating it, allowing it to remain in the liquid for 24 hours after reprecipitation; but this is not expeditious.

The new method which the author has devised depends upon the fact that if ammonium sulphate be present the ferric hydrate will combine with it instead of with the zinc oxide. The method is carried out as follows:—

2.5 grms. of the zinc ore are dissolved in 15–20 cc. of aqua regia and evaporated to dryness. 15–20 cc. of sulphuric acid are added and the mass heated till white fumes come off; it is then diluted, precipitated with hydrogen sulphide, and filtered; the hydrogen sulphide is expelled from the filtrate by heat and ammonia is added till the liquid is neutral; 40 cc. of ammonia containing 25 per cent. of ammonium carbonate are then added. After the flask has cooled its contents are made up to 500 cc. and filtered through a dry filter; 100 cc. (0.5 gm. of ore) are taken, diluted to 250 cc. and then titrated with a 2 per cent. solution of sodium sulphide. The details of the titration are as follows: A weighed quantity of pure zinc, as nearly as possible identical with the quantity in the ore, is dissolved in 4 cc. of concentrated hydrochloric acid, made alkaline with 20 cc. of ammonia, and diluted to 250 cc. This solution and that to be analysed are placed side by side and the sodium sulphide run in from a burette till the zinc sulphide is nearly all precipitated; the precipitate is then allowed to settle and a small portion of the clear liquid is placed on a porcelain plate and mixed with a spot of an 8 per cent. solution of sodium nitroprusside; this operation is repeated till a red colour is seen on mixing the drops; the flask must be well shaken after each addition of sodium sulphide.—A. G. B.

*Volumetric Estimation of Zinc according to E. Donath and G. Hattensaur.* L. Blum. *Zeits. Anal. Chem.* **29**, 271–272.

The author objects to Donath and Hattensaur's method for the titration of zinc in an "ammoniacal tartaric acid solution," with potassium ferrocyanide (this Journal, 1890, 554), on the ground that manganese when present will be precipitated under the conditions of the process (compare this Journal, 1887, 226); the results by this method are thus liable to be too high. Mahon (this Journal, 1882, 505) has also pointed out the disturbing influence of manganese when zinc is titrated with potassium ferrocyanide in hydrochloric acid solution.—A. G. B.

*Estimation of Metallic Aluminium in Commercial Aluminium.* G. Klemp. *Zeits. Anal. Chem.* **29**, 388–390.

The author treats a weighed quantity of the aluminium to be tested with a solution of potassium hydrate, and determines the quantity of hydrogen that is evolved either by measuring the volume of the gas or weighing the water produced by burning it. In the first case most of the ordinary gas analysis apparatus may be used, especially Lunge's improved apparatus (Ber. **23**, 440; this Journal, 1890, 547–549). In the second case the apparatus described by R. Fresenius for his zinc-dust estimation is suitable.

In testing the applicability of the method the author has used Fresenius' apparatus. He employs a solution of caustic potash that contains 35 grms. of KOH per 100 cc. The aluminium is disintegrated with a file and the filings weighed in a weighing tube, about 1 gm. of them then shaken into an Erlenmeyer's flask of about 150 cc. capacity, and the tube again weighed. The aluminium is covered with a little water, and some vaselin is added to prevent frothing. The hydrogen evolved by the caustic potash solution is burnt in a Fresenius' apparatus and collected in concentrated sulphuric acid. To prevent a too rapid evolution of hydrogen the caustic solution is added in small quantities. The aluminium takes about three-quarters of an hour to dissolve and, to expel the last portions of hydrogen, the liquid is warmed towards the end of the reaction.

A sample tested by this method gave 98.42 per cent. and 98.37 per cent. of Al; the same sample tested gravimetrically by precipitation with ammonia and ammonium chloride, and deducting the iron precipitated at the same time, gave 98.14 and 98.31 per cent. of Al.—H. S. P.

*Determination of Water in Superphosphates.* J. J. Stoklasa. *Zeits. Anal. Chem.* **29**, 390–397.

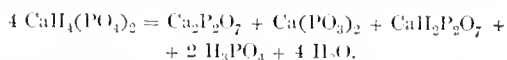
ALL methods heretofore proposed for the estimation of water in superphosphates give varying and incorrect results.

In this paper the author treats of the influence of temperature on mono-calcium orthophosphate. The experiments were made with a chemically pure preparation of the following composition:—

	Per Cent.
CaO .....	22.34
P <sub>2</sub> O <sub>5</sub> .....	56.67
H <sub>2</sub> O .....	21.53
Free phosphoric acid .....	0.014

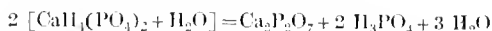
On drying 3.590 grms. of this substance at 100° its weight became constant after 50 hours, and the loss of water was 6.43 per cent., which is equivalent to one molecule of water. At 100° therefore CaH<sub>4</sub>(PO<sub>3</sub>)<sub>2</sub>·H<sub>2</sub>O loses its water of crystallisation after 40 hours' heating. The calcium phosphate so obtained forms opaque and non-hygroscopic crystals of the composition 21.02 per cent. CaO, 60.74 per cent. P<sub>2</sub>O<sub>5</sub>, and 15.00 per cent. H<sub>2</sub>O. It is soluble without decomposition in water in the proportion of 1:200, but dissolves very slowly.

If this substance be heated to 105° it undergoes very slight alteration until after 20 hours' heating, and then only a small proportion decomposes, but by raising the temperature gradually to 200° the decomposition is accelerated, and takes place as follows:—



By treating the substance so obtained with water in the proportions of 1:200, at 15° C., the undecomposed mono-calcium phosphate, the mono-calcium pyrophosphate, and the phosphoric acid are dissolved out, leaving a residue of calcium pyrophosphate and metaphosphate.

Birnbaum (*Zeits. f. angew. Chem.* 1871, 137) discovered the formation of calcium pyrophosphate and free phosphoric acid, but says nothing of finding metaphosphate and mono-calcium pyrophosphate. His equation—



is impossible as calcium pyrophosphate and phosphoric acid when heated produce mono-calcium pyrophosphate.

Drewson (*Zeits. Anal. Chem.* **20**, 54), in writing of the changes in superphosphate at high temperatures, says nothing of the formation of normal pyrophosphate and metaphosphate, but he found even at 100° that a considerable amount of mono-calcium pyrophosphate was formed. Drewson's analyses also show that when heated even to 300° the amount of soluble phosphoric acid in the superphosphate remained unchanged, which for a superphosphate of ordinary composition is certainly a great anomaly. The author repeated the experiment with an ordinary superphosphate heated to 120° and found a considerable decrease of soluble phosphoric acid in the aqueous solution of the superphosphate after heating. But by supposing that Drewson used a superphosphate in which 80 per cent. of the soluble phosphoric acid existed as free H<sub>3</sub>PO<sub>4</sub>, the anomaly would be explained by the reaction between the pyrophosphate, formed by the heating, and the free phosphoric acid according to the equation—



soluble mono-calcium pyrophosphate being formed.

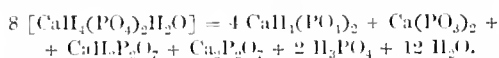
The author has tested the accuracy of this equation by heating 2 grms. of pure calcium pyrophosphate with 1.202 grms. of phosphoric acid in a platinum dish for 10 hours at 200° C. At the end of this time the aqueous solution only contained traces of free phosphoric acid. Before warming with nitric acid the solution contained mono-calcium pyrophosphate and 4 per cent. of phosphoric acid; after heating with nitric acid 56.08 per cent. of phosphoric acid was found, so that in this case first mono-calcium orthophosphate has been formed, and this has on

further heating lost water with formation of mono-calcium phosphate.

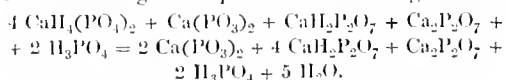
The author also finds that when mono-calcium phosphate is heated at 150° and higher, that not only the temperature but also the length of time it is heated influences the formation of mono-calcium pyrophosphate.

When mono-calcium phosphate was dried at 205° and extracted with water, the solution, after boiling with nitric acid, contained 16·2 per cent. of phosphoric acid, whilst before boiling with acid it only contained 2·3 per cent. The insoluble residue was all calcium metaphosphate. When  $\text{CaH}_2(\text{PO}_3)_2 \cdot \text{H}_2\text{O}$  was heated at 210° it lost 21·5 per cent. of water, and when treated with water left a residue of glassy calcium metaphosphate, insoluble in water, whilst only 0·86 per cent. of phosphoric was found in the aqueous solution. At this temperature, therefore, all the mono-calcium pyrophosphate formed immediately loses a molecule of water and becomes converted into calcium metaphosphate— $\text{CaH}_2\text{P}_2\text{O}_7 = \text{Ca}(\text{PO}_3)_2 + \text{H}_2\text{O}$ .

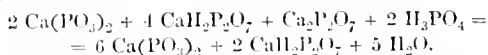
The influence of a temperature of 200° for one hour on mono-calcium phosphate is therefore expressed by the equation—



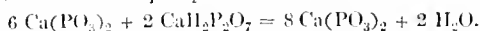
Further heating at 200° produces the following reaction:—



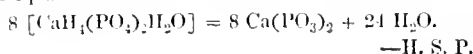
And further—



Finally at 210° the mono-calcium pyrophosphate is all converted into metaphosphate and water—



By heating the original mono-calcium phosphates from the commencement at 210° the whole reaction may be expressed by the equation—



*The Determination of the Hardness of Natural Waters by Soap Solution.* E. L. Neugebauer. Zeits. Anal. Chem. 29, 399—401.

To obviate the discrepancy found frequently in determining the hardness of water containing magnesia salts, between the hardness as shown by Clark's test and that indicated by the gravimetric analysis of the water, the author standardises his soap solutions against a mixture of 8 volumes of a 12° solution of calcium sulphate and 2 volumes of a 12° solution of magnesium sulphate, such a mixture representing an average hard water. He makes his soap solution of such a strength that 12 cc. of it produces a permanent froth in 100 cc. of this 12° mixture. The soap solution must be added slowly, in quantities of 1 cc. at first and towards the end drop by drop, and the operation is ended when a froth remains on the water for five minutes. Only 100 cc. of water are taken, and it is shaken up in a bottle of 200 cc. capacity. By diluting the 12° mixture of calcium and magnesium sulphates with distilled water, solutions can then be made of hardnesses of from 1°—12°, and the quantity of soap solutions corresponding to each degree of hardness can then be found by experiment. The author has had a burette made, graduated in degrees and tenths, corresponding to the various volumes of soap solution required by the solutions of various degrees of hardness from 1° to 12°.

In testing a natural water, the readings of such a burette will be more accurate the more diluted (within certain limits) is the normal volume of the water, and the nearer the relationship of the lime to the magnesia in the water to be tested, expressed in degrees of hardness, approaches that of 80 to 20.—H. S. P.

*The Detection of Natural Mineral Phosphates in Thomas' Phosphate Meal.* L. Blum. Zeits. Anal. Chem. 29, 408—411.

The considerable rise in price of Thomas' slag makes it worth while to adulterate it with certain classes of natural mineral phosphates as, for example, the Mons phosphates, containing 40—45 per cent. calcium phosphate, which is sold at a price equal to 0·11 franc per kilogramme of calcium phosphate. It would only pay to use such natural phosphates for adulteration as were unsuitable for making superphosphates, owing to the quantity of calcium carbonate they contain, as a sufficiently good price is given by superphosphate makers for all natural high-grade phosphates. Thomas' slag, when fresh, contains no carbonic acid, and the author only found 2·28 per cent. carbon dioxide in Thomas' slag that had been exposed in the open air for three years, and he finds that the maximum percentage of carbon dioxide in Thomas' slag examined by him is about 2·5 per cent. Such phosphate as the Mons phosphate (the grey phosphate chalk) contains about 22 per cent. of carbon dioxide.

The determination of the carbon dioxide in a sample of Thomas' meal therefore affords a test of its freedom from or adulteration with natural mineral phosphates, although it does not afford a very definite method of determining the amount of that adulteration.

It is only necessary to determine the loss by calcination to see whether it contains an undue proportion of carbon dioxide.—H. S. P.

*The Chemical Constitution of Tale.* F. W. Clarke and E. A. Schneider. Ber. 23, 1537—1540.

The sample of tale with which the experiments were carried out came from Hunter's Mill, Virginia, and when dried in air gave the following analysis:—

	Per Cent.
$\text{SiO}_2$ .....	62·27
$\text{Al}_2\text{O}_3$ .....	0·15
$\text{Fe}_2\text{O}_3$ .....	0·95
$\text{MgO}$ .....	30·95
$\text{FeO}$ .....	0·85
$\text{MnO}$ .....	Trace.
$\text{H}_2\text{O}$ (105°) .....	0·07
Loss in ignition .....	4·84
	<hr/> 100·08

These figures agree closely with the empirical formula  $\text{H}_2\text{Mg}_3\text{Si}_4\text{O}_{12}$ . Tale is usually held to be an acid metasilicate,  $\text{H}_2\text{Mg}_3(\text{SiO}_3)_4$ . Groth, however, prefers to consider it a basic pyrosilicate,  $\text{Mg}(\text{Si}_2\text{O}_5)_2(\text{MgOH})_2$ , derived from the acid  $\text{H}_2\text{Si}_2\text{O}_5$ .

Olivine behaves like tale to hydrochloric acid gas, being only superficially attacked, but tale is the only one of these minerals that withstands the action of aqueous hydrochloric acid.

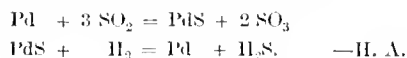
This power of tale for resisting the action of hydrochloric acid is a strong argument against considering it to be a basic pyrosilicate.—F. H. L.

*On the Action of Sulphurous Acid on Metals.* J. Uhl. Ber. 23, 2151—2154.

SULPHUROUS acid acts upon palladium and platinum with evolution of sulphuric anhydride and formation of a sulphide. With gold no sulphide is obtained. Very little or no sulphuric anhydride is evolved with copper, silver, or cadmium, the residue consisting of a mixture of sulphide, sulphate, and other sulphur compounds. Magnesium burns in a current of sulphurous acid, the residue containing sulphate, sulphite, and sulphide of magnesium. Antimony yields a mixture of the trioxide and the trisulphide. Aluminium, zinc, nickel, and cobalt apparently react with sulphurous acid, whilst mercury and bismuth are not acted upon at all.

The author proves the presence of sulphides by the evolution of sulphuretted hydrogen which takes place when hydrogen is passed over the heated residues. Serious doubts are thus thrown on the accuracy of the usual analytical method for estimating copper as  $\text{Cu}_2\text{S}$  by igniting the sulphide in a current of hydrogen, and the author also proves that reduction to metallic copper takes place even after igniting for only a quarter of an hour.

The equations explaining the above reactions are:—



*The Oxidation of Metallic Sulphides by the Electric Current.* E. F. Smith. Ber. 23, 2276—2283.

TWELVE months ago, in a preliminary note (Ber. 22, 1019) the author stated that the sulphur in metallic sulphides was converted into sulphuric acid by the action of an electric current; that, for example, the whole of the sulphur of copper pyrites might be completely oxidised in 10 minutes by this method, and that at the same time the oxides of the heavy metals, such as iron, copper, &c., might be eliminated, so that a precipitate of barium sulphate could be obtained quite white and free from the impurities that usually are so readily carried down with it. Details of the method, and apparatus used, are given in the present paper. The apparatus consists of a nickel crucible, from 1½ to 2 inches high, and from 1½ to 1¾ inch in diameter, which is supported in a ring of stout copper wire attached to a screw fixed to the base of an ordinary filter stand. The arm of the stand carries another screw, in which a strong platinum wire is fastened, which ordinarily constitutes the anode, the crucible being made the cathode. The other parts of the apparatus are, a battery, a "resistance," consisting of a wooden frame on which about 150 metres of iron wire are stretched, for regulating the amount of current, a commutator for reversing the direction of the current, and a Kohlrausch's ammeter, which is inserted between the commutator and the crucible. The "resistance" and the commutator are essential parts of the apparatus, as different sulphides require different amounts of current, and in some cases it is necessary to reverse the current, because a considerable quantity of metal becomes deposited upon the crucible, thus enclosing particles of undecomposed material.

30—40 grms. of caustic potash (not soda) are heated in the crucible until the excess of water is expelled, the weighed quantity of the sample is added to it, the crucible is placed in the copper-wire support, and the platinum wire forming the other electrode is dipped into the melted mass. It is necessary to keep the crucible covered during the oxidation. The current is then closed, and in from 10—20 minutes the sulphur is usually perfectly oxidised. The current is then interrupted, the crucible cooled, and its contents treated with water, which dissolves all but the metallic oxide that has been formed. The liquid is filtered, acidulated, and the sulphuric acid precipitated in the usual way. Should the solution become turbid when acidulated, the oxidation has not been perfect. It should be noticed whether sulphur dioxide is evolved when acid is added, and the residue insoluble in water must always be tested for sulphur. If the potassium hydrate contain sulphur, of course a correction must be made on account of this.

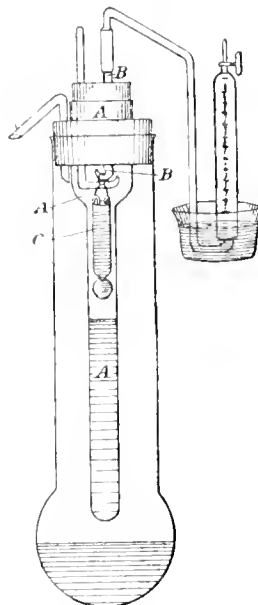
The author then describes the oxidation of various sulphides. Zinc blende, cinnabar, galena, argentite ( $\text{Ag}_2\text{S}$ ), molybdenite ( $\text{MoS}_2$ ), antimony sulphide, orpiment ( $\text{As}_2\text{S}_3$ ), jamesonite ( $\text{Sb}_2\text{S}_3\text{Pb}_2$ ), enargite ( $\text{AsS}_2\text{Cu}_3$ ), stephanite ( $\text{Sb}_2\text{S}_3\text{Ag}_{10}$ ), kobellite ( $\text{BiSb}_2\text{S}_3\text{Pb}_2$ ), tin pyrites ( $\text{SnS}_2\text{Cu}_2\text{Fe}$ ), and fahl ore were all satisfactorily oxidised, the current varying, according to the substance operated on, from 1 to 2 amperes, and the time required for oxidation from 10—20 minutes. Copper glance (redruthite,  $\text{Cu}_2\text{S}$ ) resisted all attempts to completely oxidise the sulphur, repeated reversals of a very large current only resulting in the oxidation of about half the sulphur.

The sulphides of iron act in an interesting manner. Magnetic pyrites (pyrrhotin,  $\text{Fe}_{11}\text{S}_{12}$ ) was easily oxidised.

Similarly, white pyrites (marcasite,  $\text{FeS}_2$ ) suffered complete oxidation. On the other hand, yellow pyrites (mundie,  $\text{FeS}_2$ ), though of the same chemical composition as marcasite, was most difficult to oxidise, one atom of sulphur persistently remaining unoxidised. By mixing an equal weight of cupric oxide with the pyrites, and using a current of 4 amperes, fairly satisfactory results were obtained. The use of cupric oxide was suggested by the facility with which copper pyrites could be oxidised by this method. The different behaviour of marcasite and mundie leads to the assumption that their chemical constitution is dissimilar.—H. S. P.

*An Azotometer for the Estimation of Nitrogen in Ammonium Salts.* W. Hentschel. Ber. 23, 2402—2403.

THE apparatus somewhat resembles V. Meyer's vapour density apparatus. The chief part is a cylindrical tube A, widened at the upper part, where a double-bored cork closes it, and surrounded by an outer tube, in which vapour can be

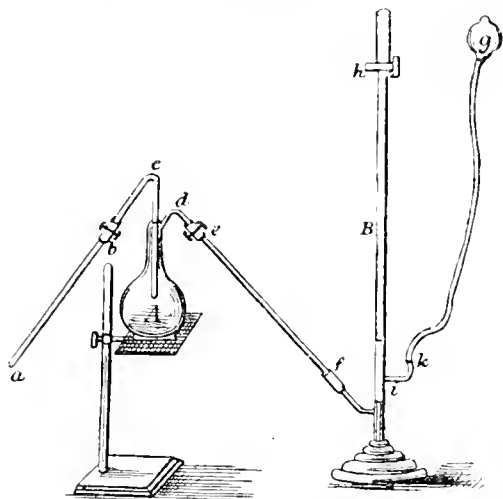


AN AZOTOMETER.

generated for heating the contents of A. The lower part of A holds a solution of sodium hypobromite. Through one hole in the cork passes the tube B, which on the outside is connected with a bent tube leading to a graduated measuring tube, whilst on the inside it is bent into a hook shape. Upon this hook is hung by a platinum wire the bucket C, which contains the solution of the ammonium salt. This "drop cylinder" or bucket is weighted with a sealed bulb containing mercury. Through the second hole in the cork passes a glass rod bent at right angles, by means of which the drop cylinder can be slipped off the hook and thus allowed to fall into the hypobromite solution below. In his experiments the author used methyl alcohol in the outer heating tube; after boiling for 15 minutes a constant temperature was obtained. When the bucket is allowed to drop the solutions of ammonium salt and hypobromite quickly mix, and in a few minutes all the nitrogen is liberated in the form of small bubbles, whilst at the same time a corresponding volume of air and nitrogen enters the measuring tube. The author has demonstrated by experiment that no correction is necessary unless extreme accuracy is aimed at, probably owing to the warm hypobromite solution having a smaller absorbing capacity than the cold solution usually used in other azotometers.—H. S. P.

*Schulze-Tiemann's Method for determining Nitric Acid in Water.* E. Schmidt. Apoth. Zeit. 1890, 287.

ACCORDING to the quantity of nitric acid present, 50—500 cc. of water are evaporated to 20—30 cc., and these are drawn up into flask A. Before connecting with the collecting



THE ESTIMATION OF NITRIC ACID.

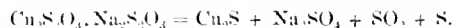
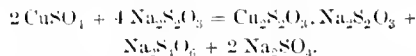
apparatus B, the latter is first filled up to *i* with mercury, and the vessel *g* attached to *h* by a piece of india rubber tubing. Schiff's collecting apparatus may now be filled with caustic soda (20—30 per cent.), which must be boiled previous to the experiment, and introduced warm. Sufficient caustic soda should be used to fill part of *g* when the latter is raised a little over the level of stop-cock *h*; *h* is then closed and *g* fixed at the same level as the stop-cock. The pinch-cock *b* being closed and *e* opened, the water in A is brought to a boil. The air bubbles which now rise in B are allowed to escape by opening *h* and raising *g*. When no more air collects in B, *c* is closed and *b* opened. The steam issuing from *a* is passed into a mixture of equal parts of a saturated and freshly prepared solution of ferrous chloride and fuming hydrochloric acid, in order to expel any air. When only 5 cc. of water are left in the flask, *b* also is closed and the flame at once removed.

By opening *b* after some time, about 10 cc. of the ferrous chloride mixture are allowed to enter the flask. *b* is again closed and heat applied until pressure is noticeable by the rubber tubing at *e* becoming inflated. The stop-cock *e* is then opened, the contents of the flask evaporated to dryness, and *e* is again closed. As soon as a negative pressure is observed in A a further 5 cc. of the ferrous chloride mixture are introduced and the operations carried out as above. On now evaporating to dryness all nitric oxide will have collected in B, and the volume may be read off after adjusting the level of the liquid in *g* to that in B. (See also this Journal, 1890, 896—897.)—A. R.

*The Chemical Society of Aachen.* Meeting of June 12th, 1890. Chem. Zeit. 14, 855—856.

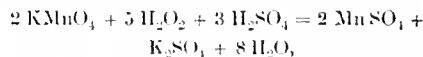
CLASSEN has been able to detect traces of lead in bismuth preparations which appeared to be free from lead by a spectroscopic examination. The substance is converted into nitrate and electrolysed in the presence of an excess of nitric acid; lead peroxide and bismuth peroxide separate out, but the greater portion of the latter dissolves. The peroxide is converted into nitrate and saturated with pure soda solution, potassium bichromate is added to the pure solution, which precipitates bismuth chromate, whilst the lead remains in solution and may be precipitated from the filtrate by acidifying with acetic acid.

Vortman described a new volumetric method for estimating copper. If sodium thiosulphate be added to a solution of a copper salt, a double salt,  $\text{Cu}_2\text{S}_2\text{O}_3 \cdot \text{Na}_2\text{S}_2\text{O}_4$  is formed which is decomposed on boiling.



If as much sulphuric acid be previously added as is sufficient to just decompose the added thiosulphate, then at the conclusion of the reaction there remains an amount of free sulphuric acid corresponding to the amount of copper taken and it may be determined according to some acidimetric process.

The titration is carried out as follows; the copper solution (preferably sulphate) must first be neutralised, this may be done by adding an excess of hydrogen peroxide and then potassium permanganate until a brown precipitate appears, when the solution will be just neutral, the following reaction having taken place:—



the reaction proceeds best at 70°—80° C. An excess of decinormal sodium thiosulphate is added to the neutralised solution and as much decinormal sulphuric acid as is necessary to just decompose the thiosulphate. The solution is boiled until completely decomposed, an excess of potassium iodide and iodate added to the solution, titrated back with decinormal thiosulphate; 4 l is equivalent to 2 Cu. The results are very satisfactory.

*Ferrous salts* may be estimated by oxidation with iodine by adding sodium acetate and acetic acid and then a measured excess of iodine solution, and after standing 15 minutes, titrating the excess of iodine.

*Nitric acid* may be estimated by electrolysis in the presence of copper sulphate, when it is completely converted into ammonia. The titrate is dissolved in a platinum dish, sulphuric acid and copper sulphate added and the solution electrolysed in the usual way; it is then distilled with soda and the ammonia which is evolved estimated in the usual manner. If the nitric acid is already in combination with a metal which is deposited on electrolysis, the copper may be omitted.

*Aluminium* in solutions containing phosphoric acid, such as occur when iron is electrolytically deposited from a solution of ferro-aluminium, may be easily separated from such metals as manganese, &c. in the following way. The solution is neutralised with soda and an excess of sodium acetate, phosphate and acetic acid added whereby all alumina is precipitated as phosphate; this is washed with dilute ammonium nitrate, ignited and weighed as  $\text{AlPO}_4$ .

*Cobalt* in ammoniacal solution is oxidised by alkaline hypochlorites whilst nickel is not. To take advantage of this for the separation of cobalt and nickel, an excess of ammonia is added to the solution and a slight excess of alkaline hypochlorite and the whole warmed until the excess of ammonia is destroyed. Potash is added to this when  $\text{Co}_2\text{H}_6\text{O}_6$  and  $\text{NiH}_2\text{O}_2$  are precipitated. The precipitate is washed and transferred to a beaker; potassium iodide and hydrochloric acid added;  $\text{Co}_2$  liberates  $\text{I}_2$  which is titrated with thiosulphate.—A. L. S.

*Estimation of the Zinc as Carbonate and Silicate in Calamine containing Lead.* W. Minor. Chem. Zeit. 14, 1003.

THIS is a modification of the author's method for the determination of the zinc existing as carbonate and silicate in calamine ores containing lead (Chem. Zeit. 13, 1670; this Journal, 1890, 417). Two grms. of calamine are boiled



with 50 to 60 cc. of dilute caustic soda solution for about 15 minutes, the insoluble matter is allowed to settle, the liquid filtered, and the residue again boiled with soda as before. The second liquid is also filtered, and the residue washed on the filter with hot dilute soda. The filtrates are added together, acidulated with sulphuric acid to remove the lead, made up to a known bulk, and, after standing several hours, half of the liquid is filtered. This solution is then made alkaline with ammonia and the zinc estimated by titration with sodium sulphide. Since the greater part of the zinc existing as sulphide in calamine ores is lost during the smelting, a method for determining that combined with carbonic and silicic acids only is of great importance. It is not necessary after adding the ammonia to filter off any alumina that might be precipitated.—A. W.

*A New Method for the Estimation of Nitrates and Nitrites in Water.* R. Ormandy and J. B. Cohen. J. Chem. Soc. (Trans.) 1890, 811—815.

THE process resembles that of Gladstone and Tribe. The zinc-copper couple is replaced by thin sheet aluminium coated with a film of metallic mercury. This is effected by shaking the foil with a solution of mercuric chloride (1 vol. of a saturated solution with 1 vol. of water). Metallic mercury adheres to the surface of the aluminium and the couple, after being washed a few times with pure water, may be introduced into the water to be analysed. The aluminium-mercury couple acts more rapidly than the zinc and copper and gives equally good results. Nitrates in small quantities such as occur in natural waters are entirely reduced by the couple in about an hour. The couple is left in contact with the measured quantity of water in the cold until the aluminium is converted into oxide. Water and precipitate are then transferred to the retort for distillation. The nitrate is converted into ammonia, the amount of which is estimated by Nessler's reagent in the usual way.

Before preparing the couple the aluminium foil must be heated in a current of hydrogen or in a Bunsen flame to remove any grease that may be present.

It is well known that albuminoid substances undergoing decomposition split up into simpler organic nitrogen compounds before free ammonia makes its appearance. The authors determined whether the zinc-copper or aluminium-mercury couple effected any change by hastening decomposition when in contact with the partially decomposed albumin. The following is the summary of the results:—

1. Neither couple affects the amount of free ammonia produced during the decomposition of the albumin, that is to say, the amount of free ammonia given off each day by the decomposing albumin was the same with or without the addition of the couple.

2. The addition of caustic soda to the albumin solution and to the solution with the zinc-copper couple set free an additional quantity of ammonia, which was the same in the two cases.

3. In the case of the albumin solution, acted upon by the aluminium-mercury couple, the amount of free ammonia produced by the addition of caustic soda was largely in excess of that in the other two solutions. It is evident that the aluminium couple acts upon the products of the decomposition of the albumin, which are decomposed only on the addition of caustic soda.—J. B. C.

*Determination of Manganese and Zinc as Pyrophosphates.* R. C. Boyd. School of Mines Quarterly, 11, 355—358.

In the precipitation of manganese as double phosphate by microcosmic salt, it has hitherto been considered necessary to work with absolutely no excess of ammonia. Slight excess of ammonia is, however, shown by the author not to be detrimental. Furthermore it has been deemed advisable to filter ice-cold. This, too, is shown to be unnecessary, as also is washing with a 10 per cent. solution of ammonium nitrate. Hence precipitation and filtration are both performed hot, and only about 50 cc. of washings become

necessary to remove the sodium salt and excess of alkaline phosphate. After ignition with the usual precautions, the precipitate becomes manganese pyrophosphate,  $Mn_2P_2O_7$ .

With regard to determination of zinc by this method, the precipitation is made as follows:—An excess of microcosmic salt is added, and the whole is heated to boiling, and then a slight excess of ammonia added. The boiling is continued until no smell of ammonia is noticeable. The precipitate is then filtered and washed by decantation, as much of the precipitate as possible being kept in the beaker. When the washings give no reaction for phosphates, the precipitate is dissolved in the beaker by the aid of a little nitric acid, and the solution is poured through the filter into a weighed platinum dish, and the zinc compound rinsed into the same with a little distilled water. After evaporating and carefully igniting, the dish and contents are weighed. By this means the reducing action of burning filter paper upon the precipitate is avoided. While cooling, the dish should be covered, as sometimes the residue "snaps," and portions may be projected from the dish unless this be covered. Analyses show very good results in the case of both manganese and zinc.—T. L. B.

ORGANIC CHEMISTRY.—QUALITATIVE.

*Adulteration of Commercial Olein with Linoleic Acid.* Grandval and Valser. Bull. Soc. d'Encour. Ind. Nat. 1890, 404—407.

SO-CALLED "olein," in reality crude oleic acid, is a by-product of candle-making, being separated from the more valuable stearic acid by pressing at carefully regulated temperatures, and is chiefly used for soap-making and as a lubricant for wool-spinning. For the latter purpose, some manufacturers prefer it to olive oil, because it is cheaper and easier to remove from the fibre, sodium carbonate serving as a detergent instead of soap, which is necessary when oil is used.

Recently goods for which oleic acid has been used have been damaged by the appearance on them of yellow stripes, which persist, even after washing and bleaching, and are fatal to the successful dyeing of delicate tints. The authors attribute this state of things to the presence of linoleic acid, and account for it in the following way:—

The growing trade in margarine has led to the practice of pressing fresh fats of animal origin for the sake of their olein, which is used in its manufacture. The process, whether carried out by candle-makers themselves or pursued as a separate industry, yields a mass of stearin so hard that it is troublesome to deal with after saponification for the preparation of stearic acid. Accordingly the requisite consistency for successful pressing is obtained by adding the fatty acids resulting from the partial saponification of linseed oil, which is chosen both because of the fluidity of the greater part of its fatty acids, and because it contains a certain proportion of solid fatty acids which augments the yield obtained. The "olein" got by pressing such a mixture of course contains drying fatty acids, of which linoleic acid is a type. When used for greasing wool, no ill-effects are noticed if the material be woven and washed soon afterwards, but if stored some weeks before weaving exposed to light and air, discolouration takes place, and is only hidden by dyeing with dark colours. Besides this defect, a good deal of waste is produced, and the fibre is harsh, sticky, and rather more troublesome to get free from grease; no other inconvenience, however, appears to result.

The detection of such adulterated oleic acid is tolerably simple, and is best effected by direct comparison with genuine oleic acid. The sophisticated variety is of paler tint than the genuine, and has a specific gravity of 0.912—0.919 at 15° C., instead of 0.905. It often contains a good deal of solid matter, and the specific gravity is best taken at a somewhat higher temperature, and corrected by use of the constant 0.00064 for each degree centigrade. On heating repeatedly to about 50° C. and cooling, the spurious grease becomes more and more buttery, while the consistency of the genuine is unaffected. When 50 grms. are shaken with

450 cc. of alcohol at 85 per cent., a shining deposit is formed; oleic acid thus treated dissolves completely. A residue is also given by any mineral rosin or fatty oil that may be present, but is easily distinguished from it by its different appearance. Definite proof of the nature of the deposit may be had by collecting it on a filter, washing with alcohol and drying, when it will be found to fuse at about  $47^{\circ}\text{C}$ ., and to dissolve easily in boiling alcohol, being deposited therefrom on cooling. It saponifies readily and completely, yielding a soap whose solutions gelatinise in the cold even when very dilute.

The recognition of drying fatty acids may also be effected by the elaidin reaction. Other tests are based on the greater tendency to dry when exposed on a clean leaden surface and the yellow colour given by shaking with an equal volume of caustic soda, pure oleic acid giving only a grey tint.

Judged by such means, adulterated samples of "olein" can be condemned and damage avoided.—B. B.

*A New Method for Detecting Colouring Matters added Fraudulently to Wine.* J. Weirich. Monit. Scient. 1890, 4, 689—699.

A SMALL quantity of the wine to be tested is brushed on a piece of paper which should not be absorbent, allowed to dry, exposed to the light, and examined from time to time, the tint communicated to the paper by a natural wine is of totally different character from that due to an artificially coloured wine. The natural colouring matters of wines and other vegetable colouring matters change each in a definite way on the paper; mineral colouring matters do not alter. Experiments should be made with natural and artificially coloured wine of different vineyards, when the working of the test will be soon understood.—A. L. S.

*Reaction of some Phenols in the Presence of Chloroform and Alkali.* Monit. Scient. 1890, 4, 710—713.

THE phenols give with chloroform and alkali a characteristic colour reaction, and this is recommended as a general reaction for identifying these compounds. To perform the test, the phenol is dissolved in chloroform, a small piece of potash added and the solution boiled. The characteristic colouration is quickly developed. Reducing and oxidising agents affect the reaction, alcohol sometimes accelerates and sometimes hinders it, ether nearly always impedes it, with aqueous solution the reaction is less sharp; in this case it is best to shake up with chloroform and proceed with the decanted chloroform as usual, or to extract with ether, boil the ether off and dissolve the residue in chloroform.

*Phenol.*—A pale red colouration is first produced which becomes brown, then yellow, and finally colourless. The reaction is sensitive with 1 part to 60,000 chloroform.

*o-Cresol.*—Lilac colouration with an orange shade. Sensitive with 1 part in 80,000.

*m-Cresol.*—As *o*-cresol but a more marked orange shade. Same sensitiveness.

*Thymol.*—Fine red colouration verging on purple with a violet tinge, which is accentuated by the addition of a little alcohol or ether, and soon disappears. Sensitive to 1 part in 20,000.

*Guaiacol.*—Cherry red colouration with a blue tinge, changing to violet-blue. Sensitive to 1 part in 100,000.

*Resorcinol.*—On warming gently, a cherry red deposit on the potash and a brownish yellow colouration of the chloroform; the chloroform ultimately takes up the colour, which is fairly stable. Sensitive to 1 part in 500,000.

*Quinol.*—Bluish-green colouration soon disappearing. Sensitive to 1 in 20,000.

*Pyrogallol.*—A reddish-brown colouration in the cold, which on warming takes on a green tinge, and on boiling soon disappears. Sensitive to 1 in 300,000.

*$\beta$ -Naphthol.*—Prussian blue colouration, changing to green and then brown. Sensitive to 1 in 80,000.

*Salol.*—Red colouration like phenol.

*Betol.*—Bluish-green colouration like  $\beta$ -naphthol.

With these last two it is best to boil with a little potash before adding the chloroform in order to saponify the ether. The colours then obtained are very characteristic.

When testing for phenols in complex liquids such as blood, the contents of a stomach, &c., it is best to acidulate and distil in a current of steam. The distillate may be examined as above.—A. L. S.

*On the Detection of Fish Oil in Linseed Oil.* A. Ruffin. Chem. Zeit. Rep. 14, 205.

THE original statement made in 1839 by Fauré, that linseed oil containing fish oil is coloured black by chlorine requires modification. By passing chlorine into pure linseed oil and excluding the air, a green colour appears after a few minutes. On now leaving in an open vessel the colour gradually turns brown and in 24 hours perfectly black. In a closed vessel this change takes place less rapidly. If the current of chlorine be stopped when the oil becomes green, the oil turns black much faster. When the action of chlorine is allowed to take place in an open vessel, after a few minutes a momentary green colouration is observed, rapidly changing to black. With mixtures of linseed and fish oil in the most varying proportions a green colouration is never observed, but previous to the oil turning black a characteristic brown colour appears. In examining linseed oil it is therefore necessary to take into account whether the oil is old or fresh, and whether the sample has been taken from closed or open, full or partly empty vessels.—A. R.

*On Orexine.* A. Donner. Pharm. Zeit. 1890, 418.

OREXINE, the hydrochloride of phenyldihydroquinazoline, is used as a stomachic (see this Journal, 1890, 887). Its formula is  $\text{C}_{11}\text{H}_{12}\text{N}_2\text{HCl} + 2\text{H}_2\text{O}$ , and it crystallises in needles which melt at  $80^{\circ}$ , the anhydrous substance melting at  $221^{\circ}$ . The water of crystallisation is given off by mere exposure to the air. It is readily soluble in water and alcohol. In the aqueous solution (1:20) mercuric chloride produces a white, potassium bichromate a yellow precipitate. Potassium permanganate is decolourised in the cold already. A characteristic reaction is to heat orexine with zinc powder over the direct flame, when a strong smell of isonitrile becomes apparent. On now treating the mixture with hydrochloric acid and adding chloride of lime to the filtrate the solution is coloured blue.—A. R.

ORGANIC CHEMISTRY.—QUANTITATIVE.

*Combustions under High Pressure.* W. Hempel. Ber. 23, 1455—1460.

It is well known that during the combustion of sulphur considerable quantities of sulphuric anhydride are produced, and that during the burning of hydrogen, coal-gas, or coal, traces of nitrous acid are formed. The following experiments shown that under pressure larger quantities of these substances are produced.

1. Sulphur burnt in a mixture of air and oxygen in excess.

No. of Experiment.	Pressure.	Sulphur Burnt.	SO <sub>2</sub>	SO <sub>3</sub>
			Per Cent.	Per Cent.
1	Atmospheric....	*5375	98*0	2*0*
2	31*0 atmospheres.	*238	66*8	32*2
3	41*5     "     "	*268	34*7	65*3*
4	72*5     "     "	*336	54*5	45*5*
5	75*5     "     "	*328	59*8*	26*4

\* Estimated.

In the last experiment a considerable amount of the sulphuric anhydride volatilised during the evaporation of the sulphurous oxide.

II. Varying mixtures of air, oxygen, and the mixture of hydrogen and oxygen formed by the electrolytic decomposition of water, compressed in a steel vessel holding about 28 cc. filled with chips of platinum moistened with concentrated potassium hydrate. The resulting nitric acid estimated as nitric oxide by Schulz's method.

No. of Experiment.	Pressure in Atmospheres.			cc. of NO.
	Air.	Oxygen.	H <sub>2</sub> +O.	
6	2.5	12.8	38.5	2.8
7	1.0	18.3	43.5	4.8
8	1.0	27.0	57.0	6.4
9	1.0	54.0	114.0	8.0
10	1.0	55.0	173.0	9.0
11	1.0	55.0	211.5	10.0

III. Combustion of nitrogen in presence of carbon and a large excess of oxygen. Potassium hydrate was employed as in series II. for the absorption of the nitric acid, and for the carbon a bituminous coal containing 2.8 per cent. nitrogen was used. The vessel, a steel autoclave, was filled with air at the ordinary pressure, the oxygen being afterwards pumped in till the necessary pressure was attained.

No. of Experiment.	Weight of Coal Burnt.	Pressure in Atmospheres.		cc. of NO.
		Air.	Oxygen.	
	Grms.			
12	.622	1.0	10.5	3.0
13	.576	1.0	33.8	3.0
14	.599	1.0	60.0	8.5
15	.793	1.0	73.0	9.0
16	.616	1.0	81.0	7.6
17	.622	24.0	92.0	14.0
18	.595	13.0	92.0	20.5
19	.711	92.0	217.5	5.0
20	.683	1.0	242.0	8.0
21	.632	12.5	88.0	25.0

In experiment 21 the .632 gm. of carbon consisted of a mixture of 25 per cent. of the coal previously employed and 75 per cent. of coke from chemically pure sugar.

Experiments 12—20 leave it doubtful whether the nitric acid was formed from the nitrogen in the coal, but No. 21 shows this not to have been the case, the sugar-coke containing no nitrogen.

The experiments also show that the proportion the different gases bear to one another and to the amount of coal burnt exerts a great influence on the amount of nitric acid produced.—F. H. L.

#### *The Examination and Valuation of Spirits.* W. Fresenius. Zeits. Anal. Chem. 29, 283—317.

THE author has analysed various samples of brandy, rum, arrac, and cherry-brandy, all of which were of ascertained genuineness. The results are given in the subjoined table.

The brandy samples 2 and 3 are very fine old brands, having been some 15 years in wood and originally containing some 61 per cent. of alcohol by volume. Samples 1, 4, and 5 are somewhat younger and originally weaker; they are,

however, of good quality. Samples 6 and 7 are much younger and either originally contained less alcohol or were reduced with water to be more quickly saleable. Sample 8 is slightly coloured with caramel.

One of the rum and of the arrac samples had one-sixth pure spirits of wine added to it, as the author believes this to be customary at times in the trade. The specific gravity was determined by the pycnometer and the alcohol found by Fehner's tables. In the rum and brandy samples the alcohol was also determined by distillation. The extract was determined by drying 50 cc. for 2½ hours in a water-oven, and from this the mineral matter by ignition. The free acid was determined by titrating 50 cc. with one-tenth normal alkali.

To determine Fehling-reducing substances the sample was so concentrated or diluted as to contain 1 per cent. of extract; the reducing power was then determined by Allihn's method, directly in one half, and after inversion in the other half; the difference before and after inversion was regarded as cane sugar. The results were calculated into invert sugar by Meissl's tables. The author remarks that if the reduction value is over 0.5 per cent. and increases considerably after inversion, the sample may be regarded as sugared. He allows a reduction value of about 0.2 per cent. for substances that are not sugar, and is of opinion that unsweetened brandies can contain more than 0.5 per cent. extract.

The determinations of fusel oil were made by Rose's method, the improvements of Stutzer and Reitnair being adopted (Zeits. Anal. Chem. 26, 376, 377; this Journal, 1886, 498).

The author has made many determinations of fusel oil, besides those quoted under the head "Chloroform-increase" in the table, both on samples of brandy, &c., and of commercial and pure alcohol. For Rose's method a large shaking apparatus was employed and 250 cc. of the alcohol and 50 cc. of chloroform were used. The spirit was distilled over potash solution and the distillate brought to 30 per cent. by volume (sp. gr. 0.9656), though it was found that a slight variation in the strength made a very little difference in the chloroform-increase. When different samples of chloroform were used the results on the same spirit differed slightly, so that for comparison the same chloroform was used for all the samples of spirit.

Several determinations were also made with Traube's capillarmeter and stalagmometer, but the reading of these instruments requires a great deal of practice.

On a previous occasion (Zeits. Anal. Chem. 26, 381) the author has asserted that even undoubtedly pure brandies give a higher chloroform-increase than pure alcohol and would consequently be reported as containing fusel oil; this he attributed to the probable presence of some higher alcohols which act similarly to fusel oil, or to added flavouring matters. This conclusion is not entirely confirmed by the present research, for it is seen by the figures in Table II. in the original paper that some of the pure brandies give the same chloroform-increase as Kahlbaum's pure alcohol. On the other hand some artificial brandies which almost certainly contain fusel oil show very little, and sometimes no, chloroform-increase; but it is quite possible that this may be due to the presence of some substance having an effect on the chloroform counter to that of the fusel oil. The author concludes that the results of testing for fusel oil are at present very uncertain and that the subject requires further investigation.

Testing for caramel was carried out according to Anstoth's directions (this Journal, 1885, 361). An expert, judging the brandies from their colour, pronounced 1, 6, and 7 as free from caramel, 2 and 3 as coloured by age, 4 and 5 as containing a little caramel, and 8 as undoubtedly coloured.

The author then proceeds to discuss the characteristics of pure brandy, and how such should be defined. The German Pharmacopœia pronounces brandy to be a product of the distillation of wine; a clear yellow fluid of peculiar spirituous smell and taste; it must be free from fusel oil and not sour; its specific gravity must be 0.920—0.924, and its alcohol contents 46—50 per cent. by weight. This definition, however, will exclude some of the finest and most undoubtedly genuine

brandies as containing too little alcohol (compare Elser, this Journal, 1888, 637).

Undoubtedly the best method of discerning a genuine spirit is by its smell and taste when such is practised by an expert. But in view of Custom house disputes it frequently falls to the lot of the chemist to pronounce upon the genuineness of a spirit and it is to be regretted that his means of doing so are so few and uncertain.—A. G. B.

(For Table see next page.)

*On some Essences used for Strengthening Spirituous Liquors and Ripening Artificial Brandies.* E. Poleske. Proceedings of the Imperial Board of Health (Russia), 1890, 294.

See under XVII., page 959.

*Detection and Determination of Starch.* A. Leclere. J. Pharm. Chim. 1890, 641.

A CONCENTRATED solution of zinc chloride (sp. gr. 1.43—1.45) will dissolve starch and sugar, but not cellulose, fat, or nitrogenous substances. In this manner the starch may be separated from the fodder. 2 grms. in the case of cereals, or 5 grms. in the case of straw, hay, &c., are well powdered, and thoroughly moistened with 10 cc. of water. 180 cc. of the neutral zinc chloride solution being added, the flask is heated in a salt-bath at 108° until the cereal has dissolved; or, in the case of straw, &c. heating should at all events be continued for 1½ hours. After cooling, the solution is made up to 250 cc. with some zinc chloride solution. Where fibrous substances are under examination, the solution is made up to 253° to allow for the extra volume of the former. The solution is now filtered, which takes a considerable time, and 25 cc. of the filtrate mixed with 2 cc. of hydrochloric acid and 75 cc. of alcohol of 90 per cent. Starch and dextrin are thus precipitated, sugar remaining in solution. It is best to decant, after 24 hours' standing, through a tared filter. For washing the precipitate, alcohol of 90 per cent. is used to 1,000 cc., of which 5 cc. hydrochloric acid have been added. Finally alcohol only is used. The starch occasionally has an admixture of mineral substances the quantity of which may be determined by igniting; 25 cc. of the zinc solution gives a precipitate in the case of maize, weighing about 140 mgrms., of which 5 mgrms. are inorganic.

The starch obtained from cereals by the above means contains traces of nitrogenous substances which must be taken into account after a nitrogen determination. Thus in the above example with maize, the precipitate will contain 1.5—2.0 mgrms. of nitrogenous substances. With oats, the proportion of the latter is somewhat greater, with straw it may be neglected.—A. B.

*Determination of the Value of Dye-wood Extracts.* L. Schreiner. Chem. Zeit. 14, 961—962.

In judging of the value of dye-wood extracts, the following chief points should be observed:—(1.) The percentage of dyestuff. (2.) Addition of foreign dyes or substances containing tannic acids. (3.) Addition of weighting substances. (4.) Fermentation and reaction.

Like tannic acids, several dyes, such as hæmatoxylin and hæmatestin are entirely absorbed from their solution by powdered skin. This is a good means of separating the dyes from the other substances in the extract, more especially from adulterations such as sugar, molasses, and salts. The only disadvantage of this method is that tannic acid and the dyes cannot be determined separately. The author considers that this is not of great importance, seeing that where such dye-wood extracts containing tannin are used, the tannin cannot generally be regarded as of less value than the dye itself.

For an analysis 50 grms. of ground dye-wood are thoroughly extracted, the solution is diluted to 1 litre, filtered, and a portion of it allowed to percolate through skin powder, which must be pure and of a woolly nature. The filtrate should be perfectly colourless and remain so after

adding ammonia. 100 cc. of this filtrate and 100 cc. of the original extract are now evaporated separately, dried at 100° in the air-bath, and the difference gives the percentage of the dye.

When the extract itself is under examination, a solution which contains about 10 grms. dry substance per litre is best prepared. This is heated on the water-bath to 50° C., filtered if necessary with addition of kaolin and the clear solution examined as above. Moreover 1—2 grms. of the extract are dried in a platinum dish at 100° for determining the quantity of water, and then ignited in order to obtain the weight of the mineral constituents.

The difference between the dry substance and the sum of the dyes and non-dyes gives the amount of insoluble substances. The quantity of inorganic constituents is subtracted from the non-dyes in the subjoined table, the figures giving the percentage:—

FRESH LOGWOOD.

—	I.	II.	III.	IV. (Fresh.)	V. after Fermenting.
Dyes .....	12.06	10.56	8.16	9.70	9.23
Non-dyes .....	2.22	1.72	1.04	2.05	1.10
Water .....	10.86	12.80	10.8	10.02	16.00

LIQUID EXTRACT, 30° B.

—	I.	II.	III.	IV. (Solid.)	V.
Dyes .....	52.52	41.39	33.52	79.77	54.36
Non-dyes .....	6.06	11.91	14.59	9.08	18.14
Portion insoluble at 50° .....	0.45	2.87	6.99	1.05	9.20
Ash .....	0.14	1.81	3.47	0.22	6.04
Water .....	40.83	42.04	41.63	9.88	12.26

The advantage of an analysis is apparent when it is added that samples I.—III. had been valued as equal.

The quantity of non-dyes is of importance in wool-dyeing chiefly, as on this depends the degree of fermentation. The more thorough the latter the more perfectly is the dye contained in the wood eliminated from the latter. With regard to the analyses of the extracts, Nos. I. and IV. are normal extracts of good logwood. No. II. contains about 20 per cent., No. III. about 10 per cent. of a syrup containing considerable quantities of lime, and in No. V. molasses and sodium sulphate have been added. The reaction of a logwood extract may be judged by the colour of its solution. When neutral it is a deep red, an alkaline solution is bluish-red, and an acid one if not fermented light yellow, otherwise orange-yellow. Extracts to which tannic acids have been added always show an acid reaction, and in distinction to the naturally acid extracts, the colour does not at once turn into the neutral red on diluting with lime water or by shaking and warming with a little calcium carbonate.

Another characteristic reaction is to mix a solution of the extract (5° B.) with some stannic chloride, avoiding excess of the latter. A dark brown precipitate is formed if the extract has fermented, if not it is coloured a light violet, while in presence of tannic acids a dirty yellow precipitate is obtained.

Another good test for tannic acids is to add to a solution of 5 grms. of dry extract in 1 litre of water one-third its volume of yellow ammonium sulphide. If the extract be pure, a slight dark flocculent precipitate will be deposited, if tannic acids be present the solution will be decolourised and a heavy light grey substance precipitated.—A. R.

## THE EXAMINATION OF SPIRITS.

THE EXAMINATION OF SPIRITS.

CARAMEL REACTION  
after Author (this Journal, 1885, 361).

SAMPLE.	Specific Gravity at 15°.	In 100 Grms.	Pehling-reducing Substances.	(Chloroform-Increase (Pure Alcohol is 5.3 cc.).	Caramel Reaction after Author (this Journal, 1885, 361).	The Paraldehyde Precipitate dissolved in Water and Mixed with Phenylhydrazine-Hydrochloride.				
		Alcohol.	Extract.	Mineral Matter.	Free Acid expressed as Acetic.	Precipitate.	Liquor.			
		From Specific Gravity.	By Distillation.	By Distillation.	From Specific Gravity.	From Gravity.	By Distillation.	From Gravity.	By Distillation.	
		Per Cent. by Volume.	Grm.	Grm.	Grm.	Grm.	Grm.	Grm.	Grm.	
<b>COGNAC.</b>										
1. ....										
2. ....										
3. ....										
4. ....										
5. ....										
6. ....										
7. ....										
8. ....										
<b>R.V.</b>										
1. Jamaica. ....										
2. Jamaica. ....										
3. Jamaica. ....										
4. Jamaica, with $\frac{1}{2}$ added spirit. ....										
<b>ARRAC.</b>										
1. Batavia. ....										
2. Batavia, with $\frac{1}{2}$ added spirit. ....										
3. Soerabaya. ....										
<b>CHERRY-BRANDY.</b>										
1. 1887 brand. ....										
2. 1887 brand. ....										
3. 1887 brand, from black cherries. ....										
4. 1887 brand, from black cherries. ....										
5. 1885 and 1886 mixed. ....										
6. From black cherries. ....										
7. From unimproved cherries. ....										
8. 1886 brand, partly from red cherries. ....										
9. 1885 brand, from improved cherries. ....										
10. 1887 brand. ....										
11. 1883 brand, from black cherries. ....										
12. Faint, 1887. ....										

*Determination of the Value of Strychnos Seeds.* B. Holst and H. Beckurts. *Arch. Pharm.* 1890, 330.

For determining the quantity of alkaloids contained in strychnos seeds, 10 grms. are powdered and extracted with 75 parts of chloroform and 25 parts of alcohol containing a little ammonia. After distilling off the chloroform and alcohol from the extract the residue is dissolved in 10 cc. of a 5 per cent. solution of ammonia and 5 cc. alcohol, and shaken in a separating-funnel with chloroform 20, 10, and 10 cc. respectively. The latter is then distilled off, the residue heated on the water-bath until all ammonia has disappeared, then gently heated with 15 cc. of decinormal hydrochloric acid for 5 minutes, filtered, and washed. The excess of acid is now titrated back with centinormal caustic soda, using cochineal as indicator. By subtracting the cc. of centinormal caustic soda used from 150, the difference gives the number of cc. of centinormal hydrochloric acid necessary to neutralise the alkaloids. 1 cc. of centinormal hydrochloric acid corresponds to 0.0364 grm. of alkaloid, on the assumption that equal parts of strychnine and brucine are present.—A. R.

*On the Estimation of Raffinose and Invert Sugar in the Products of the German Beet Sugar Manufacturers.* *Chem. Zeit.* 14, 854—855.

THE Society of German Beet Sugar Manufacturers appointed a committee of experts to inquire into the methods of determining invert sugar and raffinose in beet-sugar syrups. The committee recommend that all analyses should be made in one definite agreed-upon manner, and submit the following details:—

*Polarisation.*—(a.) Whether the sample is warm or not is immaterial. (b.) It is not easy to dispense with the employment of lead acetate when examining the first products with the Ventzke-Soleil-Scheibler instrument. The lead acetate should be prepared according to the German Pharmacopœia (2nd edition, p. 170); sp. gr. 1.235—1.240. Colloidal alumina, prepared according to Vivien's directions, should also be used in all cases. (c.) In clarifying the dark after-products, the use of other substances besides lead acetate and alumina is to be avoided if possible. The charcoal used should be the best bone charcoal which has been washed with hydrochloric acid and subsequently ignited. (d.) Half the standard weight of molasses having been clarified with lead acetate and alumina. The solution is made up to 100, and the optical activity read in a 200 mm. tube. The result of the analysis (disregarding the volume of the precipitate) is multiplied by 2.

*Determination of the Invert Sugar.*—(a.) The syrup is first of all tested qualitatively with Fehling's solution, the procedure being exactly the same as if a quantitative analysis were being made. If there be much reduction, a quantitative analysis should be made. (b.) If a quantitative analysis is required, the sample may be clarified with lead acetate if necessary. The determination should be carried out in detail, as recommended by the Invert Sugar Committee in 1885 (*Zeit. f. Zuckerind.* 1886, 36, 6 and 7). This committee recommended that particular attention be paid to the purity of the reagents used in preparing the Fehling's solution. A good solution should give not less than 16 and not more than 22 mgrms. of copper with pure cane sugar. The use of Soldani's solution is not to be recommended. If less than 50 mgrms. of copper is obtained, the invert sugar is to be considered as *not estimable*. Molasses containing not more than 1 per cent. invert sugar are to be treated in the same way as solid sugar, and the results calculated according to Herzfeld's table. Molasses containing more than 1 per cent. invert sugar are treated in the same way, only less is taken for analysis according to the amount of invert sugar present. The cane sugar and raffinose are estimated according to Clerget's process.

Very full details of the precautions to be observed and the calculation of results are given in the report.—A. L. S.

*Determination of Crude Fibre and Starch.* M. Honig. *Chem. Zeit.* 14, 868 and 902—903.

IN the method about to be described both starch and crude fibre may be determined on the same portion, simply and quickly. In elaborating it the author has made the following observations:—

(1) Starch is converted *quantitatively* into a mixture of dextrin and soluble starch when heated with glycerol to 190° C. as described by Kabsch and Zulkowsky. At 110°—150° C. water vapour is given off, and continues to be evolved while the liquid, at first turbid, becomes opalescent and finally clear at 180°—190° C.

(2) The products of the above reaction contain so little dextrin that they are quantitatively precipitated from their aqueous solution by a mixture of four parts of alcohol and one of ether. This fact and that mentioned under (1) were proved by operating on an air-dried specimen of potato starch, which when converted into sugar direct gave 81.81 per cent. of real starch, as against 81.53, 81.68, and 81.75 when treated by the new method.

(3) Grape sugar is dehydrated and converted into caramel when treated with glycerol at 210° C., and its products yield nothing that might be reckoned as starch.

(4) Cellulose is unaffected by the treatment.

(5) Egg albumen treated by the process described above yields a soluble product, which is however not precipitated by ether alcohol, and may therefore be regarded as unlikely to influence the results.

On these data the following process is based:—2 grms. of the finely-divided substance are placed in a test tube which is ground into the neck of a flask provided with a side tube and containing sulphuric acid; 60 cc. of anhydrous glycerol are poured into the tube, a thermometer inserted as a stirring rod, and the temperature of the outer bath raised until 210° C. is registered. Complete decomposition is usually effected in half to three-quarters of an hour, and the cooling of the inner tube is hastened by removing it from the bath. Its contents is then poured in a thin stream into 200 cc. of 95 per cent. alcohol, stirring well and washing in any particles of cellulose by a fine stream of hot water, not more than 50 cc. being used.

After cooling the mixture, 50—60 cc. of ether are added with the double object of completing the precipitation of soluble starch and rendering the liquid easier to filter. After filtering on a plaited filter and washing with a mixture of five parts of alcohol and one of ether, the greater part of adhering liquid is removed from the filter by spreading it on a porous tile, and the precipitate, consisting of soluble starch and unattacked cellulose, is then washed with a flask with 100—150 cc. of hot water, and heated on a water-bath to dissipate the alcohol. Preferably the digestion is continued under a condenser after the addition of 10 cc. of hydrochloric acid of sp. gr. 1.125 for half an hour, the short heating leaving the cellulose unaffected. The cellulose, or rather crude fibre, is collected on a weighed filter, washed with hot water until the iodine reaction for starch is no longer given, and dried at 110° C. until its weight is constant. Besides cellulose and mineral constituents it contains a little nitrogenous matter, but the quantity is small and a sufficiently close result is got by merely deducting the ash.

The hydrochloric acid filtrate is made up to 250 cc., 200 cc. taken, 12 cc. hydrochloric acid, sp. gr. 1.125, added, and inverted by heating for  $2\frac{1}{2}$ —3 hours in a boiling water bath under a condenser, and titrated with Fehling's solution in the usual way.

Some results by the new method are appended:—

	Cellulose.		Starch.	
	I.	II.	I.	II.
Wheat.....	6.41	6.29	58.79	59.03
Maize.....	4.94	4.83	51.85	51.73
Oats.....	20.68	20.36	41.30	41.17
Barley.....	6.58	6.72	51.62	51.85

*The Analysis of Celluloid.* H. Zaunschirm. Chem. Zeit. **14**, 903.

CELLULOID generally consists of nitrocellulose and camphor coloured with a little mineral matter or some aniline dye. For most technical purposes a sufficiently approximate method consists in dissolving 0.25—0.3 grm. of the finely-shredded material in 5 cc. of concentrated sulphuric acid in the cup of a nitrometer, stirring with a platinum wire to prevent local heating and charring, and determining the evolved nitric oxide in the usual way, calculating it to nitrocellulose on the assumption that commercial dinitrocellulose has been used in the manufacture, and that therefore the nitrogen found amounts to 10.5 per cent. of the nitrated body, the theoretical percentage being 11.1. The difference between the percentage thus found and 100, gives the camphor, after allowance for any ash if mineral colouring matter has been used; an organic dye, if present, is generally in imponderable quantity. The presence of a carbonate or similar substance capable of evolving gas when treated with sulphuric acid would lead to difficulties, but no such constituent has hitherto been found in commercial samples. It is just possible that though camphor dissolves without evolution of gas in sulphuric acid, and is therefore apparently inert, it may undergo nitration to some extent, and forming a true nitro-derivative, unlike so-called nitrocellulose, may fail to yield its nitrogen when shaken in the nitrometer. This would of course lead to low results, and needs investigation. A more exact scheme suggested by the author, but not hitherto worked by him, consists in dissolving the substance in alcohol or methyl alcohol, and absorbing it in a weighed quantity of purified asbestos or pumice, drying and extracting with chloroform to remove camphor, the loss of weight indicating its amount, and dissolving out the nitrocellulose with absolute methyl alcohol, determining its proportion in the same way, and testing it afterwards if necessary in the nitrometer. Any mineral matter, together with traces of nitrocellulose, is left on the asbestos.—B. B.

*Cause of Error in Tartaric Acid Determinations.* Lampert and Co. Chem. Zeit. **14**, 903. (Compare this Journal, 1890, 420.)

THE numerous disputes and rival methods of analysis of sundry crude materials whence tartaric acid is obtained, have led to the belief that some humous substance must interfere with the ordinary reactions and be the cause of contradictory results. The authors show, however, that alumina is the real cause of the discrepancies, its presence lowering the percentage of acid found by the Goldenberg method, so that it is quite possible if enough be present to find no tartaric acid in a solution known to contain it in large quantity. Thus in a mixture of alum and tartaric acid containing 25 per cent. of the former a deficiency of 5 per cent. was observed, and in one containing 50 per cent. 15.5 per cent. was unaccounted for. How deceptive this may be is shown by the fact that an experienced chemist found 49.4 per cent. in a solution in which after it had been freed from alumina he discovered 80 per cent., a result confirmed by crystallisation on the large scale.

The method for separating alumina, being of some commercial value, is not given.—B. B.

*The Acetyl Numbers of Fats.* B. Wachtel. Chem. Zeit. **14**, 904.

THE validity of the application of the determination of the acetyl number of fats to the estimation of their content of hydroxy fatty acids by Benedikt and Ulzer has been disputed by Lewkowitsch (this Journal, 1890, 660) who has obtained evidence of the absorption of acetic anhydride by fats usually supposed free from such constituents. The author has tested the method on samples of commercial stearic and oleic acids and finds them to possess acetyl numbers of 0 and 6.2 respectively, results quite in accordance with Benedikt's view, instead of the figures 82.29 and 125.55 obtained by Lewkowitsch. (The fact

that oleic acid gives an acetyl number is accounted for by the oxidation which samples have generally suffered before examination.) This discrepancy led the author to examine a sample of beef tallow 15 years old, and one of the fat of the stag 28 years old, with the result that the former gave an acetyl number of 53.7 and the latter one of 40.1; it therefore appears that old samples may contain a sufficient quantity of hydroxy acids to lead to such figures as Lewkowitsch gives, without the accuracy of the method or worker being impugned. The figures for the stag fat are sufficiently interesting to reproduce at length. Acid number 61.7, saponification number 233.0, saponification number for the fatty acids 213.9, Reichert-Meissl number 8.0, acetyl number 40.1. Butter fat on the contrary does not form any large quantity of hydroxy acids on keeping, the acetyl number of one sample examined being only 9.6.—B. B.

*Contributions to the Study of the most Volatile Portion of Coal Tar.* J. Biehringer. Jour. f. Gasbeleuchtung, **33**, 341—345 and 358—359; also Dingl. Polyt. J. **276**, 78 et seq.

See under III., page 928.

*The Estimation of Cyanogen in Gas.* W. Leybold. J. f. Gasbeleuchtung, **33**, 427—428. (Appendix to the paper on "Cyanogen in Gas Manufacture," p. 923.)

A MIXTURE of 30 cc. of ferrous sulphate solution (1:10) and 20 cc. of caustic soda (1:3) is poured into 3 Woulff's bottles having a capacity of about 250 cc. and containing in each 20 cc. of the same soda solution. The mixture should be divided so that the first bottle contains 25 cc., the second 15 cc., and the last 10 cc. 100 litres of gas are passed through the system, by the aid of an aspirator if necessary, the meter employed being protected from the tar by a filter of wool. After the passage of the gas the contents of the bottles are washed out into a flask and boiled for not longer than 15 minutes, then allowed to settle and cool, and filtered into a 500 cc. graduated flask and made up to the mark with water. Of this solution 100 cc. (if the gas be taken from before the first purifier, otherwise 200 cc.) are acidified with hydrochloric acid, and ferric chloride (1:10) added in excess. The blue is filtered off and washed till the washings are colourless, put into a beaker with the filter paper and a little caustic soda added. After decomposition the ferric oxide is filtered off and washed till free from ferrocyanogen. The filtrate is then evaporated in a platinum basin to about 30 cc. and strongly acidified with sulphuric acid (1:10), evaporated to dryness, and ignited. The residue is extracted with 100 cc. of dilute sulphuric acid and washed with 50 cc. of water, and poured into a 250 cc. flask. 1 cc. of copper sulphate solution (1:10) and 10 grms. of pure zinc are added, and the whole allowed to stand till completely reduced. The iron is then determined with permanganate in the usual way.—F. H. L.

*Report on Textile Fibres and Derived Products.* Relazione nel. Lab. Chim. Centr. Gabelle, 358—402.

See under V., page 936.

## ANALYTICAL AND SCIENTIFIC NOTES.

*Formation of Nitrous Acid and Ammonia from Free Nitrogen.* O. Loew. Ber. **23**, 1443—1447.

IN experiments conducted with stringent precautions against the introduction of either nitrous acid or ammonia, the author found that dry platinum black which does not show even traces of either of these substances when treated with water develops evidences of both of them when treated with a solution of sodium hydroxide; with very dilute solutions (1 NaHO to 1,000 H<sub>2</sub>O), however, reactions for nitrous acid only are obtained. Inasmuch as the test for ammonia (Nessler's) was quite as delicate as the test employed for



the nitrous acid (sulphanilic acid and naphthylamine), the latter fact is regarded as indicating a twofold character in this platinum black and sodium hydroxide reaction:—1. The oxidation of the nitrogen condensed with the oxygen on the platinum black with the production of nitrous acid. 2. The reaction between the nitrogen and water in the presence of strong soda with the production of ammonia.

Passing air into potash holding platinum black in suspension is not a favourable manner of showing the reaction owing to the settling of the black, and it is better to provide an opportunity for the condensation of oxygen (and nitrogen) on the surface of the black. Moist platinum black, 23 to 24 grms. dry, mixed with about 5 grms. of freshly crystallised barium hydroxide and exposed two days in a desiccator over calcium chloride yielded about 0.7 mgrm. of nitrous acid according to an approximate estimation by the sulphanilic acid and naphthylamine reaction. Platinum black well washed with dilute sulphuric acid and water, suspended in absolute alcohol, then in a mixture of alcohol and chloroform, finally in chloroform alone, pressed and dried over sulphuric acid, yielded neither nitrous acid nor ammonia when treated with sodium hydroxide.—D. A. L.

## New Books.

A PRACTICAL TREATISE ON THE EXAMINATION OF AIR. With an Appendix on Illuminating Gas. By J. ALFRED WANKLYN and W. J. COOPER. London: Kegan Paul, Trench, Trübner, & Co., Limited. 1890.

SMALL 8vo. volume, bound in cloth, price 5s., containing Preface, Table of Contents, text covering 80 pages, and an Alphabetical Index. There are nine illustrative plates and six text illustrations. Pages 65 to 80 are devoted to an Appendix "On Illuminating Gas."

The gas apparatus of Hempel is that adopted and employed for the methods of analysis given. The authors say, on page 16, "The introduction of the gas burette, an implement which renders gases almost as manageable as liquids, has greatly aided in the popularisation of gas analysis. The most convenient of these implements is Hempel's. . . ."

The matter is subdivided as follows:—INTRODUCTION. 1. Nature and General Properties of Air and Gases. 2. Chemical Composition of the Atmosphere. 3. The Atmosphere as a whole, its Extent and Distribution. General Atmosphere above Sea Level. The Underground Atmosphere. 4. General Scope of Sanitary Air Analysis. CHAP. I. Endiometry and Complementary Endiometry. CHAP. II. Oxygen ( $O_2$ ). CHAP. III. Nitrogen ( $N_2$ ). CHAP. IV. Carbonic Acid ( $CO_2$ ). CHAP. V. Respired Air. Air from Furnaces. Carbonic Oxide, its Measurement in Air. Physiological Aspect of the Subject. Rules for reporting on Air. APPENDIX. On Illuminating Gas. Tables for Calculation. As regards analysis of illuminating gas, it may be well to refer for more detailed treatment to Wanklyn's "Gas Engineers' Chemical Manual."

The new method of determining carbonic oxide is based on the ascertained facts (1) that carbonic oxide, even in traces in the air, is extracted completely from that air by solution of subchloride of copper in hydrochloric acid, and (2) that when this copper solution is placed in a suitable vessel, the dissolved carbonic oxide can be driven out by means of acid solution of bichromate of potash and may be collected and measured. The method of determining oxygen in the air is an adaptation of the Cavendish and Priestley method, by absorption with nitric oxide gas. "The mode of operation is to add excess of nitric oxide to the sample of air, then to read the contraction. Under the heading, "The Metrical System and English Weights and Measures," the following statement occurs, which may be usefully quoted:—

"In Bunsen's table we read that a litre of nitrogen gas at 760 mm. pressure and zero centigrade weighs 1.2566 grms.

Therefore a cubic foot of nitrogen gas of 760 mm. pressure and zero centigrade weighs 1.2566 ounces avoird."

With regard to the relative importance of air and food the following sentence is quoted: "Between 30 and 40 lb. of air are breathed daily by each of us, and it is hardly an exaggeration to say that our aerial food which enters by way of the lungs dwarfs our liquid and solid food into insignificance." Of the importance of slight variations in the constituents of the air it is said, "There is nothing far-fetched or unreasonable in attaching importance to minute changes in the composition of the air we breathe."

THE CULTURE OF THE SUGAR-BEET AND MANUFACTURE OF BEET SUGAR. By H. W. WILEY, Chemist to the U.S. Department of Agriculture, Division of Chemistry. Washington: Government Printing Office. 1890.

OCTAVO volume, in paper cover, containing Introduction, 245 pages of Subject-matter, illustrated by 11 plates and 49 wood engravings, and an Alphabetical Index. The text is subdivided as follows:—"Early History of the Sugar-Beet and the Manufacture of Sugar therefrom." "History and Progress of the Culture of the Sugar-Beet and the Manufacture of Sugar therefrom in the United States of America." "Beet Sugar Factories at Chatsworth and other Places." "Comparative Development of the Cane and Beet Sugar Industry." "Statistics." "Production of Seed." "Varieties of Beets Grown in France and elsewhere. "Soil, Planting, and Cultivation." "Methods of Cultivation in Bohemia. Harvesting the Beets. Silos and Cellars. The Sugar-Beet in Oregon and Washington Territory. Fertilisers. Studies on the Development of the Sugar-Beet. Contribution to the Chemistry and Physiology of the Sugar Beet. Typical Forms of Sugar Beets. Meteorological Conditions. Recent Experiments looking to the Introduction of the Beet Sugar Industry. Experiments in Iowa. Experiments in Nebraska. "Experiments in South Dakota." Experiments in California. Tables of Work at Factory for each Week from September 16th to November 11th. Yield per Acre. Miscellaneous Experiments at the Factory. Beet Sugar Industry in Canada. Beet Sugar Industry in England. Beet Sugar Industry in the North-West. Use and Treatment of Pulp. Manufacture of Sugar.

## Trade Report.

### TARIFF CHANGES AND CUSTOMS REGULATIONS.

(From the Board of Trade Journal.)

#### SWITZERLAND.

#### Classification of Articles in Customs Tariff.

Note.—Quintal = 220.4 lb. avoirdupois. Franc = 9 $\frac{1}{10}$ d.

The following decisions affecting the classification of articles in the Swiss Customs tariff have recently been given by the Swiss Customs authorities:—

Sesquichloride of chrome (chromium).—Category 16. Duty, 30 cents, per quintal.

Aniline oil (raw aniline).—Category 17. Duty, 1 fr. per quintal.

#### UNITED STATES.

#### Customs Decisions.

Wood-pulp being manifestly not a manufacture of paper, but of wood, and not being intended for paper makers' use, imported expressly for filtering purposes, is dutiable at the rate of 35 per cent. *ad valorem*.

Binetro toluole, a chemical compound composed in part of benzole, which was probably derived from coal-tar,

and in part of nitric acid, which is obtained from a different source, is dutiable at the rate of 25 per cent. *ad valorem*.

Certain naphthol salt held to be dutiable, at the rate of 25 per cent. *ad valorem*, under the provision for chemical compounds and salts, in T. L., 92.

In case of oxygen gas imported in iron bottles or cylinders it was held that such bottles or cylinders are dutiable at the same rate as their contents, the value thereof being included in the dutiable value.

#### CONGO FREE STATE.

##### Export Duties on Caoutchouc and Ivory.

The *Bulletin du Musée Commercial* says that from and after the 1st October (1890), exports of caoutchouc and ivory are to be subjected to the payment of the following duties:—

Caoutchouc, 50 francs per 100 kilogrammes; ivory, 200 frs. per 100 kilos.

#### CANADA.

##### New Customs Tariff.

The following is a statement of rates of import duty now levied under the new Customs tariff of Canada. As the whole tariff is too voluminous for publication in one issue of the *Board of Trade Journal*, it is proposed to continue it in the next number:—

Articles.	Duties Levied.	
	Lb.	Dols. Cts.
Tallow.....	Lb.	0'01
Paraffin wax, stearic acid, and stearin of all kinds	"	0'03
Candles, tallow .....	"	0'02
Do., paraffin wax .....	"	0'05
Do., all other, including sperm.....	25 % <i>ad val.</i>	
Soap, common brown and yellow, not perfumed	Lb.	0'01½
Do., perfumed or toilet, the weight of the inside packages and wrappers to be included in the weight for duty .....	"	0'10 and 10 % <i>ad val.</i>
Castile soap, mottled or white, and white soap.	Lb.	0'02
Soap powders, pumice, silver, and mineral soap, sapollo, and other like articles, the weight of the package to be included in the weight for duty.....	"	0'03
British gum, dressing, sizing cream, and enamel sizing.....	"	0'01
Glue, sheet, broken sheet, and ground .....	"	6'03
Mucilage and liquid glue.....	30 % <i>ad val.</i>	
Saccharin, or any product containing over one-half of 1 per cent. thereof .....	Lb.	10'00
Spirituos or alcoholic liquors distilled from any material, or containing or compounded from or with distilled spirits of any kind and any mixture thereof with water, for every gallon thereof of the strength of proof, and when of a greater strength than that of proof at the same rate on the increased quantity that there would be if the liquors were reduced to the strength of proof. When the liquors are of a less strength than that of proof the duty shall be at the rate herein provided, but computed on a reduced quantity of the liquors in proportion to the lesser degree of strength; provided, however, that no reduction in quantity shall be computed or made on any liquors below the strength of 15 per cent. under proof, but all such liquors shall be computed as of the strength of 15 per cent. under proof, as follows:—		
(a.) Ethyl alcohol or the substance commonly known as alcohol, hydrated oxide of ethyl, or spirits of wine, gin of all kinds, not elsewhere specified, rum, whiskey, and all spirituous or alcoholic liquors, not otherwise provided for .....	Gall.	2'00
(b.) Amyl alcohol or fusel oil, or any substance known as potato spirit or potato oil	Gall.	2'00

#### CUSTOMS TARIFF OF CANADA—cont.

Articles.	Duties Levied.	
	Dols. Cts.	
Spirituos or alcoholic liquors, &c.— <i>cont.</i>		
(c.) Methyl alcohol, wood alcohol, wood naphtha, pyroxylic spirit, or any substance known as wood spirit or methylated spirit; absinthe, arrack, or palm spirit; brandy, including artificial brandy and imitations of brandy; cordials and liqueurs of all kinds not elsewhere specified; mescal, pulque, rum shrub, selvadon and other schnapps; tafia, angostura, and similar alcoholic bitters or beverages .....	"	2'00
(d.) Spirits and strong waters of any kind mixed with any ingredient or ingredients, and being or known or designated as anodynes, elixirs, essences, extracts, lotions, tinctures, or medicines not elsewhere specified .....	"	2'00 and 30 % <i>ad val.</i>
(e.) Alcoholic perfumes and perfumed spirits, bay rum, cologne, and lavender waters, hair, tooth, and skin washes and other toilet preparations containing spirits of any kind, when in bottles or flasks weighing not more than 4 oz. each.....	50 % <i>ad val.</i>	
When in bottles or flasks or other packages weighing more than 4 oz. ....	"	2'00 and 40 % <i>ad val.</i>
(f.) Nitrous ether, sweet spirits of nitre, and aromatic spirits of ammonia .....	"	2'00 and 30 % <i>ad val.</i>
(g.) Vermouth and ginger wine, containing not more than 40 per cent. of proof spirits.	"	0'75
If containing more than 40 per cent. of proof spirits .....	"	2'00
(h.) In all cases where the strength of any of the foregoing articles cannot be correctly ascertained by the direct application of the hydrometer, its shall be ascertained by distillation of a sample, or in such other manner as the Minister of Customs directs.		
Proprietary medicines, to wit, all tinctures, pills, powders, troches or lozenges, syrups, cordials, bitters, anodynes, tonics, plasters, liniments, salves, ointments, pastes, drops, waters, essences, oils or medicinal preparations or compositions recommended to the public under any general name or title as specifics for any diseases or affections whatsoever affecting the human or animal bodies, not otherwise provided for; all liquids 50 % <i>ad val.</i> , all other 25 % <i>ad val.</i> .....	50 % <i>ad val.</i> and 25 % <i>ad val.</i>	
Sulphuric ether .....	Lb.	0'05
Acid, acetic, and pyroligneous, not elsewhere specified, and vinegar, a specific duty of 15 cents for each gallon of any strength not exceeding the strength of proof, and for each degree of strength in excess of the strength of proof an additional duty of 1 cent. The strength of proof shall be held to be equal to 6 per cent. of absolute acid, and in all cases the strength shall be determined in such a manner as is established by the Governor in Council .....	Gall.	0'15 and 0'01 additional.
Acid, acetic, and pyroligneous, of any strength, when imported by dyers, calico printers, or manufacturers of acetates or colours for exclusive use in dyeing or printing, or for the manufacture of such acetates or colours in their own factories, under such regulations as are established by the Governor in Council.....	"	0'25 and 20 % <i>ad val.</i>
Do., muriatic and nitric .....	20 % <i>ad val.</i>	
Do., sulphuric.....	Lb.	0'00½
Do., sulphuric and nitric combined, and all mixed acids .....	25 % <i>ad val.</i>	
Do., phosphate .....	Lb.	0'03
Red and yellow prussiate of potash.....	10 % <i>ad val.</i>	
Aniline dyes not otherwise provided for.....	10 % <i>ad val.</i>	
Blacking, shoe and shoemakers' ink, and shoe, harness, and leather dressing, and harness soap.....	30 % <i>ad val.</i>	
Bluing, laundry bluing of all kinds.....	30 % <i>ad val.</i>	

CUSTOMS TARIFF OF CANADA—*cont.*

Articles.	Duties levied.
Lamp-black and ivory black.....	Dols. Cts. 10 <sup>7</sup> / <sub>16</sub> <i>ad val.</i>
Lead, nitrate and acetate of, not ground .....	5 <sup>7</sup> / <sub>16</sub> <i>ad val.</i>
Dry white and red lead, orange mineral, and zinc white.....	5 <sup>7</sup> / <sub>16</sub> <i>ad val.</i>
Colours, dry, not elsewhere specified .....	20 <sup>7</sup> / <sub>16</sub> <i>ad val.</i>
Oxides, ochres, and ochery earths, fireproofs, umbers and siennas, ground or unground, washed or unwashed, calcined or raw .....	30 <sup>7</sup> / <sub>16</sub> <i>ad val.</i>
Paints and colours, pulped or ground in oil or other liquids, and all liquid prepared or ready mixed paints not elsewhere specified .....	30 <sup>7</sup> / <sub>16</sub> <i>ad val.</i>
Paints, ground or mixed in or with either japan, varnish, lacquers, liquid driers, collodion, oil finish or oil varnish, rough stuff and fillers, the weight of the package to be included in the weight for duty .....	Lb. 0'55 and 25 <sup>7</sup> / <sub>16</sub> <i>ad val.</i>
Paints and colours, ground in spirits, and all spirit varnishes and lacquers .....	Gall. 1'00
Paris green, dry.....	10 <sup>7</sup> / <sub>16</sub> <i>ad val.</i>
Putty.....	25 <sup>7</sup> / <sub>16</sub> <i>ad val.</i>
Turpentine, spirits of.....	10 <sup>7</sup> / <sub>16</sub> <i>ad val.</i>
Varnishes, lacquers, japans, japan driers, liquid driers, collodion, and oil finish not elsewhere specified .....	Gall. 0'20 and 25 <sup>7</sup> / <sub>16</sub> <i>ad val.</i>
Carbolic or heavy oil, for any use.....	10 <sup>7</sup> / <sub>16</sub> <i>ad val.</i>
Cod liver oil, medicated .....	20 <sup>7</sup> / <sub>16</sub> <i>ad val.</i>
Essential oils for manufacturing purposes ....	20 <sup>7</sup> / <sub>16</sub> <i>ad val.</i>
Pomades, French or flower odours preserved in fat or oil for the purpose of conserving the odours of flowers which do not bear the heat of distillation, when imported in tins of not less than 10 lb. each .....	15 <sup>7</sup> / <sub>16</sub> <i>ad val.</i>
Perfumery, including toilet preparations (non-alcoholic), viz.:—Hair oils, tooth and other powders and washes, pomatums, pastes, and all other perfumed preparations not otherwise provided for used for the hair, mouth, or skin .....	30 <sup>7</sup> / <sub>16</sub> <i>ad val.</i>
Illuminating oils composed wholly or in part of the products of petroleum, coal, shale, or lignite, costing more than 30 cents per gallon .....	25 <sup>7</sup> / <sub>16</sub> <i>ad val.</i>
Oils, coal, and kerosine distilled, purified, or refined: naphtha, benzole, and petroleum: products of petroleum, coal, shale, and lignite, not elsewhere specified .....	Imp. gall. 0'07 <sup>1</sup> / <sub>2</sub>
Lubricating oils, composed wholly or in part of petroleum, and costing less than 30 cents per gallon .....	" 0'07 <sup>1</sup> / <sub>2</sub>
All other lubricating oils .....	25 <sup>7</sup> / <sub>16</sub> <i>ad val.</i>
Lard oil.....	20 <sup>7</sup> / <sub>16</sub> <i>ad val.</i>
Linseed or flax-seed oil, raw or boiled .....	Lb. 0'01 <sup>1</sup> / <sub>2</sub>
Nutsfoot, olive, sakul, or sesame seed oil.....	20 <sup>7</sup> / <sub>16</sub> <i>ad val.</i>
Vaseline, in bulk .....	Lb. 0'04

## THE McKINLEY TARIFF.

We print below a synopsis of the drug and chemical clauses of the new United States Tariff Bill as they have been amended by the Senate, to whom the Bill was sent up from the House of Representatives. So far as is known no alteration of any importance has been made in the Senate's decisions affecting this part of the tariff by the lower House, and our list virtually represents this part of the Bill as it has received the Presidential sanction. On the whole the House of Representatives has shown itself decidedly more Protectionist than the Senate, and had it had its way many duties would have been advanced or

retained that have now been left undisturbed or reduced. Among the principal reductions or abolitions of duties are those on tannic acid, beeswax, bristles, refined camphor, crude coal-tar, chloroform, dyewoods, nitrous and sulphuric ether, glycerin, indigo, iodine, iodoform, liquorice paste, magnesia, morphia, crude opium, several potash salts, strychnine, refined saltpetre, sulphur, and varnishes. The following are among the chief articles on which the duty has been increased:—Brushes, glue, gelatin, gold-leaf, hops, isinglass, liquorice juice, linseed and linseed oil, mercantiles, olive oil (salad), prepared opium, phosphorus, poppy oil, quicksilver, bichromate and chromate of soda, and sugar of milk. Peppermint oil will not, as stated by us in our editorial note of last week, be made dutiable at the rate of 1 dollar per lb. The House of Representatives proposed that duty, but the Senate struck out that clause and retained the 25 per cent. duty.

Articles.	Old Tariff.	Senate Bill.
Acid, acetic .....	2 at 10 c. lb.	1 <sup>1</sup> / <sub>2</sub> at 4 c. lb.
Acid, chromic .....	15 c. lb.	6 c. lb.
Acid, sulphuric.....	Free.	<sup>1</sup> / <sub>2</sub> c. lb.
Acid, tannic.....	\$1 lb.	50 c. lb.
Ammonia, muriate.....	10 %	<sup>1</sup> / <sub>2</sub> c. lb.
Ammonia, carbonate.....	20 %	1 <sup>1</sup> / <sub>2</sub> c. lb.
Ammonia, sulphate .....	20 %	<sup>1</sup> / <sub>2</sub> c. lb.
Acetate of lead, brown .....	4 c. lb.	3 <sup>1</sup> / <sub>2</sub> c. lb.
Acetate of lead, white .....	6 c. lb.	5 <sup>1</sup> / <sub>2</sub> c. lb.
Aniline oil .....	20 %	80 c. gal. at 30 %
Alcohol spirits.....	\$1 at 2 gall.	\$2 gall.
Antimony regulus .....	10 %	<sup>1</sup> / <sub>2</sub> c. lb.
Beeswax.....	20 %	Free.
Barytes.....	10 %	\$1 ton.
Blues, Berlin, Prussian, Chinese.....	20 at 25 %	6 c. lb.
Blanc fixe .....	25 %	<sup>1</sup> / <sub>2</sub> c. lb.
Blue vitriol .....	3 c. lb.	2 c. lb.
Bay rum.....	\$1 gall.	\$2 gall.
Bristles.....	16 c. lb.	Free.
Brushes.....	20 %	35 %
Camphor, refined .....	5 c. lb.	4 c. lb.
Cotton-seed oil.....	25 c. gall.	10 c. gall.
Chalk, French and red .....	20 %	1 c. lb.
Croton oil.....	50 c. lb.	30 c. lb.
Coal-tar, crude.....	10 %	Free.
Chloroform .....	50 c. lb.	25 c. lb.
Chrome colours .....	25 %	4 <sup>1</sup> / <sub>2</sub> c. lb.
Cocoa butter .....	"	3 <sup>1</sup> / <sub>2</sub> c. lb.
Cork and cork bark .....	25 %	5 c. lb.
Cork, manufactured.....	25 %	7 <sup>1</sup> / <sub>2</sub> c. lb.
Cod-liver oil .....	25 %	15 c. gall.
Degras oil.....	25 %	<sup>1</sup> / <sub>2</sub> c. lb.
Dyewoods .....	10 %	Free.
Dyewoods extracts, not enumerated.....	10 %	<sup>1</sup> / <sub>2</sub> c. lb.
Dandelion root.....	2 c. lb.	1 <sup>1</sup> / <sub>2</sub> c. lb.
Ether, nitrous spirit .....	30 c. lb.	25 c. lb.
Ether, sulphuric .....	50 c. lb.	40 c. lb.

THE McKINLEY TARIFF—*cont.*

Articles.	Old Tariff.	Senate Bill.
Fish oils, not enumerated.....	25	8 c. gall.
Gluce.....	20 <sup>2</sup>	1½ c. lb. at 30 <sup>2</sup>
Gelatin.....	30	1½ c. lb. at 30 <sup>2</sup>
Glycerin, crude.....	2 c. lb.	1½ c. lb.
Glycerin, refined.....	5 c. lb.	1½ c. lb.
Glucose.....	20 <sup>2</sup>	½ c. lb.
Gold-leaf.....	\$1.50 pkg.	82 pkg.
Grease.....	10	Free.
Hops.....	8 c. lb.	15 c. lb.
Hemlock extract.....	20 <sup>2</sup>	½ c. lb.
Hemp seed.....	½ c. lb.	25 c. bush.
Herring oil.....	25	8 c. gall.
Isinglass.....	25	1½ c. lb. at 30
Indigo.....	10	½ c. lb.
Indigo, carmine.....	10 <sup>2</sup>	10 c. lb.
Iodine.....	40 c. lb.	30 c. lb.
Iodoform.....	82 lb.	\$1.50 lb.
Kaolin.....	\$1.50 ton	83 ton
Liquorice paste.....	7½ c. lb.	5 c. lb.
Liquorice juice.....	3 c. lb.	5 c. lb.
Linseed.....	20 c. bush.	25 c. bush.
Linseed oil.....	25 c. gall.	32 c. gall.
Logwood extract.....	10	½ c. lb.
Magnesia.....	5 c. lb.	1 c. lb.
Magnesia, calcined.....	10 c. lb.	8 c. lb.
Magnesia, sulphate.....	½ c. lb.	3—10 c. lb.
Mercurial medicinal preparations.....	20 <sup>2</sup>	35
Mineral waters, artificial.....	34	..
Morphia.....	\$1 oz.	50 c. oz.
Matches.....	35	1 c. 1,000
Olive oil, salad.....	25	25 c. gall.
Orange mineral.....	3 c. lb.	3½ c. lb.
Ochre, dry.....	½ c. lb.	½ c. lb.
Opium, crude.....	\$1 lb.	Free.
Opium, prepared.....	\$10 lb.	\$12 lb.
Phosphorus.....	10 c. lb.	30
Potash, crude.....	20	Free.
Potash, carbonate.....	20 <sup>2</sup>	..
Potash, caustic.....	20 <sup>2</sup>	1 c. lb.
Potash, chlorate.....	3 c. lb.	Free.
Potash, sulphate.....	20 <sup>2</sup>	Free.
Poppy seed.....	½ c. lb.	25 c. bush.
Poppy-seed oil.....	Free.	32 c. gall.
Quicksilver.....	10 <sup>2</sup>	10 c. lb.
Sienna, dry.....	½ c. lb.	½ c. lb.
Soap, Castile.....	20	1½ c. lb.
Sumac extract.....	20 <sup>2</sup>	½ c. lb.
Seal oil.....	25	8 c. gall.

THE McKINLEY TARIFF—*cont.*

Articles.	Old Tariff.	Senate Bill.
Soda, bichromate and chromate.....	25 <sup>2</sup>	3 c. lb.
Strychnia.....	50 c. oz.	19 c. oz.
Salt-petre, refined.....	1½ c. lb.	1 c. lb.
Soda, bicarbonate.....	1½ c. lb.	1 c. lb.
Sulphur, refined.....	\$10 ton	88 ton
Sulphur, flowers.....	\$20 ton	\$10 ton
Spices, ground.....	5 c. lb.	3 c. lb.
Sugar of milk.....	Free.	8 c. lb.
Santonin.....	\$3 lb.	\$2.50 lb.
Turpentine spirits.....	20 c. gall.	Free.
Ultramarine.....	5 c. lb.	1½ c. lb.
Umber, dry.....	½ c. lb.	½ c. lb.
Varnishes.....	40	35
Varnishes, spirit.....	11 & 81.32 gall. 35	& 81.32 gall.
Vermilion.....	25	12 c. lb.
Whale oil.....	25	8 c. gall.
Wood tar.....	10	Free.
Wash blue.....	20	3 c. lb.

The duties on the following articles remain unchanged:—

Acid citric (10 c. per lb.), acid hydrochloric (free), acid tartaric (10 c. per lb.), alumina and sulphate of alumina (60 c. per cwt.), alkalis not enumerated (25 per cent.), alkaloids not enumerated (25 per cent.), alcoholic perfumery (82 per gall. and 50 per cent.), alcohol compounds (82 per gall. and 25 per cent.), barytes, manufactured (½ c. per lb.), borax, crude (3 c. per lb.), borax, manufactured (5 c. per lb.), boric acid, pure (5 c. per lb.), borate of lime (3 c. per lb.), bichromate of potash (3 c. per lb.), bone-black (25 per cent.), barks, beans, berries, balsams, or buds, not enumerated (10 per cent.), castor beans (50 c. per bushel), castor oil (80 c. per gall.), cream of tartar (6 c. per lb.), chalk (20 per cent.), chromate of potash (3 c. per lb.), copperas (¾ c. per lb.), coal-tar colours (35 per cent.), all other coal-tar products (20 per cent.), cocoa, manufactured (2 c. per lb.), dextrine (1 c. per lb.), dried insects (10 per cent.), ether not enumerated (\$1 per lb.), fruit essence (\$2.50 per lb.), gums and gum resins not enumerated (10 per cent.), glassware (45 per cent.), gum substitutes (1 c. per lb.), hempseed oil (10 c. per gall.), herbs not enumerated (10 per cent.), ink and ink-powders (30 per cent.), lard (2 c. per lb.), litharge (3 c. per lb.), leaves not enumerated (10 per cent.), medicinal preparations without alcohol (25 per cent.), medicinal preparations with alcohol (50 c. per lb.), ground mustard (10 c. per lb.), nutgalls not enumerated (10 per cent.), ochre in oil (1½ c. per lb.), oils, essential, distilled, or rendered, not enumerated (25 per cent.), opium extract (40 per cent.), peppermint oil (25 per cent.), potassium iodide (50 c. per lb.), potassium iodide, red prussiate (10 c. per lb.), potassium iodide, yellow prussiate (5 c. per lb.), paints or colours not enumerated (25 per cent.), rapeseed oil (10 c. per gall.), Rochelle salts (3 c. per lb.), roots and stems not enumerated (10 per cent.), sienna in oil (1½ c. per lb.), soap (20 per cent.), soap, fancy (15 c. per lb.), sponges (20 per cent.), sumac, ground (¾ c. per lb.), soda ash or crystals (½ c. per lb.), soda bichromate (3 c. per lb.), soda caustic (1 c. per lb.), soda sulphate (20 per cent.), soda silicate (½ c. per lb.), spices, ground, not enumerated (10 per cent.), seeds not enumerated (10 per cent.), silver-leaf (75 c. per package), and starch (2 c. per lb.).

—*Chemist and Druggist.*

## THE FINAL SHAPE OF THE MCKINLEY BILL.

*O. P. & D. Reporter* states that the following alterations have been made in the drug and chemical section of the McKinley Bill since it was sent back from the Senate to the House of Representatives:—Tannic acid has been changed to 75 c. per lb.; barytes to \$1-12 per ton; manufactured ditto to \$6-72 per ton; bronze powders to 12 c. per lb.; bay rum to \$1-50 per gallon; bristles to 10 c. per lb.; brushes to 10 per cent. *ad val.*; cork and cork bark to 10 c. per lb.; manufactured cork to 15 c. per lb.; Dutch metal-leaf to 8 c. per package; hemlock extract to  $\frac{1}{2}$  c. per lb.; liquorice paste and juice to  $5\frac{1}{2}$  c. per lb.; logwood extract to  $\frac{7}{8}$  c. per lb.; peppermint oil to 80 c. per lb.; phosphorus to 20 c. per lb.; ground sumac to  $\frac{1}{4}$  c. per lb.; and extract to  $\frac{7}{8}$  c. per lb.; sulphate of soda to \$1-25 per ton; tallow to 1 c. per lb.

## UNITED STATES.

*The New American Tariff.*

The special point in connexion with the new American tariff regulations is the requirement of the consular and Customs authorities that manufacturers and shippers of merchandise to the United States should make a declaration showing (1) the cost of the raw goods and their manufacture combined; (2), all other charges incident to preparing goods ready for shipment, and (3) a percentage—not less than 8 per cent. on items Nos. 1 and 2—to bring the value up to market value. In view of the new law it is felt that there is no option but to give the required information to the consular and Customs authorities, but serious objection is taken to supplying particulars to consignees in America of the cost of producing all goods that are shipped, and the private charges thereon. The form above referred to is to be filled up in triplicate, one copy being for the Consul on this side, one for the Customs authorities, and one for the consignee. There is a natural objection to supplying the third form, and the London Chamber of Commerce is in correspondence with the American Consul in London with the object of ascertaining how far any modification of the regulations is possible. In this connexion the following paragraph is of interest:—

*Instructions to the Customs under the McKinley Bill.*

The following circular instructions have been distributed by the Secretary to the Treasury of the United States to collectors and other officers of Customs to govern proceedings under the Customs administrative law—otherwise known as the McKinley Bill—which came into effect on the 1st August:—(I.) It is proper that certain general principles should be announced which in the opinion of the department must govern the application of the various provisions of the new law to the current business at the several ports. (II.) The duties upon imported merchandise are held to attach to the merchandise at the time of its arrival in a port of the United States. The date of arrival of merchandise intended for immediate transportation without appraisement will be that of its arrival in an exterior port of the United States. Therefore the assessment of duties upon goods which arrive on or before July 31st instant, will be made on the basis of the present laws, even if entry of such merchandise shall be deferred until after the 31st day of July. (III.) Invoices authenticated before the 1st day of August, proximo, will be accepted at any time thereafter, provided the consular certificates conform to the present requirements of law. Consular certificates after the 1st day of August must conform to the requirements of the Act of June 10th, 1890, but in view of possible difficulties in obtaining an immediate conformity with such requirements invoices which are accompanied by certificates not in all respects in accordance with the law of June 10th, 1890, will be regarded as informal and will be accepted as *pro forma* invoices, under bond to produce a properly certified invoice within six months from the date of entry. (IV.) In the entry of merchandise bills of lading drawn to the order of persons doing business as bankers and indorsed by them to the order of the actual importers of the merchandise specified

therein will be accepted when presented by such importers in making entry of said merchandise the same as if originally drawn to their order. (V.) After July 31st, 1890, no damage warrants shall be issued by collectors of Customs, but proceedings under warrants issued on or before that date will be carried on as under the present law. (VI.) In case of protest against the action of collectors or of appraisers which may be filed on or after the 1st day of August, proceedings will be governed by the law of June 10th, 1890, even when such protests shall relate to merchandise which shall have arrived on or before July 31st, 1890. But all proceedings on the estimate and assessment of duties upon such merchandise and upon the original appraisement and liquidation of the same shall conform to the requirements of the law under which the goods were imported. Invoices covering goods which are in bonded warehouse on August 1st will not therefore require re-liquidation under the new law. (VII.) A list will be furnished prescribing such fees as will be charged after August 1st, 1890. All other fees are held to be abolished under the new law. (VIII.) Attention is called to the provisions of section 19 of the new law which define the dutiable value of imported merchandise to consist of all costs and charges that attach to the goods in condition packed ready for shipment to the United States. All such costs and charges which are specified in the invoice will therefore be included in determining the value for the original estimate of duty. (IX.) It is the opinion of the department that section 20 of the new law permits the retention of merchandise in bonded warehouse for more than one year, and not more than three years from date of importation without the imposition of the additional duty of 10 per cent. provided by section 2970 Revised Statutes upon its withdrawal for consumption.—*London Chamber of Commerce.*

## EXTRACTS FROM DIPLOMATIC AND CONSULAR REPORTS.

## GERMANY.

The following figures indicate the principal chemicals (in quantities of 100 kilos. = 2 cwt.) exported from Germany to Great Britain during the last three years:—

	1887.	1888.	1889.
Ultramarine .....	12,352	10,322	20,053
Alum, alumina .....	19,568	18,836	26,891
Soda, calcined .....	4,098	2,576	3,500
Soda, crystals .....	3,793	334	619
Potash .....	16,647	16,257	15,092
Alizarine .....	24,305	29,050	31,637
Aniline and transition products to tar colours .....	1,563	1,729	2,653
Aniline dyes and tar colours .....	11,385	11,848	13,866
White lead and zinc white .....	71,130	79,150	117,974
Chloride of potash .....	254,999	153,215	148,315
Glycerin, refined and crude .....	2,338	4,644	7,550
Sulphate of potash .....	36,912	44,579	57,335
Mineral waters, artificial and natural	25,956	25,832	33,400
Soda and sulphate of soda .....	8,022	4,472	5,548
Saltpetre (soda) .....	4,311	5,292	11,777
Saltpetre (potash) .....	14,091	16,637	40,044
Vitriol of every description .....	506	1,257	3,125
Tartaric acid .....	6,983	8,019	8,107

The following are the principal exports of chemicals from Great Britain to Germany:—

	1887.	1888.	1889.
Caustic soda.....	12,090	9,365	8,576
Alum, alumina .....	1,270	526	447
Chloride of lime .....	29,787	40,819	33,910
Dye-wood extract .....	2,295	2,766	3,134
Bicarbonate of soda.....	3,637	4,884	6,704
Calced soda.....	9,434	4,094	3,387
Soda crystals .....	3,297	1,056	281
Ammonia, carbonated sal-ammonia, &c.	5,062	5,327	8,858
Sulphate of ammonia .....	245,519	246,004	278,295
Aniline dyes, &c.....	1,274	1,934	925
Cutch and gambier .....	19,735	16,976	33,758
Cinchona .....	25,147	22,894	27,658
Chlorate of potash .....	5,060	4,062	5,312
Chromate of potash .....	5,954	8,416	9,473
Dye-woods .....	30,340	21,324	52,133
Glycerin, refined and crude.....	10,034	15,221	18,616
Indigo.....	5,238	6,132	7,489
Bone black .....	14,697	29,499	54,219
Sulphate of soda.....	3,243	824	2,430
Saltpetre (Chili) .....	17,466	46,234	52,341
Sulphur .....	2,369	6,289	4,129
Turpentine, &c.....	5,622	6,497	6,799
Vitriol.....	926	734	6,373
Quicksilver.....	1,263	1,078	1,134
Pepper.....	16,280	15,541	17,668
Tea.....	6,123	6,475	6,293
Cotton oil .....	37,987	34,828	39,628
Linseed oil.....	170,058	199,799	246,610
Olive oil .....	1,204	1,918	1,174
Palm and coconut oil .....	67,083	65,762	65,128

#### MEXICO.

##### Imports from United Kingdom into Vera Cruz.

Last year the imports from the United Kingdom into Vera Cruz increased 76 per cent., from all other countries 27 per cent., in spite of the fact that cottons (of which a large percentage of the British imports consists) pay an average duty of 133 per cent., and that the average of all dutiable goods imported from this country is 121 per cent., while the average of goods from all countries is "only" 91 per cent.

Drugs pay an average duty of 60 per cent. *ad valorem*. The value of the imports during the last two years has been as follows:—

	1889.	1888.
	£	£
Drugs from United Kingdom .....	13,650	18,513
Drugs from all parts.....	120,632	95,005
Quicksilver from United Kingdom...	50,695	42,951
Quicksilver from all parts.....	100,605	85,384

#### Openings for Industries in Mexico.

The duties which protect the national industry are made to press so heavily on ordinary articles that none but the rich purchase those imported, "everyone is forced to consume national products" being a maxim of the Mexican lawgivers. The duties on glassware are from 3 to 45 centavos per kilo., which has to be paid even on articles broken in transit. Raw materials for making articles of this kind abound in the country, but the national products are still so dear that the poorer classes cannot procure enough for their requirements, and they use the most ordinary qualities. Mexican factories only produce window glass and glass bottles at fabulously high prices; an 18-inch square of common glass costs 75 centavos (equal to about 1s. 10½d.), while itinerant dealers buy up old bottles at the rate of about 3s. 4d. the dozen. As to the chemical industry, the Belgian Consul in Mexico is of opinion that an establishment engaging therein would be excessively productive. Only two chemical factories exist at present, which turn out specially sulphuric, hydrochloric, and nitric acids, securing enormous profits. Foreign sulphuric acid sells at 16 centavos per kilo., hydrochloric acid at 40 centavos, and nitric acid 65 centavos per kilo. On the other hand, Mexican-made sulphuric acid sells at 12 to 14 centavos the kilo. The dearthness of sulphuric acid is such that many metallurgical works make their own acid. Cheap acid would not only "regenerate" industry, but those who produce it would "doubtless do brilliant business." In other allied branches the Consul is also of opinion that there are suitable openings for manufacturers who will study the matter.

#### SIAM.

##### Sapan Wood.

The export of this valuable dye-wood from Bangkok has decreased each year since 1884, when the export was 3,079 tons. In 1887 it fell to 1,772 tons, and in 1888 to 1,372 tons. The sapan wood forests are, it appears, much exhausted, and without forest regulations there is danger of the wood ceasing to be an article of export. The supply from the Raheng district, whence most of the wood formerly came, has now almost ceased, and that which finds its way to Bangkok is derived chiefly from the Lakhon forests, the trees being torn up by the roots and floated down by way of Raheng to Bangkok. Most of the sapan wood shown in the returns is exported in the form of dunnage with rice cargoes.

##### Cutch.

The returns show an export of cutch to the value of 411, only from Bangkok. The bark of the cutch tree, which grows abundantly in the districts of Lakhon and Phræ in Northern Siam, is boiled into conical lumps the size of a fowl's egg by the inhabitants of those provinces for local use; but the industry appears to be little developed. The admixture of a small piece of cutch with the other ingredients of a quid of betel is considered a delicacy amongst the Laos. The use of the bark as a dye is little known or cultivated by them.

## MISCELLANEOUS TRADE NOTICES.

## THE COST OF LABOUR IN THE UNITED STATES.

Some useful figures have been published by the Boston, Mass., Labour Bureau, showing the proportion to the cost of certain goods, manufactured in that State, of wages, raw

material, and general expenses, such as insurance, taxation, rent, &c. The following are some figures representing the cost of manufacturing chemicals in a wholesale way, the figures in the first column representing the quantities upon which the cost was actually tested:—

	Quantity Experimented on.	Raw Material.	Wages.	General Expenses.
		Per Cent.	Per Cent.	Per Cent.
Compound chemical preparations (unenumerated) .....	1,000 galls.	95.24	1.76	..
Sulphuric acid .....	1 ton	53.41	14.87	32.22
Prussian blue, needles .....	2,000 gross	15.65	13.09	71.26
Illuminating oil .....	Per barrel	67.50	6.79	25.71
Copperas .....	3,185 tons	41.66	22.91	35.43
Brunswick black (Japan) .....	Per lb.	70.59	23.53	5.88
Green paint .....	1,000 lb.	90	10	..
Mixed paints (unenumerated) .....	Per gall.	90	10	..
Candles .....	Per lb.	95.83	4.17	..
Soap .....	Per cwt.	66.67	8	25.53
Soft soap .....	20 barrels	75	12.50	12.50
Fuller's earth (mineral soap) .....	Per box	80.47	10.53	..
Soap for textiles .....	980 tons	61.36	8.79	30.05
Powdered soap (large quantity) .....	Per lb.	57.14	7.14	35.72
Powdered soap .....	1,400 lb.	77.78	22.22	..
Tallow .....	175 tons	95	4.80	..
Tallow oil .....	100 lb.	93.75	6.25	..
Glue .....	150 tons	86.80	10.41	2.79
Starch .....	Per barrel	81.45	18.55	..
Dyestuffs .....	Value, \$72,000	..	20.50	..
Oils and illuminants .....	Value, \$97,000	..	5.92	..
Paints, colours, and heavy chemicals .....	Value, \$164,000	..	16.50	..

The maximum weekly wage of operatives employed in the preparation of chemicals is 3*l.* to 4*l.*; in dyestuffs the average wage is 2*l.* to 2*l.* 8*s.*; in the oil trade, also 2*l.* to 2*l.* 8*s.*; in paints and crude chemicals, 2*l.* 8*s.* to 3*l.*; in dye and bleaching works, about 1*l.* 8*s.* to 1*l.* 10*s.*

## MANGANATE OF SODA.

At the usual weekly meeting of the members of the London county council on October 21st, held at County Hall, Spring Gardens, Charing Cross, Sir J. Lubbock, Bart., M.P., presiding, one of the committees said the chemist to the council had reported that during the past summer there had been used in deodorising the sewage 1,654 tons of manganate of soda, and 749 tons of sulphuric acid, at a cost of £4,560*l.* 18*s.* 1*d.* The cost for similar operations in the year 1889 was 25,305*l.* In order that the most advantageous terms might be obtained from contractors, who could work to better advantage when ample notice was given, the committee recommended: "That an advertisement be issued inviting tenders for the supply of 3,500 tons of manganate of soda of 30 per cent. quality, and 1,000 tons of sulphuric acid, during the summer of 1891." This was agreed to.—*Chemist and Druggist.*

## TRADE IN CUBA.

Trade in Cuba during 1889 was satisfactory on the whole, and the output of iron ore also augmented. The exports of wax were 34,430 lb., against 32,316 lb. of the previous year. Four-fifths went to the United States, and the rest to Germany and Spain. The average price was 2*l.* 8*s.* 6*d.* per 100 lb. The exports of iron ore are, so far, confined to the output of the Juragua Iron Company, which is steadily on the increase. In 1889 the production amounted to 248,430 tons of ore, containing on the average 64 per cent. of metallic iron, against 202,625 tons in the preceding year. The ore is not sold in Cuba, but is sent to the United States for account of the company. The price realised in the States is not given, but it costs the company 6*s.* 4*d.* per ton put on board ship at St. Jago, exclusive of first cost of the mines, and is yielding a profit of from 2 dols. to 2½ dols.—say 8*s.* 1*d.* to 10*s.* 5*d.*—per ton. Manganese is comparatively a new item of export, having only commenced in 1888, during which year 1,981 tons of ore, containing from 50 to 55 per cent. of metallic manganese, were shipped to the United States, and 604 tons went on last year. This industry is on the increase, and yields a fair profit to the miners. It costs about 30*s.* to put on board ship at St. Jago, excepting the ore from some mines at a further distance, which costs about 12*s.* per ton more.—*Ironmonger.*



## GERMAN PHARMACEUTICAL INDUSTRY.

The following interesting figures are published in the *Chem. Zeit.* as indicative of the increase or decrease in the consumption of some important drugs and dyestufferies in Germany. The figures represent quantities of 1 ton (20 cwt.) weight:—

Year.	Dyewoods.	Indigo.	Cinchona.	Copra, Cocoa, and Palm Nuts.	Cochineal.
1836-40	9,007	361	..	..	..
1846-50	12,900	884	..	..	..
1850-60	20,603	654	..	..	..
1871-75	20,931	1,050	1,028	21,146	526
1881-85	41,212	1,181	2,876	60,651	111
1888	55,733	1,016	4,671	100,258	77
1889	53,173	1,119	5,120	101,832	35

Year.	Cutch.	Gum Arabic and Tragacanth.	Shellac	Olive Oil in Casks.	Camphor.
1872-75	3,513	1,393	..	..	..
1876-80	4,292	1,084	..	..	..
1881-85	5,972	1,513	1,137	19,895	..
1888	5,754	1,190	1,396	9,795	378
1889	5,791	1,318	919	11,213	353

## CHEMICAL WORKS IN VICTORIA.

According to a Government paper recently issued, there are in the colony of Victoria 11 chemical works, employing in all 188 persons, and with plant worth 26,100/. Surgical instrument factories appear to the number of five, with 28 hands and plant worth 1,465/. The aerated waters industry includes 160 works, employs 1,183 persons, and owns plant worth 124,688/.

## BOARD OF TRADE RETURNS.

## SUMMARY OF IMPORTS.

	Month ended 30th September	
	1889.	1890.
Metals.....	£ 1,823,855	£ 1,921,250
Chemicals and dyestuffs.....	461,394	502,119
Oils.....	669,389	680,241
Raw materials for non-textile industries.....	1,665,594	1,676,511
Total value of all imports ....	33,406,385	35,551,688

## SUMMARY OF EXPORTS.

	Month ended 30th September	
	1889.	1890.
Metals (other than machinery) ....	£ 3,200,232	£ 3,578,969
Chemicals and medicines .....	611,152	779,928
Miscellaneous articles.....	2,433,175	2,977,539
Total value of all exports.....	19,932,545	22,764,677

## IMPORTS OF METALS FOR MONTH ENDED 30TH SEPTEMBER.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Copper:—			£	£
Ore..... Tons	8,866	9,993	66,628	91,617
Regulus..... "	9,581	9,553	231,958	281,199
Unwrought .... "	3,880	4,501	170,914	274,223
Iron and steel:—				
Iron ore..... "	279,525	282,768	208,657	216,264
Iron bolt, bar, &c. "	11,265	10,849	100,055	106,683
Steel, unwrought.. "	563	1,119	4,907	11,312
Lead, pig and sheet ..	12,828	11,713	161,831	158,009
Pyrites..... "	39,277	18,667	76,283	88,767
Quicksilver..... Lb.	219,030	22,500	39,100	3,637
Tin..... Cwt.	57,739	25,136	261,848	125,883
Zinc..... Tons	6,125	6,602	121,185	139,639
Other articles... Value £	..	..	386,106	421,797
Total value of metals	..	..	1,823,855	1,921,250

## IMPORTS OF CHEMICALS AND DYESTUFFS FOR MONTH ENDED 30TH SEPTEMBER.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Alkali..... Cwt.	3,852	2,133	£ 2,676	£ 2,219
Bark (for tanners, &c.)..... "	32,579	57,925	19,376	20,196
Brimstone..... "	10,572	10,894	9,211	9,421
Chemicals..... Value £	..	..	110,364	113,532
Cochineal..... Cwt.	565	651	3,162	4,585
Cutch and gambier Tons	1,877	2,612	53,351	72,350
Dyes:—				
Aniline..... Value £	..	..	21,837	19,970
Alizarine..... "	..	..	20,800	36,180
Other..... "	..	..	1,353	929
Indigo..... Cwt.	3,727	821	47,845	19,570
Madder..... "	475	765	520	1,016
Nitrate of soda.... "	53,197	96,700	22,253	40,874
Nitrate of potash . "	19,271	19,810	17,389	18,379
Valonia..... Tons	1,169	1,303	16,131	25,639
Other articles... Value £	..	..	121,506	126,554
Total value of chemicals	..	..	661,394	502,119

## IMPORTS OF OILS FOR MONTH ENDED 30TH SEPTEMBER.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Cocoa-nut..... Cwt.	45,406	23,902	£ 58,673	£ 45,209
Olive ..... Tuns	862	924	33,257	34,734
Palm ..... Cwt.	112,312	90,318	123,953	111,834
Petroleum ..... Gall.	10,343,164	8,667,306	250,088	195,442
Seed ..... Tons	1,633	3,311	43,679	84,117
Train, &c..... Tuns	2,197	1,816	48,275	37,576
Turpentine ..... Cwt.	33,383	51,751	51,627	82,087
Other articles .. Value £	..	..	57,857	88,942
Total value of oils ...	..	..	669,389	680,241

## IMPORTS OF RAW MATERIALS FOR NON-TEXTILE INDUSTRIES FOR MONTH ENDED 30TH SEPTEMBER.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Bark, Peruvian .. Cwt.	12,432	9,751	£ 36,464	£ 28,566
Bristles..... Lb.	296,140	361,826	37,523	51,033
Caoutchouc..... Cwt.	22,500	24,767	239,263	358,291
Gum:—				
Arabic..... ..	4,076	1,928	12,499	7,305
Lac, &c..... ..	4,855	2,624	17,614	12,804
Gutta-percha ....	4,702	7,075	42,506	79,708
Hides, raw:—				
Dry..... ..	33,123	34,178	87,301	86,635
Wet..... ..	76,823	48,668	178,520	111,263
Ivory..... ..	833	1,921	37,828	95,672
Manures:—				
Guano..... Tons	2,361	2,403	14,345	14,183
Bones..... ..	2,771	3,191	13,812	16,170
Paraffin..... Cwt.	28,993	45,189	35,316	58,670
Lozen rag3..... Tons	3,254	2,889	31,400	28,564
Esparto, &c. ....	13,888	9,430	66,301	48,886
Putrified wood ....	10,965	13,515	60,615	76,581
Rosin..... Cwt.	69,819	158,158	22,022	35,534
Tallow and stearin ..	81,776	116,669	105,394	153,582
Tar..... Barrels	26,071	40,944	22,366	28,270
Wood:—				
Hewn..... Loads	286,136	257,326	689,839	573,785
Sawn..... ..	722,491	737,373	1,816,173	1,623,120
Staves..... ..	21,340	21,490	82,885	83,216
Mahogany .... Tons	2,237	2,724	20,730	24,913
Other articles.... Value £	..	..	944,618	1,003,773
Total value .....	..	..	1,665,394	1,676,544

Besides the above, drugs to the value of 61,000*l.* were imported, as against 72,283*l.* in September 1889.

## EXPORTS OF METALS (OTHER THAN MACHINERY) FOR MONTH ENDED 30TH SEPTEMBER.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Brass..... Cwt.	7,789	7,931	£ 30,959	£ 39,235
Copper:—				
Unwrought..... ..	63,288	78,116	145,331	244,852
Wrought..... ..	34,596	14,928	95,459	54,371
Mixed metal ....	32,709	29,208	80,441	89,044
Hardware..... Value £	..	..	209,770	236,340
Implements..... ..	..	..	87,132	111,594
Iron and steel..... Tons	351,057	323,320	2,341,396	2,488,331
Lead..... ..	3,195	4,057	43,875	58,911
Plated wares... Value £	..	..	36,665	34,129
Telegraph wires... ..	..	..	38,099	98,104
Tin..... Cwt.	6,698	7,239	31,708	36,229
Zinc..... ..	3,593	11,629	3,288	12,011
Other articles .. Value £	..	..	56,109	74,917
Total value .....	..	..	3,200,232	3,578,959

## EXPORTS OF DRUGS AND CHEMICALS FOR MONTH ENDED 30TH SEPTEMBER.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Alkali..... Cwt.	488,933	597,686	£ 126,737	£ 210,253
Bleaching materials ..	139,834	183,741	51,257	53,703
Chemical manures. Tons	28,918	34,662	165,108	180,434
Medicines..... Value £	..	..	63,473	91,118
Other articles ...	..	..	204,577	235,420
Total value .... ..	..	..	611,152	770,928

## EXPORTS OF MISCELLANEOUS ARTICLES FOR MONTH ENDED 30TH SEPTEMBER.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Gunpowder..... Lb.	806,300	880,600	£ 20,671	£ 21,138
Military stores.. Value £	..	..	119,571	114,916
Candles..... Lb.	862,400	1,048,100	15,413	20,173
Caoutchouc..... Value £	..	..	97,996	116,426
Cement..... Tons	23,006	53,837	46,669	119,984
Products of coal Value £	..	..	84,359	121,531
Earthenware ... ..	..	..	158,518	158,951
Stoneware..... ..	..	..	11,320	11,860

EXPORTS OF MISCELLANEOUS ARTICLES, &c.—*cont.*

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Glass:—			£	£
Plate..... Sq. Ft.	208,619	160,978	16,132	11,522
Flint..... Cwt.	6,120	10,942	15,993	24,297
Bottles..... "	62,531	84,857	8,992	40,667
Other kinds.... "	20,309	18,534	14,745	16,114
Leather:—				
Unwrought .... "	12,826	13,855	117,941	121,480
Wrought ..... Value £	..	..	38,096	34,664
Seed oil..... Tons	4,110	7,294	92,684	123,833
Floor cloth .... Sq. Yds.	1,213,200	1,357,200	56,911	63,326
Painters' materials Val. £	..	..	99,137	137,579
Paper ..... Cwt.	50,610	87,960	95,116	151,146
Rags..... Tons	3,977	4,796	32,642	33,828
Soap..... Cwt.	36,138	39,504	36,795	44,968
Total value .....	..	..	2,433,475	2,977,539

## TRADE BETWEEN SPAIN AND THE UNITED KINGDOM.

## IMPORTS INTO THE UNITED KINGDOM FROM SPAIN FOR THE THREE MONTHS ENDED 30TH SEPTEMBER.

Principal Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Chem. products unenumerated Value £	..	..	£	£
Copper ore and regulus ..... Tons	20,949	19,389	362,561	457,850
Copper, unwrought, and part wrought. "	..	..	..	..
Lead ore..... "	690	408	5,770	3,524
Lead, pig and sheet .....	19,111	16,478	243,492	217,176
Manganese ore.. "	3,630	1,650	9,210	3,870
Pyrites of iron or copper .....	115,414	140,797	225,643	240,783
Quicksilver ..... Lb.	132,186	46,952	12,027	5,760
Rags, esparto ... Tons	13,916	14,172	82,989	82,520
Zinc ore .....	..	218	..	926
Total value .....	..	..	2,316,390	2,617,156

## EXPORTS OF HOME PRODUCE FROM THE UNITED KINGDOM TO SPAIN FOR THE THREE MONTHS ENDED 30TH SEPTEMBER.

Principal Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Alkali..... Cwt.	79,439	86,385	£	£
Caoutchouc manufactures .... Value £	..	..	5,526	1,647
Cement ..... Tons	1,737	1,747	3,497	3,425
Chemicals (including dye-stuffs) ..... Value £	..	..	14,684	19,621
Products of coal (including petroleum) ..	..	..	3,855	8,832
Glass manufactures ..	..	..	2,682	2,992
Manure..... "	..	..	27,360	12,865
Oil, seed ..... Tons	79	149	1,728	3,295
" other sorts.. Value £	..	..	3,055	4,975
Oil and floor cloth ... Sq. yds.	123,300	133,500	4,846	4,976
Painters' colours &c. .... Value £	..	..	8,734	9,598
Paper of all sorts Cwt.	855	1,419	2,053	3,214
Soap..... "	702	561	789	709
Total value .....	..	..	1,976,120	1,252,919

## Monthly Patent List.

\* The dates given are the dates of the Official Journals in which acceptances of the Complete Specifications are advertised. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the said dates.

## I.—GENERAL PLANT, APPARATUS, AND MACHINERY.

## APPLICATIONS.

14,543. E. F. Edgar. Improvements in furnaces. Complete Specification. Filed September 16. Date applied for, March 27, 1890, being date of application in United States.

14,846. W. H. Kennedy. See Class X.

15,008. H. H. Hazard. See Class II.

15,254. J. Joly. Improved method of desiccating substances. September 26.

15,277. F. M. Robertson. A new or improved process for desiccation or evaporation. September 26.

15,604. W. Cole. An improved apparatus for supplying air under pressure. October 2.

15,605. W. Cole. An improved apparatus for supplying air under pressure. October 2.

15,659. F. Garside. An improved method for the evaporation of water or other liquids in order to extract materials held in solution, and apparatus therefor. October 3.

15,698. R. A. Robertson (of the Firm of The Mirreles, Watson, and Varyan Co., Lim.) and D. Ballingall. Improvements in apparatus for evaporating, concentrating, and distilling liquids. October 1.

15,930. C. Kellner. See Class XIX.

15,931. C. Kellner. See Class XIX.

15,975. G. J. Davis and E. Jones. An improved manufacture of compound suitable for forming joints in steam cylinders and pipes, also vessels for containing water, acids, vapours, and for other purposes. October 8.

15,976. G. J. Davis and E. Jones. An improved manufacture of compounds suitable for coverings for floors, walls, ceilings, also for tubes, insulating coverings, and other purposes. October 8.

16,192. A. Estrayer. Improvements in filter presses and oil presses. October 11.

16,251. H. H. Badams. Improvements in filter presses. October 13.

16,263. W. S. Dimes. Improvements in apparatus for agitating and mixing liquids. October 13.

16,326. T. Slater. Improvements in multiple evaporating apparatus. October 14.

16,381. J. Dunn. Improvements in apparatus for drying semi-liquid granular or pulverulent substances. October 15.

16,404. T. E. Caddy. Improvements in or relating to furnaces. October 15.

16,528. T. E. Wilson. Improvements in apparatus for evaporating, distilling, or concentrating liquids. October 17.

16,552. F. W. Scott, jun. Improvements relating to vacuum and evaporating pans. October 17.

16,557. W. P. Thompson.—From J. L. J. Vzn, Holland. Improvements in pumps for viscous, pulpy, or semi liquid substances. October 18.

#### COMPLETE SPECIFICATIONS ACCEPTED.\*

1889.

16,452. F. Hocking. Apparatus for evaporating, heating, cooling, and condensing. October 22.

19,103. G. H. Lloyd, S. S. Lloyd, jun., and W. S. Sutherland. Distributing and mixing the inflammable gases or vapours and air used in gas or blow-pipe welding, and apparatus therefor. October 15.

19,533. S. Alley. Rotative furnaces for roasting ores, &c. October 8.

19,906. W. H. Lonsdale. Cement for covering boilers, steam-pipes, &c. September 21.

1890.

10,856. A. Silverberg and C. Detering. Carbonising apparatus. October 8.

11,686. S. M. Lillie. Evaporating apparatus. October 1.

11,747. G. W. Haslam and P. Cornish, jun. Drying and desiccating wet substances. October 22.

12,260. A. W. Ellis. See Class XVII.

12,505. S. Seckendorf. Drying and pressing apparatus for granular and pasty matters. October 1.

13,930. H. E. Newton.—From W. J. Macdonagh and others. Appliance for injecting liquid and gaseous fuel into furnaces. October 8.

14,059. H. E. Hansen. Apparatus for complete evaporation of solutions. October 22.

## II.—FUEL, GAS, AND LIGHT.

### APPLICATIONS.

14,526. J. R. Shearer. A method of using the dead or heavy oils for illuminating and heating purposes. September 15.

14,618. W. B. Lampard. Improvements in apparatus for charging inclined gas retorts. September 16.

14,648. J. Balbirnie. Obtaining and applying natural gas. September 17.

14,649. J. Balbirnie. Obtaining and applying natural gas. September 17.

14,650. J. Balbirnie. Obtaining and applying natural gas. September 17.

14,651. J. Balbirnie. Obtaining and applying natural gas. September 17.

14,652. J. Balbirnie. Obtaining and applying natural gas. September 17.

14,720. J. Ruseoc. Improvements in apparatus for charging and drawing gas retorts. September 17.

14,737. D. Rylands and D. Johnson. Improvements in and in the manufacture of firelighters. September 18.

14,832. J. H. W. Stringfellow. Improvements in the treatment of hydrocarbons. September 19.

14,873. E. Sparshott and The Atlas Engine Co., Lim. Improvements in and relating to apparatus for the manufacture and storage of oil-gas. September 20.

14,906. C. W. Jones. Improvements in the revivification of materials employed for the purification of coal-gas, and in the treatment of the residues. September 20.

14,925. J. H. Parkinson. Improvements in the means of obtaining and separating oxygen and nitrogen from atmospheric air. September 22.

14,968. W. R. Lake.—From N. O. Goldsmith, United States. Improvements in and relating to apparatus for withdrawing coke from ovens, and for similar purposes. Complete Specification. September 22.

14,988. W. Smith. Improvements in the manufacture of artificial fuel. September 23.

15,008. H. H. Hazard. System for condensation, treatment, and utilisation of smoke, gases, fumes, and chemical products arising from furnaces and fireplaces of all kinds. September 23.

15,012. W. B. McClure, E. Corning, T. Hodgson, J. E. McWilliams, and J. W. White. Improvements in artificial fuel and in the process of making the same. Complete Specification. September 23.

15,158. J. H. Parkinson. Improvements in the means of obtaining and separating oxygen and nitrogen from atmospheric air. September 25.

15,379. J. Platt. Improvements in apparatus and appliances for obtaining more perfect combustion of fuel in the furnaces of steam generators. September 29.

15,388. J. F. Braidwood. An improved method of, and apparatus for, charging retorts for the manufacture of gas and other like purposes. September 29.

15,742. E. T. Ruthven-Murray. Improvements in machinery for charging and drawing gas retorts. October 4.

15,998. J. C. Lawson. Improvements in the manufacture of oxygen. October 9.

16,167. J. H. Hilton. Improvements in apparatus for carburetting gases. October 11.

16,207. F. Siemens. Improved means of utilising waste gases in regenerative gas furnaces. October 11.

16,213. A. Coze. Improvements in apparatus for charging inclined gas retorts. October 11.

16,410. T. W. Smith. Improvements in the manufacture of water-gases and of illuminating gas. October 16.

16,517. G. E. Davis and A. R. Davis. The purification of coal gas, and treatment of the by-products. October 17.

\* See Note \* on previous page.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

- 11,920. F. J. Jones. Production of coke. October 15.  
 15,947. J. H. Fergusson. Manufacture of illuminating gas. October 8.  
 16,009. J. Lawson. Oil-gas producing apparatus. October 22.  
 16,795. D. Dixon. Method and apparatus for generating gas. October 15.  
 17,131. J. Jones. Method and apparatus for burning fuel, debris, and water, consuming smoke, and utilising the products. October 22.  
 17,480. J. H. Wood. Artificial fuel. September 24.  
 17,535. F. B. Dortmund. Manufacturing coke. September 24.  
 17,945. J. Morley. Manufacture of firelighters. October 8.  
 17,954. T. D. Rock. Improvements in coking, and in coke ovens and plant. October 15.  
 18,543. J. E. Butterworth. Firelighters. October 8.  
 18,831. Kirkham, Hulett, and Chandler, Lim., and J. Chandler. Apparatus for separating tar and other impurities from gas. October 15.  
 19,121. W. G. Robinson. Apparatus for generating and burning gas from heavy oils or carbonaceous fluids. October 1.  
 20,031. W. Smith. Improved gas-lighting. October 22.  
 20,083. F. Siemens. Regenerative gas furnaces and gas-producers. October 22.  
 20,368. W. L. Wise.—From L. Senet. Coke ovens. October 22.

1890.

- 11,784. J. W. C. C. Schirm. Producing intense light by magnesium or other glowing matters. October 22.  
 12,036. I. Lilley and G. H. Hopkins. Manufacture of compressed fuel, and apparatus therefor. September 24.  
 12,860. J. T. Key. Process and apparatus for manufacturing gas. September 24.  
 13,033. F. Leonard and E. W. Harding. Manufacture of artificial fuel. September 24.  
 13,767. J. Gridley. Rendering wood highly inflammable. October 15.

## III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

## APPLICATION.

- 16,444. J. C. Hamilton. Improvements in obtaining substances of the class comprising phenol and cresol. October 16.

## IV.—COLOURING MATTERS AND DYES.

## APPLICATIONS.

- 14,620. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik, Germany. The manufacture and production of new substantive dyestuffs and of copper compounds thereof on the fibre. September 16.  
 14,621. B. Willeox.—From The Farbenfabriken vormals F. Bayer and Co., Germany. Improvements in the manufacture and production of rosaniline derivatives or leuco bodies of the tri- or di-phenyl-naphthyl-methane series and colouring matters therefrom. September 16.

11,836. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik, Germany. The manufacture and production of a sulpho-acid suitable for dyeing and printing. September 19.

15,120. O. Imray.—From The Society of Chemical Industry, Switzerland. A process for the manufacture of black colouring matters. September 21.

15,246. J. Wright. Improvements in black dyeing materials composed of mixtures of animal and vegetable fibre. September 26.

15,345. B. Willeox.—From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture and production of blue colouring matters or dyestuffs. September 27.

15,346. S. Pitt.—From L. Cassella and Co., Germany. The manufacture of azo-dyestuffs, and of materials for their production. September 27.

15,347. S. Pitt.—From L. Cassella and Co., Germany. The production of azo colours in substance or on the fibre. September 27.

15,674. J. R. Geigy. Production of brown colours which may be diazotised on fibre, and then combined with phenols or amines. October 3.

15,743. W. Davison. Improvements in colours. October 4.

15,778. M. Lange. New process of separating orthotoluid, sulphonic and paratoluid-sulphonic acids. October 6.

15,803. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik, Germany. The manufacture and production of a new naphthalene derivative suitable for use in the manufacture of dyestuffs. October 6.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

- 18,247. C. Romen. Rendering dry alizarine fit for colouring and printing purposes. September 24.  
 18,351. B. Willeox.—From The Farbenfabriken vormals F. Bayer and Co. Production of direct-dyeing yellow colouring matters. October 1.  
 18,517. B. Willeox.—From The Farbenfabriken vormals F. Bayer and Co. Production of azo-colouring matters. September 24.  
 18,519. B. Willeox.—From The Farbenfabriken vormals F. Bayer and Co. Production of direct-dyeing colouring matters. October 8.

20,219. J. Imray.—From La Société Anonyme des Matières Colorantes et Produits Chimiques de St. Denis. Manufacture of azo-oxyaniline, and of colouring matters therefrom. October 22.

20,292. J. Imray.—From La Société Anonyme des Matières Colorantes et Produits Chimiques de St. Denis. Production of a substituted metadiamine, and of colouring matter therefrom. October 22.

20,668. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik. Manufacture of colouring matters suitable for dyeing and printing. October 22.

20,678. O. Imray.—From The Farbenfabriken vormals Meister, Lucius, and Brüning. Production of compounds of the diphenylmethane group and the rosaniline series. October 22.

## V.—TEXTILES, COTTON, WOOL, SILK, Etc.

## APPLICATIONS.

15,170. G. Schuler. Improvements in the manufacture of linoleum, lincrusta, cere-cloth, and the like. September 25.

15,580. C. R. F. Schloesser and J. M. Campbell. Improvements in the manufacture of certain waterproof or water-repellant materials and fabrics. October 2.

15,758. M. Lister. Improvements in machinery for pulling and breaking silk waste, rabbits' down, fur, and other light fibre. October 6.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

17,137. E. Johnson.—From T. Barrows. Means and apparatus for ungumming, &c. flax and other like fibres. October 8.

1890.

8296. The Serrell Automatic Silk Reeling Company, Limited. Preparing cocoons for reeling. October 22.

10,856. A. Silverberg and C. Detering. Apparatus for carbonising wool. October 8.

## VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

## APPLICATIONS.

14,829. F. Walton. A process for bleaching ground cork. September 19.

14,839. O. Hoffmann. Process and appliances for washing, bleaching, and dyeing fibrous materials. September 19.

14,897. W. P. Thompson.—From F. Kornfeld, Austria. Improvements in dyeing, bleaching, washing, or otherwise treating cops of yarn or the like, and apparatus therefor. Complete Specification. September 20.

15,142. M. J. Norwood. Making stained or coloured made paper equal in appearance to stained glass, and rendering same clear by the use of turpentine, varnish and copal, terebene, pine oil, boiled oil, linseed oil, bicarbonate of potash, size, and dextrin. September 24.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

18,073. B. Willeox.—From The Farbenfabriken vormals F. Bayer and Co. Dyeing and printing animal and vegetable fibres with dinaphthyl-diquinhydrone or with betanaphthoquinone or tetraoxydinaphthyl. October 8.

18,115. A. Graeniger. Machines for dyeing, bleaching, &c. fibres. September 24.

19,408. C. Weber-Jacquel. Apparatus for dyeing, bleaching, &c. October 8.

19,863. C. Corron. Apparatus for dyeing and otherwise treating textile fabrics. October 15.

20,064. R. Holliday. Producing azo-colours on cotton or other vegetable fibres. October 1.

1890.

11,580. W. Marriott. Mordant for black dyeing. October 15.

12,900. T. Strahan. Colouring or decorating wall papers or coverings. October 8.

12,950. H. Thies and E. Herzig. Bleaching. September 24.

13,048. S. Pitt. From A. Smith and Son's Carpet Co. Dyeing apparatus. October 8.

## VII.—ACIDS, ALKALIS, AND SALTS.

## APPLICATIONS.

14,542. A. Boake. Improvements in the manufacture of phosphoric acid. September 16.

14,544. H. J. Allison.—From M. M. Monsanto, United States. Improved process for purifying brines. Complete Specification. September 16.

14,931. J. H. Parkinson. Improvements in the method of producing a porous mass of permanganate of potash. September 22.

15,159. J. H. Parkinson. Improvements in the method of producing a porous mass of permanganate of soda. September 25.

15,394. E. Richters. Process for utilising the silicic fluoride obtained by the treatment of certain phosphates with sulphuric acid. September 29.

15,416. R. D. Bowman. Improved process for the production of manganate of soda. September 29.

15,637. T. Goode and A. Smith. An improved process for recovering sulphur from alkali waste. October 2.

15,648. J. Leith. Improvements in the production of sulphhydrate of calcium to be used in the manufacture of alkali and sulphuretted hydrogen, and in apparatus therefor. Complete Specification. October 3.

15,942. J. A. Nettleton. Improving the process of vinegar making. October 8.

16,049. R. Haldan.—From the Viscount de Lambilly, France. New process for manufacturing alkaline or alkaline-earth cyanides through atmospheric nitrogen, and the application of the same to the production of ammonia. Complete Specification. October 9.

16,218. M. Netto. Improvements relating to the manufacture of hydrated oxide of iron. October 11.

16,233. H. C. Bull. Improvements in processes for the manufacture of sulphate of lead, and apparatus therefor. October 13.

16,460. D. Gamble.—From A. R. Péchiney, France. Improvements in the manufacture of chlorate of soda. October 16.

16,505. J. Beveridge. Improvements in the treatment of bisulphites, and in apparatus therefor. October 17.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

17,217. F. M. Lyte and J. G. Tatters. Recovery of chlorine from solutions of calcium chloride, magnesium chloride, the residual liquors of the ammonia-soda process, and other residues. October 8.

17,218. F. M. Lyte and J. G. Tatters. Production of hydrochloric acid from certain industrial residues, together with recovery of sulphur and oxide of magnesium. October 1.

17,888. F. M. Spence and D. D. Spence. Manufacture of alum. October 22.

18,356. E. Bowen. Manufacture of ammoniacal salts. October 22.

18,904. W. P. Thompson.—From E. de Lom de Berg. Manufacture of picric acid by crude phenols. October 1.

1890.

5488. W. Donald. Obtaining chlorine. October 1.

## VIII.—GLASS, POTTERY, AND EARTHENWARE.

## APPLICATIONS.

14,519. D. Chapman. Improvements relating to the decoration of china, earthenware or pottery and glass goods. September 15.

14,628. J. Sherwin. Improved devices for supporting earthenware while being fired. Complete Specification. September 16.

14,771. J. I. de Jongh. Glass tiles suitable for being used in conjunction with sheets of corrugated iron in the making of roofs and the like. September 18.

14,935. H. R. Lumley. An improved process and apparatus for producing glassware. September 22.

14,983. J. T. King.—From E. Jones and J. A. Jones, United States. Improvements in apparatus for the manufacture of glassware. Complete Specification. September 23.

15,197. H. C. Bull and P. Hargan. An improved process for the manufacture of pure silicates for use in glass making from common sand and pure metallic chlorides, the recovery of the hydrochloric acid arising therefrom, and apparatus therefor. September 25.

15,301. D. Rylands. Improvements in or applicable to machinery for the manufacture of bottles or other hollow glass ware or pressed ware. September 27.

15,468. J. B. Turnhurst. Decorating bisquit or unglazed china and earthenware or other similar articles or bodies. September 30.

15,753. W. B. Fitch. Improvements in the manufacture of glass bottles and similar articles. October 4.

16,112. W. Boulton. Improvements in machinery for manufacturing cups and other articles in pottery ware. Complete Specification. October 10.

16,131. J. W. Payne. A new or improved portable or fixed continuous firing kiln for burning and staining glass, china, tiles, enamels. October 11.

16,153. D. Rylands. Improved means of manufacturing glass conduits or tubes for electrical or other purposes. October 11.

16,369. D. Rylands. Improvements in machinery for the manufacture of glass bottles and other glass ware. October 15.

16,422. C. K. Mills.—From J. B. Granjon and F. Allemand, France. An improved process for moulding and firing hollow articles. October 15.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

16,696. F. R. Rostaing, L. A. Garchey, and N. E. Geille. Manufacture of vitreous material suitable for artificial marble, stained glass, &c. October 8.

18,951. W. J. Hague. Kilns for burning clay goods, furnaces, and like fires, and utilising heat from gas-producers in connexion therewith. October 1.

17,933. D. Rylands and J. W. Horner. Means for withdrawing molten glass or metal from furnaces, and controlling the outflow thereof; applicable for conveying liquid chemicals from larger to smaller vessels. October 15.

19,101. T. W. Gimson, T. W. Gimson, W. Kent, and T. Banks. Preparing the prints used for printing potters' ware, and apparatus therefor. October 22.

20,572. A. Griffiths. Manufacture of "swan neck" fireclay pipes. October 15.

1890.

13,222. S. Sambrook. Enamelling, hardening on, and fret kilns for earthenware and china manufacturers. October 8.

13,374. J. J. Barclay and J. Meikle. Kilns for drying and burning bricks and pottery. October 22.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

## APPLICATIONS.

14,775. A. Glover. An improved paving composition. September 18.

15,407. F. H. Rosher. Improvements in the process of, and apparatus for burning cements, plasters, and analogous materials. September 29.

15,678. W. A. Robinson and J. Y. Terry. Improvements in plastering compositions. Complete Specification. October 3.

15,782. J. F. Chiverall and A. Chiverall. An improvement in the manufacture and polishing and enamelling imitation stone, slate, marble, and wood. October 6.

15,784. G. A. Wright. An improved brick, block, tile, or slab for building and other purposes. Complete Specification. October 6.

15,900. L. Sterne. Improvements in the manufacture of cellular non-conducting blocks. Complete Specification. October 7.

15,940. H. J. T. Bennett. The use of peat moss and combinations therewith in the manufacture of building and paving blocks, roofing, and flooring felts, and card or mill boards. October 8.

16,231. P. Crean. An improved method of, and kiln for burning bricks. Complete Specification. October 13.

16,281. F. G. Edwards. Improvements in the use of concrete, cement, artificial stone, and similar materials for building purposes. October 13.

16,384. J. Dunn. See Class I.

16,426. E. Robbins. A new or improved manufacture of perforated or cellular constructed materials and other works, in plain monochromatic or decorative art work in "robings" for architectural, horticultural building, and for other purposes. October 15.

16,553. G. F. Redfern.—From W. Heller, Germany. Improvements relating to the treatment of gypsum for building, statuary, and other purposes. Complete Specification. October 17.

16,626. G. Brewer.—From G. Candiani, Italy. Improvements in the utilisation of the residue and waste of pyrites for the manufacture of bricks and other refractory material. October 18.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

15,737. W. T. Timewell. Manufacture of Portland cement from hard limestone. September 24.

16,327. G. B. Jerram. Paving for roads, paths, and floors. October 8.

17,623. R. E. B. Crompton and B. H. Jenkinson. Resistant coverings for battery room and laboratory floors. September 24.

1890.

3462. Baron A. von Solemacher. Manufacture of bricks, plates, and tiles. October 1.

11,443. A. Buecher. Manufacturing light bricks from gypsum, &c. October 1.



## X.—METALLURGY, MINING, Etc.

## APPLICATIONS.

14,565. S. O. Cowper-Coles. Improvements in the deposition of alloys. September 16.

14,646. C. A. Burghardt. An improvement in the manufacture of tin andterne plates. September 17.

14,658. J. F. Hall. Improvements in the manufacture of steel. September 17.

14,731. J. Broome and B. Edwards. Improvements in the means or apparatus for charging crude metal and scrap into open-hearth or other furnaces. September 18.

14,768. F. W. Martino and F. R. Martino. Certain new or improved metallic alloys. September 18.

14,781. G. G. M. Hardingham.—From M. A. Brogowski, Russia. Improvements in the manufacture of sodium and in the employment thereof for the production of aluminium, and in apparatus applicable for use in obtaining those products. September 18.

14,828. E. A. Cowper. A method of recovering fine particles of gold from pulverised mineral containing them. September 19.

14,837. C. E. Bernard. The manufacture and production of a dissolving and reducing agent and the employment of the same in coating metals with metals, or combinations with metalloids, extracting metals from their ores or combinations, and in other arts and manufactures in which dissolving or reducing action is required. Complete Specification. September 19.

14,846. W. H. Kennedy. Improvements in the method and apparatus for producing mineral wool. Complete Specification. September 19.

14,904. O. Murray.—From E. du Bois, Lukis, Mexico. A process and apparatus for extracting gold from crushed mineral containing it. September 20.

14,966. J. B. Hamay. Improvements in extracting precious metals from ores or minerals containing them, and apparatus therefor. September 22.

14,990. J. Muir. Improvements in the means or apparatus for the manufacture of iron or steel. September 23.

15,102. T. Sturgeon and T. P. C. Crampton. Improved method of casting metals in moulds. September 24.

15,110. Davies Bros. and Co., Limited, and E. A. Davies. Improvements in coating plain and corrugated metal sheets with metal or alloy, and in apparatus employed therein. September 24.

15,174. C. L. C. Bertou. A process for the recovery of the tin and the iron from waste pieces or clippings of tinned sheet or other iron or steel. September 25.

15,260. F. Braby. An improvement in the process of coating iron with other metals. September 26.

15,261. F. Braby. An improvement in the process of coating iron with other metals. September 26.

15,341. O. B. Peck. Improvements relating to the treatment of slag, and to the removal thereof from furnaces. Complete Specification. Filed September 27. Date applied for, March 3, 1890, being date of application in United States.

15,342. O. B. Peck. Improvements in apparatus for the treatment of slag. Complete Specification. Filed September 27. Date applied for, March 3, 1890, being date of application in United States.

15,343. O. B. Peck. Improvements in centrifugal apparatus for treating molten material. Complete Specification. Filed September 27. Date applied for, March 3, 1890, being date of application in United States.

15,348. O. B. Peck. Improvements relating to centrifugal apparatus for treating molten material. Complete Specification. Filed September 27. Date applied for, March 3, 1890, being date of application in United States.

15,349. O. B. Peck. Improvements relating to centrifugal apparatus for treating molten material, and to means for removing the said material from such apparatus. Complete Specification. Filed September 27. Date applied for, March 3, 1890, being date of application in United States.

15,366. L. Richards. Improved method for taking away the pigs from the casting beds of blast furnaces, and for breaking the same for sampling. September 29.

15,423. W. Wright. An improved method of reducing, blending, and welding metals, principally iron and steel. September 30.

15,433. S. Pearson and J. H. Pratt. Improvements in metallic alloys. September 30.

15,475. G. A. Decosse and V. Lespinats. An improved process of making all kinds of rolled iron. September 30.

15,506. G. H. Cannell. Improvements in the manufacture of armour plates. October 1.

15,520. H. B. Fletcher. Improvements in the process of annealing metallic castings. October 1.

15,551. A. J. Boulton.—From J. W. Bookwalter, United States. Improvements in converters. October 1.

15,582. J. Spence. Improvements in the manufacture of aluminium. October 2.

15,806. A. J. Campion and J. E. Tenison-Woods. Improvements in the treatment and reduction of metallic ores or compounds, and the recovery of metals and products therefrom. October 6.

15,830. J. T. King.—From T. S. Blair, jun., United States. Improvements in the reduction of ores. October 7.

15,833. E. Tweedy. Improvements in tempering fluid for treating steel. Complete Specification. October 7.

15,838. J. Hofmann. A process of and apparatus for making manganese bricks, and recovering sulphate of potash from "flue dust" resulting from the manufacture of ferro-manganese. October 7.

15,859. J. L. Hopper. Improvements in a process of and composition for softening and subduing refractory ores. Complete Specification. October 7.

16,060. L. Grabau. An improved method of obtaining metallic sodium. Complete Specification. October 9.

16,107. J. O. Day. Improvements in alloys and compositions for use in covering or coating surfaces of iron and steel and the surfaces of other materials, in order to prevent corrosion and fouling thereof when submerged. October 10.

16,344. R. S. Casson. Improvements in the manufacture of steel and ingot iron. October 14.

16,543. W. Pilkington. Improvements in manipulating and annealing metals. October 17.

16,545. H. H. Lake.—From O. B. Peck, United States. Improvements in apparatus for decomposing metallic salts and desulphurising ores. Complete Specification. October 17.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

12,181. E. Guillemin-Tarayre. Working, preparing, and concentrating auriferous alluvium. October 8.

15,301. W. H. Akester, W. T. Glover, and H. Edmunds. Manufacture of metals and alloys. October 1.

16,776. J. B. Alzugaray. Metallurgical dry process. September 24.

16,794. D. Dixon. Manufacture of aluminium and other metals. October 8.

17,699. C. Davy. Means for suspending and manipulating ingots or forgings. October 15.

17,878. J. Meese. Means and process for treating iron and other metals, and producing fireproof coating thereon. September 24.

17,890. A. Eyraud. Tempering, annealing, or treating castings or forgings of copper, nickel, cobalt and aluminium, or alloys. September 24.

- 17,933. D. Rylands and J. W. Horner. See Class VIII.  
 18,048. S. Pearson and J. H. Pratt. Alloys of iron and aluminium or steel and aluminium. October 1.  
 18,297. T. Haeghe and The Cape Copper Co., Lim. Process and apparatus for obtaining sulphate of copper and precious metals from copper or cupreous compounds. October 22.  
 19,333. W. Sellar. Method and apparatus for manufacture of iron or steel. October 22.  
 19,501. C. Rabache. Extraction or reduction of zinc. October 15.  
 19,865. E. P. Peyton. Cleaning sheets, &c., of wrought iron and utilising the waste products obtained, and apparatus therefor. October 15.  
 20,210. J. Hobson. Separating particles of iron or magnetic oxide from other substances. October 15.  
 20,586. J. H. Darby. Manufacture of steel or ingot iron. October 22.  
 20,677. W. L. Wise.—From J. B. Walker. Apparatus for separating or amalgamating finely-divided metalliferous material. October 15.

1890.

- 11,851. C. Rabache. Apparatus for extraction or reduction of zinc and other metals. October 15.  
 11,868. W. W. Keys. Process and furnace for treating copper and its alloys. Sept. 21.  
 12,553. I. A. F. Bang and M. C. A. Ruffin. Process and means for separation of tin from scrap and other metals. October 15.

## XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

### APPLICATIONS.

- 14,604. M. W. Dewey. Improvements relating to the heating of metals and other materials by electricity. Complete Specification. September 16.  
 14,624. A. S. Elmore. Improvements in the manufacture of metallic articles by electrolysis, and apparatus for that purpose. September 16.  
 14,693. J. P. Bayly.—From J. Silico, Mexico. Galvanic battery. September 17.  
 14,776. C. T. J. Vanlin. An improved process and means for the electro-deposition of metals. September 18.  
 15,054. S. O. Cowper-Coles. Improvements relating to the electro-deposition of metallic alloy. September 23.  
 15,114. G. C. Fricker and J. R. Bainton. Improvements in the manufacture of plates for secondary batteries, and in the construction of such batteries. September 24.  
 15,153. W. J. S. Barber-Starkey. Improvements in separators for galvanic batteries. September 25.  
 15,455. W. P. Thompson.—From C. L. Coffin, United States. Improvements in or appertaining to welding metals electrically. Complete Specification. September 30.  
 15,456. W. P. Thompson.—From C. L. Coffin, United States. Improvements in or appertaining to welding metals electrically. Complete Specification. September 30.  
 15,482. H. H. Lake.—From The Crosby Electric Co., United States. Improvements in and relating to electric batteries. Complete Specification. September 30.  
 15,640. E. Hancock and A. J. Markquand. Improvements in electric or secondary batteries. October 2.  
 16,005. W. J. S. Barber-Starkey. Improvements in separators for galvanic batteries. October 9.  
 16,054. H. G. C. Serrin. Improvements in electric batteries. October 9.  
 16,064. J. K. Pumpelly and F. Butterworth. Improvements in electrical secondary batteries. Complete Specification. October 10.

- 16,279. N. Slawianoff. Improvements in the process and apparatus for electrical casting of metals. October 13.  
 16,280. N. Slawianoff. Improved means of preventing defects, such as blow-holes and the like, in castings by electricity. October 13.  
 16,322. S. C. C. Currie. Improvements in insulating cells for electric batteries. Complete Specification. October 14.  
 16,346. S. C. C. Currie. The regulation and control of storage battery systems. Complete Specification. October 14.  
 16,399. A. J. Boulton.—From L. A. J. Joray, France. Improvements in or relating to the electro-deposition of metals. October 15.  
 16,415. M. W. Dewey. Improvements in methods of and apparatus for utilising electricity in the formation of sheet metal articles. Complete Specification. October 15.  
 16,522. G. D. Burton. Improvements in the methods of and apparatus for working metals by electricity. Complete Specification. October 17.  
 16,571. C. W. Kennedy and H. Groswith. Improvements in secondary batteries. Complete Specification. October 18.  
 16,603. C. J. Hall. Improvements in secondary batteries. October 18.

### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

- 15,963. P. Schoop. Manufacture of electrodes or plates for batteries. October 8.  
 17,205. A. S. Elmore. Obtaining zinc by electrolysis. October 8.  
 17,831. J. V. Sherrin. Electric battery carbon, and carbon compound elements. September 24.  
 17,974. H. H. Lake.—From H. Lemp. Electrical apparatus for welding, tempering, annealing, and treating metals. October 22.  
 18,350. J. H. Davies. Dynamo-electric machines. October 22.  
 18,755. A. Reckenzaun. Plates for secondary batteries. October 22.

1890.

8800. W. P. Thompson.—From I. Pearl. Electric accumulators. September 24.  
 12,628. H. F. Fowler. Welding metals by electricity. September 24.  
 13,003. D. Pepper, jun. Electrodes for primary and secondary batteries. October 15.  
 13,006. H. H. Lake.—From E. Thomson. Welding and otherwise working metals by electricity, and apparatus therefor. September 24.  
 13,013. D. Pepper, jun. Electrodes for primary and secondary batteries. October 1.  
 13,021. G. O. Rennerfelt. Electrolysis of melted haloid salts. October 15.  
 13,344. H. J. Allison.—From P. Hathaway. Galvanic batteries. October 1.  
 13,635. W. P. Thompson.—From C. L. Coffin. Electric welding. October 8.  
 13,636. W. P. Thompson.—From C. L. Coffin. Electric welding. October 8.  
 13,807. C. M. Reed. Battery compound. October 22.  
 13,905. M. W. Dewey. Welding metals by electricity. October 15.  
 13,906. M. W. Dewey. Welding and brazing metals by electricity. October 15.  
 14,604. M. W. Dewey. Heating metals by electricity. October 22.

## XII.—FATS, OILS, AND SOAP MANUFACTURE.

## APPLICATIONS.

14,697. J. B. Torres and J. D. Benjumea. Improvements in and connected with the clarification, purification, and refining of the greasy and oleaginous bodies. September 17.

15,404. W. Majert. Improvements in the manufacture of spermine. September 29.

15,817. C. Morfit. A new or improved process of refining linseed oil. October 7.

15,818. C. Morfit. Improvements in the refining of linseed oil. October 7.

16,025. W. H. Lever, J. D. Lever, and E. G. Scott. Improved plant or apparatus for extracting oil or grease from granular or pulverulent material. October 9.

16,059. C. A. O. Rosell and J. C. Pennie. Improvements in the manufacture of soap. Complete Specification. October 9.

16,579. W. Wright. An improved soap powder. October 18.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

17,870. R. Hunt. Treating crude cotton-seed oil, and oils containing resinous matter and free fatty acids, to obtain oil-soap and resinous colouring matter. October 8.

18,988. W. H. Lever, J. D. Lever, and E. G. Scott. Extracting oil or grease. October 22.

1890.

4607. C. Baker and W. E. Crane. Filtering oil. October 8.

13,985. G. P. Clark.—From D. R. S. Galbraith and T. Bell. Soap powders. October 15.

## XIII.—PAINTS, PIGMENTS, VARNISHES, AND RESINS.

## APPLICATIONS.

14,513. T. H. Cobley and W. Elmore. Improvements in the manufacture of white lead, meaning carbonate of lead and other compounds of lead, from lead, lead ore, and other substances containing lead. September 15.

15,126. J. P. Bayley.—From C. Neal and J. M. Douglas, United States. Paint compound. September 24.

15,564. J. P. Bayley.—From J. Baulch, United States. Improved blacking for leather. October 1.

15,614. A. Melville. Improvements in solid inks for writing, copying, drawing, &c. October 2.

15,857. C. M. Higgins. Printing or stamping inks. Complete Specification. October 7.

15,858. C. M. Higgins. Printing or stamping inks. Complete Specification. October 7.

15,899. H. H. Lake.—From H. Dutrioux, Belgium. An improved varnish. October 7.

15,903. N. K. Morris. Improvements relating to the manufacture of carbonate of lead, and to apparatus therefor. October 7.

16,017. W. Dyshko. Improvements in preparing and waterproofing skins for various purposes. Complete Specification. October 9.

16,023. J. Robson. Improvements in the method of protecting iron and steel surfaces from corrosion, and in coatings for the same. October 9.

16,093. R. W. E. Maelvor and W. Smith. Improvements in the production of "white lead" or basic carbonate of lead, and apparatus therefor. October 10.

16,321. S. Banner. Improvements in compositions applicable in the manufacture of oil cloth, varnish, pigment, menstrua, cement, and the like, and method of manufacturing the same. October 14.

## COMPLETE SPECIFICATION ACCEPTED.

1889.

18,642. C. Lahusen and C. Fenerlein. Separation of "Lanoresin" or gum resin from the waste waters of wool-washing and combing mills. September 24.

## XIV.—TANNING, LEATHER, GLUE, AND SIZE.

## APPLICATION.

14,943. H. Belcher. Improvements in the treatment of skins and hides for the preparation of leather. September 22.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

18,730. W. P. Thompson.—From C. Heepe. A substitute for leather or wood, and manufacture of same. October 1.

20,403. J. Hutchings and W. N. Hutchings. Method and apparatus for extracting tannin from bark, &c. October 22.

## XV.—AGRICULTURE AND MANURES.

## COMPLETE SPECIFICATION ACCEPTED.

1889.

18,677. W. B. Kemshead. Treating sewage and producing fertilising products therefrom. October 22.

## XVI.—SUGARS, STARCHES, GUMS, Etc.

## APPLICATIONS.

14,625. W. Schückher. A process and apparatus for the manufacture of nitrated starch. Complete Specification. September 16.

15,056. M. Mackay. Improved cementitious compounds. September 23.

15,620. S. Wohle. A process for the manufacture of dextrin. October 2.

## COMPLETE SPECIFICATIONS ACCEPTED.

1890.

4754. A. J. Boulton.—From L. Bon. Apparatus for drying sugar-cane trash. October 8.

12,635. H. H. Lake.—From L. Sterberg. Sugar refining. October 8.

14,625. W. Schückher. Process and apparatus for manufacture of nitrated starch. October 22.

## XVII.—BREWING, WINES, SPIRITS, Etc.

## APPLICATIONS.

15,352. H. H. Lake.—From A. Brunn, Denmark. Improvements in and relating to the manufacture of yeast. September 27.

15,790. E. R. Budden. Improved method of and apparatus for maturing wine and like beverages. October 6.

15,881. B. Dukes.—From J. Hradil, Germany. Improvements in the method of obtaining refined spirits of wine. October 7.

16,605. C. Pontifex. Improvements in spargers. October 18.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

16,079. C. Huelser.—From N. Reif. Cooling apparatus for brewing purposes. September 24.

17,434. J. White. Manufacture of degerminated, flaked, boiled, and torrefied maize for brewing and distilling purposes, and as a prepared food, and apparatus therefor. October 22.

19,003. H. T. Brown, G. H. Morris, and E. R. Moritz. Manufacture of beer. September 24.

1890.

4108. J. McKinless. Process and apparatus for maturing spirits and liquors. October 1.

12,260. A. W. Ellis. Distillation, and apparatus therefor. October 22.

13,257. G. F. Redfern.—From G. Perschied. Manufacture of wine and apparatus therefor. October 1.

13,428. H. H. Lake.—From The Handford-Stanford Co. Apparatus for cooling and aerating wort. October 8.

# XVIII.—CHEMISTRY OF FOODS, SANITARY CHEMISTRY, AND DISINFECTANTS.

## APPLICATIONS.

*A.—Chemistry of Foods.*

14,888. G. F. Meyer. Method and apparatus for extracting salt from victuals by osmose. Complete Specification. September 20.

15,854. F. T. Simson and J. Cox. Kola or caffeine or theine farinaceous and gluten food preparations. October 7.

15,981. W. Geddes. Improved malted food preparations. October 8.

16,260. J. Bowing. Improvements in the manufacture of wholemeal bread. October 13.

16,362. G. F. Redfern.—From E. Schramm and A. Chechong, Germany. Improvements relating to the preservation of bread and similar substances, and to apparatus therefor. Complete Specification. October 14.

16,508. G. W. Homer and J. Hazel. The manufacture of cream, and for the protection of cream, meat, fish, and other perishable articles from atmospheric air and other influences. October 17.

*B.—Sanitary Chemistry.*

14,858. R. Leigh. See Class C.

15,405. J. Hardwick and L. A. Newton. The treating of sewage and sewerage sludge. September 29.

*C.—Disinfectants.*

14,858. R. Leigh. Improvements in deodorising, disinfecting, and oxidising substances. September 20.

15,544. H. Peile. An improved preparation of disinfectants. October 1.

## COMPLETE SPECIFICATIONS ACCEPTED.

*A.—Chemistry of Foods.*

1889.

17,434. J. White. See Class XVII.

20,182. E. Sonstadt. Processes and apparatus for preparing an extract of tea, and utilising the by-products. October 22.

1890.

12,955. G. H. Hamrick. Manufacture and preservation of butter. October 8.

13,089. C. L. Bachelerie. Process and apparatus for preserving organic substances, especially alimentary substances. September 24.

*B.—Sanitary Chemistry.*

1889.

18,677. W. B. Kemshead. Treating sewage and sewage matters. October 22.

18,946. J. W. Calvert and J. Chaffer. Purifying the waste matters from tanneries, factories, dye-works, or other sources. October 8.

1890.

8239. W. S. West. Apparatus for treating sewage. October 8.

11,747. G. H. Haslam and P. Cornish, jun. Drying and desiccating sludge and manure. October 22.

*C.—Disinfectants.*

1889.

18,460. M. Hargreaves. Disinfectants and antiseptics, and disinfectant and antiseptic soaps. September 24.

1890.

13,046. W. P. Thompson.—From J. R. Bate and others. Preserving compounds for embalming purposes. September 24.

13,488. J. Y. Johnson.—From E. Salomon. Production of a new antiseptic. October 1.

## XIX.—PAPER, PASTEBOARD, Etc.

## APPLICATIONS.

14,586. J. L. Geddes and J. W. Johnston. Improvements in and relating to the manufacture of parchment paper. September 16.

15,432. J. Box. Improvements in paper making. September 30.

15,697. A. W. Hayles. The manufacture of paper and paper pulp from home and foreign grown marsh reeds, Danube reeds in particular. October 4.

15,776. F. Hawke. An improvement in the manufacture of copying paper. October 6.

15,868. C. R. Bonne.—From P. Hunaens, Germany. Improvements in the manufacture of celluloid articles. October 7.

15,930. C. Kellner. Improvements in lining boilers and digesters used in the manufacture of paper pulp, and for other similar purposes. October 8.

15,931. C. Kellner. Improvements in lining boilers or digesters used in the manufacture of paper pulp, and for other similar purposes. October 8.

15,995. F. V. L. Biorth. Improvements relating to apparatus for boiling wood pulp. Complete Specification. October 8.

16,408. E. Misch. Improvements in the manufacture of cellulose. October 15.

16,546. B. J. Beckmann. Improvements relating to the manufacture of paper, pasteboard, and the like, and to apparatus therefor. October 17.

## COMPLETE SPECIFICATIONS ACCEPTED.

1890.

12,139. W. Rogers. Treating paper for copying documents, and chemical compounds therefor. September 24.

12,375. W. M. Campbell-Callender. Substitute for bone or celluloid. October 1.

## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

## APPLICATIONS.

15,687. F. Valentiner. A process or processes for the manufacture of artificial musk. Complete Specification. October 3.

16,392. R. J. B. Mills.—From G. P. Monnier and Cartier, France. A receiver for the preservation and application of chloride of ethyl. October 15.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

18,131. G. Lugin. Treatment of disulphones for medicinal purposes. October 8.

18,990. W. H. Lever, J. D. Lever, and E. G. Scott. Manufacture of tetrachloride of carbon. October 1.

## XXI.—PHOTOGRAPHIC PROCESSES AND MATERIALS.

## APPLICATIONS.

11,681. T. A. Kennedy. Improvements connected with photography. September 17.

15,991. A. McDonald. An improved preparation of chemicals adapted to the development of photographic plates and the like. September 24.

16,238. H. H. Beale. The treatment of negatives in the art of photography by means of a liquid to be called "Non-actinic." Complete Specification. October 13.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

18,517. B. Jumeaux. Compound or developer for use in photography. September 24.

19,205. J. B. Payne.—From F. C. Brinus. Process for giving a matt surface to albuminised silver paper photographs or prints. October 8.

## XXII.—EXPLOSIVES, MATCHES, Etc.

## APPLICATIONS.

11,625. W. Schüchler. See Class XVI.

14,947. E. Davies. Improvements connected with explosive and non-explosive projectiles, and in an explosive compound to be used with the explosive projectiles. September 22.

15,001. W. Norres. Safety fuse for exploding petards. September 23.

15,183. H. H. Lake.—From H. S. Maxim, United States. Improvements relating to the manufacture of explosives, and to apparatus therefor. September 30.

16,002. J. S. Taylor and S. W. Challen. Improvements in processes for producing gunpowder cake. October 9.

16,075. C. O. Lundholm and F. Lyell. Improvements in the manufacture of explosives. October 10.

16,189. H. H. Lake.—From E. T. G. Thorn, G. R. O. Westendorp, and G. L. C. Pieper, Germany. Improvements in the manufacture of explosives. October 11.

16,342. A. Lucien. Improvements in fuses and slow matches. October 11.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

11,081. C. E. Kelway. Shell and torpedo fuses. October 22.

16,629. J. S. Taylor and S. W. Challen. Plant for preparing gunpowder carbon, using gaseous or liquid fuel. September 24.

1890.

7596. E. Turpin. Preparation and application of explosive substances, and apparatus connected therewith. October 1.

8843. E. Muller. Apparatus for nitrifying cotton, cellulose, straw, &c. September 24.

11,745. H. J. Haddan.—From J. H. Voelkel. Vacuum drying apparatus for explosives. October 1.

13,085. M. Meisgen and M. Hett. Fuse or priming machine. October 15.

11,625. W. Schüchler. See Class XVI.

## XXIII.—ANALYTICAL CHEMISTRY.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

19,669. R. M. Douglas. Improvements in Sykes' hydrometer. October 8.

20,755. M. Gehre. Process and apparatus for determining the amount of water in steam. October 15.

1890.

1840. E. Chameroy. Apparatus for weighing and measuring the density of bodies. October 8.

# THE JOURNAL OF THE Society of Chemical Industry:

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SESSION 1890—91.

Dec. 1st.—Mr. Wm. Webster. "The Electrical Treatment of Sewage," 1891.

Jan. 5th:—

Mr. Percy Gilchrist. "The Basic Copper Process."

Mr. W. C. Young. "On Standard Sperma Candles."

Feb. 2nd.—Dr. W. S. Squire. "The Artificial Production of Cold Theoretically and Practically considered."

Meeting held Monday, November 3rd, 1890.

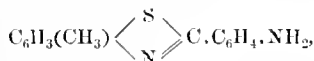
MR. THOS. TYRER IN THE CHAIR.

### ON THE DIAZOTYPE PROCESS OF PHOTOGRAPHIC DYEING AND PRINTING.

BY A. G. GREEN, C. F. CROSS, AND E. J. BEVAN.

THE subject of this paper is a new application of a group of colour compounds discovered by one of us now more than three years ago. This group, of which the dyestuff known as "primuline" is the typical representative (A. G. Green, this Journal, 1888, 179), is characterised by the special property of dyeing cotton and other textile fabrics from an alkaline or neutral bath in such a way that the colour compound, which is a bio-amido derivative, may be diazotised upon the fibre without in any way affecting the union with the fibre substance; and the diazo derivative may then be combined, also *in situ* with various phenols and amines, to produce colouring matters exhibiting a wide range of tone and shade. These colouring matters being thus synthesised within the fibre are distinguished by considerable resistance to alkaline detergents, and when thus produced are known as "Ingrain Colours."

Primuline itself is the sodium sulphionate of a complex amido base, which is obtained by heating para-toluidine with sulphur. Its formation is preceded by that of dehydrothiotoluidine, a body which appears to be an amidobenzenyl-amidothiocresol—



and primuline-base appears to result from a further condensation of this compound and to differ from it in the same way that dehydrothiotoluidine itself differs from para-toluidine: this view is expressed in the formula for primuline proposed by Gattermann:—



(A. G. Green, J. Chem. Soc., 1889, 227; Ber. 22, 968; P. Jacobsen, Ber. 22, 330; L. Gattermann, Ber. 22, 422; W. Pfitzinger and L. Gattermann, Ber. 22, 1063). Homologues of primuline closely similar to it in properties are similarly obtained from meta-xyldine and pseudo-cumidine.

The special photographic application of this group of colouring matters, which we are about to describe, depends in the first instance upon the sensitiveness to light of their diazo derivatives, this sensitiveness being increased in a very remarkable degree by combination with the complex colloids which constitute the textile fibres. Under these circumstances the diazo derivatives are completely decomposed by light, the product of decomposition being inert substances incapable of combining with amines or phenols. This fact, taken in conjunction with the special affinity of these bodies for textile fibres, give all the conditions necessary for the production of photographic pictures; the actual photographic agent being the diazo-primuline, in which form the image is produced, and the development of the image consisting in its conversion into any of the colouring matters obtainable by combination with phenols and amines. It is obvious that the pictures so produced are positives. As an illustration of the process we may take the production of coloured photographic designs upon cotton cloth.

The material is first dyed with primuline from a hot bath, to which some common salt may be added, by which it acquires a primrose-yellow colour. It is then washed and diazotised by being immersed for half a minute in a cold solution of sodium nitrite ( $\frac{1}{4}$  per cent.) which has been sharply acidified with hydrochloric, sulphuric, or other acid. The material is now washed in cold water and stretched out on a level white surface, and exposed to light beneath the object of which it is required to produce a positive reproduction. We may, of course, print from ordinary photographic positives, or from natural objects, or from any painted, printed, or photographed design or picture upon any sufficiently transparent material. Either sunlight or an artificial light of sufficient intensity may be employed, the time of exposure, of course, varying with the intensity of the light: in printing by daylight, the exposure varies from half a minute in bright sunshine to nearly half an hour in very dull weather.

When the decomposition of the diazo compound in the "high lights" of the picture is complete, which can easily be ascertained by means of a test slip exposed simultaneously, and touched with a solution of  $\beta$ -naphthol at intervals, these portions will be found to have changed from an orange to a pale yellow. The material is now either passed into the developing bath at once, or is kept in the dark until it is convenient to develop the image. The developing bath consists of a weak solution (about  $\frac{1}{4}$  per cent.) of a suitable phenol or amine, depending upon the colour in which it is required to produce the design. The following phenols and amines amongst others have been found to give good results:—

*For red.*—An alkaline solution of  $\beta$ -naphthol.

*For maroon.*—An alkaline solution of  $\beta$ -naphthol-disulphonic acid.

*For yellow.*—An alkaline solution of phenol.

*For orange.*—An alkaline solution of resorcin.

*For brown.*—A slightly alkaline solution of pyrogallol or a solution of phenylenediamine hydrochloride.

*For purple.*—A solution of  $\alpha$ -naphthylamine hydrochloride.

*For blue.*—A slightly acid solution of amido- $\beta$ -naphthol- $\beta$ -sulphonate of sodium (eikonogen or anglol).

If it is required to produce the design in two or more colours, the respective developers, suitably thickened with starch if necessary, may be applied locally by means of a brush or pad. After development, which with cotton is complete in less than a minute, the material is washed, and the picture requires no further fixing. In the case of the purple and blue developers it is necessary to wash the material finally in a very weak solution of tartaric acid.

Velveteen, linen, silk, wool, and other fabrics are treated in the same way, except that in the case of wool and silk a longer exposure to light is necessary, and the immersion in the nitrite and developing baths must also be more prolonged. The maroon and blue developers are not suitable for silk and wool.

Architectural and engineering drawings can be copied with clearness and accuracy upon paper or calico which has been dyed with primuline, diazotised, and dried. The diazo paper for this purpose can be kept in the dark for some time without undergoing decomposition. The copy is developed by brushing over the surface a solution of  $\beta$ -naphthol or  $\alpha$ -naphthylamine.

Transparent photographic pictures can be produced upon glass by coating it with a film of gelatin containing primuline, then diazotising with nitrous acid, printing, say from a carbon or silver positive, and developing as before. In all the above applications the primuline may be replaced by its higher homologues already mentioned; and for producing designs upon silk, dehydrothiotoluidine sulphonic acid and its homologues may be employed, which are equally sensitive to light.

Such is the process which we have devised and tested for a very considerable range of applications.

In point of simplicity, it certainly leaves nothing to be desired; and in regard to cost, for both material and time, offers considerable advantages over any known process of photographic printing.

It is hardly, perhaps, the place or time to enlarge upon the technical or aesthetic merits of the process. These it will have to vindicate by results, and in competition in a field where a very high order of perfection obtains.

We confine ourselves rather to that aspect of our subject from which it is to be regarded as a contribution to the progress of chemical science.

So far as we are aware, it is the first process which has been devised in which derivatives of the aromatic hydrocarbons are employed for the production of a *positive* image capable of development in colour. In this respect it differs altogether from that recently published by A. Feer. This process depends upon the sensitiveness to light of the diazo-sulphonates, produced by the action of the inorganic sulphites upon diazo compounds.

The action of the light consists in a dissociation of the diazo-sulphonate, the diazo group being set free. If, therefore, a mixture of such a compound with a phenol capable of reacting with the diazo derivative (to form a colouring matter) be exposed to light, synthetic union takes place between the diazo group and the phenol in proportion as the former is set free, and if, therefore, the exposure takes place as in the ordinary process of photographic printing, a coloured image or picture results. The product of the union being a colouring matter sufficiently insoluble to resist washing, the unaltered mixture of diazo-sulphonate and phenol being, on the other hand, freely soluble, the print is finally fixed by copious washing. By this method it is obvious that a *negative* is produced, and is fully *developed* in and by the printing process, whereas in the primuline process, the light acting as a destructive, not constructive agent, the exactly opposite result is produced, and the development of the image is a subsequent operation.

It is, perhaps, scarcely necessary to point out that *development* has not the same significance when used in relation to this process as in the ordinary methods of printing, e.g., with silver compounds. In the latter case, the picture may, in a sense, be said to be *evolved* by the developer, the intensity of the image being, to a certain extent, under the control of the operator; in the primuline process, on the other hand, the development is a strictly quantitative effect, determined by the amount of the diazo primuline on each portion of the printed surface. The intensity of a print is in this case simply determined by the weight of primuline per unit of surface. It is to be noted, however, that the diazotype process does also afford a means of intensifying the print. In cases when a developer is used, e.g.,  $\alpha$ -naphthylamine or phenylenediamine, which yields a colouring matter with a free amino group, the

product may be re-diazotised, and the diazo derivative again combined with an amine or phenol, thus obtaining a diazo compound of darker shade.

In comparing the diazotype process with the ordinary methods of photographic printing, the next question which arises is as to the mode of action of the light, considered (a) physically and (b) chemically.

(a) The rays which decompose the diazotised primuline are different in order and distribution from those which attack silver bromide. Photographs of the spectrum by the two processes taken side by side show, in the case of the diazo print, a sensible shifting of the area of maximum effect towards the red, and the diminution of activity is a very gradual one, probably extending beyond the visual limit of the red. We have, as might be expected, experienced some difficulty in carrying out this part of our researches, and we must content ourselves, pending their completion, with this very general indication of their results.

(b.) In regard to the chemical changes which accompany the "discharge" of the diazo primuline on exposure, the main point which we have established is that the diazo group ( $-N=N-$ ) is completely eliminated as free nitrogen. The evolution of gas is easily observed by allowing the light to act upon the sensitive surface submerged in water; and quantitative observations showed that the volume of gas evolved was in accordance with the requirements of the formula, on the hypothesis that the diazo nitrogen was split off. This result is confirmed by the observation that the product of decomposition is not reconverted into primuline nor into the corresponding hydrazine by treatment with sodium hyposulphite ("hyposulphite") or with stannous chloride. As to the constitution of the product, it remains undecided whether it is the phenol corresponding to the diazo primuline, the decomposition being analogous to that of diazobenzene by heat, or whether the primuline residue enters the molecule of the cellulose or other medium which carries it. The latter hypothesis is supported by the fact already mentioned that the molecular union of the diazo-primuline with the colloid medium is a necessary condition of the reaction, for the free diazo primuline when exposed to light in a thin film is either not decomposed at all or only after very prolonged exposure. The investigation of these problems is rendered very difficult by the complex character of the products, and again we must content ourselves, pending its completion, with a general indication of the results.

We hope shortly to be able to publish results which will more completely elucidate the mechanism of the reactions upon which the process depends.

In conclusion, we wish to express our indebtedness to Professor Norman Lockyer, F.R.S., for affording us valuable aid in the work of photographing the spectrum, and to the Autotype Company, through their manager, Mr. W. S. Bird, for the loan of some of their beautiful positives for printing specimens in illustration of the process.

#### DISCUSSION.

The CHAIRMAN said that one of the difficulties under which authors laboured who had many experiments to show, and who were thoughtful about them, was that they could scarcely think about their manuscript, it was a great advantage therefore that the members of the Society could rely on being able to read the paper for themselves in the Journal. In the present instance, however, it would be impossible for them to forget when doing so the more than eloquent appeals to their imagination and conviction which had been carried to their minds by the experiments which they had seen. The fabrics which had been dyed and finished were sufficient evidence that the new process of photography was one which would have very important effects on the future, at least of the textile industry. In decorative arts the process would have a very wide field of application, and he could not but congratulate the authors upon having applied their invention so successfully to the specimens before them. One could not but be reminded by this paper of the classical researches of Greville Williams on the cyanines, and it was a matter of scientific interest—which perhaps Mr. Green might see fit to refer to in his

reply upon the discussion—to determine the position which the diazotype process occupied in relation to those researches and to the ordinary processes of photography. Of course the two things were not on the same level; but there was a possible practical development of the process which they would do well not to overlook. Those who were interested in the duplication of machine drawing would have recognised in the admirable specimen shown, ample proof that the new process was applicable to that purpose in an equal degree to the well known ferrotype process.

Dr. R. BOURCAST said that he admired the process described by the authors exceedingly; it was very interesting, quite novel, and would, he thought, prove to be very useful. While in Mulhouse last autumn, his attention had been drawn to a process which appeared to him to be the reverse of that described by Mr. Green, the printed fabrics being quite devoid of colour when printed by night, and the colours being developed in a few seconds on exposing them to daylight. He had looked into the Journal of the Society of Mulhouse hoping to find a full description of that process, but without success. In September 1889, however, a note appeared in which the materials employed were described as diazosulphonic bases upon which phenols acted. It seemed that the fabric was prepared with phenols, and was then printed with a mixture of the base with nitrite of soda, hydrochloric acid and ice. But the fabric was colourless until exposed to the light. Paper printed in this way, when placed in the camera of a photographic apparatus, gave very sharp photos. The process now before them was entirely different. In that case they had a colouring matter, primuline, which when combined with phenols was capable of giving an immense variety of colours. The base, primuline, before combination with the phenols, was yellow, and its colour would fade very quickly in light; but it appeared to be protected from the destroying action of the light by the phenols. There were some other colouring matters which also faded very quickly on exposure to light. Some eosine exposed by him to sun rays was completely destroyed in 110 minutes, and in 3½ minutes a distinct decolourising effect was produced by the sun. He had heard of other colouring matters which were still more fugitive, quinoline blue, for instance. In the case of primuline, however, although it was a colour which faded in the light, it could, as he had said, be protected from fading quickly away through the light, by combination with phenols into diazo compounds, and it then became quite as fast as most aniline colours of the best kind; he even had noticed that azo colours were much less sensitive to the action of light than aniline or phenol colours. He had had experience of another substance which had the same properties. It was a compound used for obtaining positive copies of designs for engineering purposes. A sheet of white paper was painted with a mixture of perchloride of iron and tartaric acid. It was allowed to dry in darkness, and on then being exposed to the light with the Chinese ink drawing upon it, the colour faded except where it was protected by the ink. The design thus produced was then fixed by immersing the paper in a solution of gallic acid. He had used the process for producing drawings of such delicate objects as the wings of insects, and had found it to answer admirably. In conclusion, it appeared to him that primuline might be very usefully employed as a standard for measuring the amount of light obtainable in a room. For example, if an architect had to build a workshop in which a certain amount of light was required, by means of this primuline he might obtain a photograph which would prove whether or not he had satisfied the conditions required of him. In fact he had seldom listened to a paper of so novel and interesting a character, and so well given.

Dr. C. R. ALDER WRIGHT had listened with the greatest interest to about two-thirds of the valuable paper just read, and deeply regretted that he had missed the first portion of it. Perhaps, in consequence of that omission, there were one or two points which led him to inquire whether the last speaker was right in supposing that it was the primuline itself which was acted upon by light, or whether it was not the diazo compound thereof. He had gathered that it was

the diazotised primuline which was affected by light, and one proof of that would, he thought, have been afforded by the experiment, which the lantern did not allow them to see, in which the nitrogen was to be eliminated under the influence of light. As regarded the technical uses of the process, there could be no doubt that there existed a large number of ways in which such matters could be successfully employed. Notwithstanding which, the question occurred to him whether in this country the amount of actinic rays available during the year was sufficiently large to enable the process to compete with more sunny climates in an industry which required that particular sort of mechanism to bring about the necessary reactions. If some cheap form of chemical could be found which would produce the necessary alteration of the diazo compound, that might enable us to compete with other nations more favoured in the matter of sunlight. The process would then become more or less analogous to the old style of discharge process, the operations of which were effected by chemical reagents rather than by light. With regard to the use of inorganic substances for bringing about such reactions, he would point out that iron was not the only metal available. Several other metals were known—more expensive, perhaps—which, from experiments made with them, appeared to present at least the possibility of useful application in that direction. Some of the compounds of the rarer metals—for instance, tungsten, molybdenum, perhaps even vanadium—were capable of being acted upon by light, and when subjected to the action of developers of an organic character, were capable of giving prints somewhat similar to those given by the ferrotype process. Some experiments in that direction were at present being made in his own laboratory by Dr. Leon, who was unfortunately prevented by illness from attending that meeting and giving the Society some information on the subject so far as he had gone into it. The peculiarity that struck him most in this peculiar method of dyeing, from the chemical point of view, consisted in the complexity of the fundamental substance, primuline, if the gigantic formula written on the blackboard was to be regarded as representing its constitution. When the body became diazotised, it was the  $\text{NH}_2$  group which was affected, and not the other nitrogen atoms. He would like to ask, therefore, whether the authors possessed any information as to the nature of the chemical change which light produced on the diazotised body. He understood that nitrogen was evolved, and that suggested that the compound was analogous to the earlier-discovered diazotised bodies, which had not infrequently been the source of very uncomfortable explosions. The late Dr. Peter Griess had, he believed, been the victim of several accidents whilst investigating the properties of that class of compounds. He would like to ask, therefore, whether, if a large quantity of the material was being acted on, the evolution of nitrogen was likely to be so considerable and so sudden as to generate such a pressure as would lead to that violent expansion which in popular language was called an explosion.

Mr. JOHN SPILLER said that when, about two years ago, Mr. Green gave an account of his then newly-discovered base, primuline, he had but an imperfect idea of the alterable nature of that substance when exposed to light in a diazotised condition. That property had hitherto been regarded as an obstacle to the use of the substance as a colouring matter. But in the hands of Mr. Green it had become one of its prime qualifications, constituting it a photographic agent by the use of which they could obtain the effects of light exposure which had been exhibited to them that evening. He was especially interested in the production of the blue print which was before them, as it seemed to him that the capability of producing such prints would enable the process to compete directly with the ferro-prussiate process which was so much in vogue as a means of reproducing engineers' mechanical drawings. The use of Mr. Green's new material would certainly run that process very closely; and if the results obtained by it were as permanent as Mr. Green's experiments (and in a smaller measure his own) gave them every reason to expect, they would have not only a radically new process, but one possessing advantages which could not be overlooked in comparing it with other methods now in use.

Mr. T. BOLAS wished to point out that from a photographer's point of view one of the first questions that would arise with regard to the new process would be its permanency. He was formerly employed in a carbon printing works, and had had a great deal to do with the testing of pigments for use in the so-called carbon printing process. No doubt the members were aware that ordinary carbon prints were produced from a mixture of carbon as a base, toned down by certain pigments (generally indigo and alizarine lake containing an excess of iron or alumina). The ordinary method of testing a pigment for carbon printing consisted in exposing a print prepared from it in a high place and inclined a little to the south, so as to get the full benefit of the light. If at the end of so short a time as six months of summer light the lighter tones showed any signs of fading, the mixture was at once rejected. Mr. Spiller seemed to him to speak somewhat doubtfully of these prints in saying, if the results were as permanent as Mr. Green's experiments, and in a smaller measure his own, gave them reason to expect. With a view of testing their permanency, he had prepared a few prints by means of the primuline methods (including Feer's) some weeks ago, and had exposed them in a high place, intending to leave them there till the summer end of the season. But he happened to look at them two days after placing them out to the light, and was surprised to find indications of fading in the lighter tones. It would be interesting to know how far the prints before the meeting had been fairly tested for light, because from the photographer's point of view, a print which would fade by six months' exposure to summer light would be out of the question for practical use. When the matter was under discussion at a meeting of the Photographic Society, Mr. Spiller mentioned some tests which he had made, but he did not understand him to state what length of exposure he had given. It would be interesting to have some information on that point from him; and Mr. Green might possibly be able to add something also, though he feared that the process had not been known sufficiently long for thorough tests to be made. In the declining light of autumn, a few months' exposure was hardly equivalent to a few weeks in the early part of the summer. He had with him two prints that had been exposed for a week, one developed with  $\alpha$ -naphthylamine, and the other with orcin, which gives a colour very like that of resorcin. It would be seen that where the prints were not covered with paper the intensity had been very much reduced—so much that in the case of half-tone the lighter tints would be entirely gone.

Mr. A. H. ALLEN wished to know whether the diazotised primuline was insoluble in water, and the product of its decomposition by light soluble. If it were soluble in water, Mr. Green ought to be able to dissolve it out in a pure form, sufficiently pure, at any rate, to enable him to ascertain its nature. He would suggest that that should be done, if he was right in his supposition.

Mr. D. HOWARD wished to call the attention of the authors to a point which might be worth their consideration. He would suggest that they should ascertain how far the photographic power of their substance was isochromatic. If it were a true isochromatic agent, provided one could get a negative which would give even one print, it would be valuable, because it would be possible to reproduce further copies from that with other more permanent agents which did not themselves possess the advantage of being isochromatic. The progress already made in isochromatic photography encouraged one to follow up any possible lead in that direction.

Mr. JOHN SPILLER remarked that in his previous comments on the process before the meeting he had spoken only of the researches of Mr. Green, accidentally overlooking for the moment that Messrs. Cross and Bevan were associated with him in the matter. He should of course have spoken of the new method as the work of Messrs. Cross, Bevan, and Green.

Mr. W. E. FRIESE-GREEN said that since the authors had brought their discovery before the Photographic Society,

he had made some experiments on it, one of the results of which was to prove that the process was more sensitive if nitrite of silver were used instead of nitrite of sodium. With regard to spectrum work, he had found that the green band was acted on more than any other. He regarded the process as extremely valuable, but would like to see an improvement made in it in the direction of substituting a white background for the present yellow one. If that could be done the application of the process might be extended to the illustration of books and papers.

Mr. A. G. GREEN, in reply to the Chairman's inquiry as to whether there was any similarity in the constitution of the two bodies cyanine and primuline, said that there was very little resemblance. In a sense, of course, there was a connexion, cyanine being a quinoline derivative and primuline being distantly related to the quinoline series. He did not think that the sensitiveness to light was due to the primuline radicle itself, but rather that the primuline provided the means of fixing an amido compound on animal or vegetable fibres, and in that way enabled the diazo compound to become sensitive. His experience was that when so applied, or when in conjunction with gelatin, nearly all the diazo compounds were sensitive to light, however little they might possess that property when separate from the fibre or the gelatin. If one took some primuline, and smeared it over paper it would take hours to decompose completely; whereas if the same paper were dyed with it it could be decomposed by sunshine in one or two minutes. He was well acquainted with the process referred to by Dr. Bourcart. It was known as Feer's process, and was applicable to any diazo compound. But it gave a totally different reaction to that which he had described, because it was a constructive process, the azo colour being formed by the light, and not decomposed by the light as in the case of primuline. Dr. Wright had inquired whether it was the diazo compound of primuline or the primuline itself which was decomposed by light. He was quite right in supposing that it was the diazo compound which was affected. Primuline itself would stand exposure to sunlight, certainly for several days. It did change slightly by exposure to light, but so did most colouring matters. He had omitted to say that for practical purposes the electric light would have to be used, since they could not depend on daylight for continuous working. They had tried printing by it with considerable success, and with proper arrangements it would be much more convenient to use it, as the time required for exposure varied considerably. With regard to instability of diazo primuline to heat, the diazo compounds of high molecular weight were not very unstable, even when existing separately, and certainly not when in a very diffused condition on the fibre. The diazo compound of primuline could be boiled in water for an hour with scarcely any decomposition, whereas diazobenzene would decompose in water which was scarcely warm. Mr. Bolas had inquired about the stability of the compound to light. That was a point upon which they could not say much at present, for the simple reason that they had not had time to thoroughly test it. He hardly expected that diazo prints would be equal to carbon prints in that respect. At the same time he thought that the degree of stability would vary under different conditions, and that prints would be much faster to light on gelatin films than on cotton. The material printed upon was concerned in the reaction and greatly affected the results. It also made a difference to the stability whether the compound was or was not completely diazotised and developed. If the development were weak the colour would be unstable. In reply to Mr. Allen's inquiry about solubility, he would say that the ground substance was perfectly insoluble and could not be removed. They had tried to remove it so as to get a white ground, but had been unsuccessful so far. Though the yellow back was in many respects artistic, it would be useful to be able to have a white ground; and on silk they could nearly obtain it by using the sulphonic acid of dehydrothiotoindine, the diazo compound then leaving a nearly colourless background when decomposed by light. They had not tried any experiments in using primuline for obtaining isochromatic plates; but he believed that such experiments were being

made by one or two gentlemen. He was much interested by Mr. Friese-Green's statement that silver nitrite gave a more stable product than sodium nitrite. He would not have expected that the nitrite would make any difference, as an acid was present. But the reaction had been so little studied as yet that it was quite possible that many things would turn up with respect to it of which they were ignorant at present.

## THE PERMANENCY OF CREOSOTING AGENTS.

BY FRANKLIN S. CLARK, PH.D.

IN America, the processes of preserving wood have, up to the present time, been confined more especially to the treatment of timber exposed to the ravages of sea-worms.

The impregnation of railroad ties, such an important part of the work in European countries, is here a matter of secondary importance, since the easy access which American railroad companies have hitherto had to inexpensive wood has led to the replacement of ties in preference to the expensive methods incurred by an artificial treatment.

The present indications are, however, that many of the roads over whose lines the price of the timber has been enhanced by cutting, will gradually resort to some method of preservation.

The selection of process and principle in the American treatment is therefore primarily directed to the conditions necessary to protect timber from the attacks of sea-worms, so destructive to all wooden structures exposed in the waters of the South Atlantic, Gulf and Pacific coasts of the United States, as well as the sea-coasts farther south.

Experience has shown that wood that has been rendered invulnerable against the ordinary processes of decay will, in these exposures, fail, and that only by combining the most efficient process and preserving agent may the wood be expected to remain uninjured.

Notwithstanding the low price of timber and the comparatively high cost of the prepared material, American engineers have been compelled to resort to some methods of preserving sea structures exposed to the attacks of the teredo and limnoria.

Examinations made upon the various agents which had found favour in Europe for preserving wood has shown that the greater number of these are not adapted for the treatment of wood to be exposed in sea-waters.

Thus of the four more prominent agents of wood treatment literature, copper sulphate, zinc chloride, mercuric chloride, and dead oil, the latter alone has been extensively adopted in American practice.

Of the objections which have been offered against the metallic salts, none is so vital as that founded upon their ready solubility in water, the insolubility of the agent now being recognised as, perhaps, of more importance than the antiseptic power of the body itself. Consistent with this view the metallic salts are no longer used for the preservation of wood used in salt water, but in their place we find the oily products resulting from the destructive distillation of bodies capable of yielding creosote.

A reference may here be made to the ill-appropriated name creosote, as applied to the heavy fraction of coal tar. It has been stated that true creosote has never been used for the treatment of wood antiseptically. As will be seen from this communication, the oily products resulting from the destructive distillation of wood are extensively used in America for creosoting work, and as the oil is used and sold under the name wood creosote oil, or creosote oil, there is a possibility of rendering the name ambiguous in trade as well as in technical literature.

Fortunately, the heavy oil or creosote oil of the English works is in America more commonly called dead oil. Since chemistry has conferred the name creosote upon certain constituents of wood tar, it is but rational that this name should be applied to the oil containing the true creosote.

Wood creosote oil is the principal competitor of the dead oil of coal tar in the United States.

In Dr. Lunge's "Distillation of Coal Tar," we read, "For preserving wood, coal tar was recommended as early as 1799 by Le Bon, but for this purpose it is inferior to wood tar, which even now is exclusively used in shipbuilding. Wood tar penetrates more deeply into the pores of the wood, covers it with a coherent coat, and the preservative action of its phenols is assisted by its paraffin."

Dr. Ch. Heinzerling, while admitting the high price of the tar from wood, declares it to be uniformly the best tar for impregnating wood. (Die Conservirung des Holzes.)

The mechanical difficulties attending the use of wood tar in the creosoting cylinder have precluded its use on a large scale, but these difficulties are not encountered when, instead of the crude tar, we used the wood creosote oil resulting from the dry distillation of the long-leaved pine, *Pinus palustris*, of the South Atlantic and Gulf States.

A detailed description of the manufacture of this oil appeared in the School of Mines Quarterly for January 1888 (this Journal, 1888, 197), and only a general description will be necessary here.

The basis of the operations is the long-leaved pine now much used for constructive purposes in engineering, but more familiarly known as the source of the turpentine of commerce.

The tree as it stands in the forest is first tapped for turpentine; this operation extending over several years, or until no considerable quantity of the volatile oil is obtained, and the blazed surface is covered with rosin. Such trees are then abandoned by the turpentine producer, and as they stand in the forest become year by year more resinous. These trees are now in a condition suitable for the retorting process, and after felling and cording the wood is drawn to the river bank and by flats taken to the cities where the creosoting works are located.

At the present time there are in the United States three works at which timber is impregnated with wood creosote oil, but otherwise by the ordinary process of heat, vacuum, and pressure. These works, situated at Wilmington, N.C., Charleston, S.C., and Ferdinand, Flo., carry on the process of oil manufacture as well as treating timber. Work is done with dead oil if the contract calls for it.

The practice at the Wilmington works is representative of the industry as carried on in the United States and will be given here in brief.

The oil plant consists of 20 retorts having a charging capacity of four cords each. A cord is a pile of wood 4 ft. broad, 4 ft. high, and 8 ft. long. A bushel fills 1.2836 cub. ft. The apparatus may be of any of the ordinary types used by the wood distiller.

The products are collected in two portions, a light oil (and water) having a sp. gr. 0.875—0.975, and a heavier oil (with pyroligneous acid) or second running having a sp. gr. of 0.975—1.045. The latter is separated (by gravity) from the wood acid and held for further treatment. The light oil, after the separation of the water, is subjected to successive distillations with caustic soda solution, there resulting thereby a colourless hydrocarbon, which (under the name "palustrine") is used as a substitute for spirits of turpentine, and a residue of soda creosote which is added to the second running mentioned above.

The addition of the soda creosote to the heavier product of the retorts effects the neutralisation of the acetic acid contained in the latter to an important extent, and enriches the oil by the addition of creosote and other heavy bodies. Much objection has been urged against dead oil on account of the corrosive action of its phenols upon the lead sheathing of electric cables in cases where the conduits have been creosoted.

The wood creosote oil has the same effect if the acetic acid is not neutralised.

After the soda creosote treatment the oil is allowed to settle and the acetate of soda drawn off. The oil is now washed with water to remove any soluble matter, and after settling forms the wood creosote oil, which is the subject of this communication.

The acid resulting from the distillation is utilised in the manufacture of methylic alcohol and pyrolignite of iron.

The charcoal is used for fuel under the retorts, and the wood gas has found application in heating the steam boilers of the refining works. The charcoal is of too inferior quality to be used in smelting.

By utilising the by-products as described, the wood creosote oil may be made at a lower price than the dead oil is sold in the United States.

Analyses of the soda creosote, second running, and wood creosote oil gave the following percentages, by weight, of the different constituents at 50° F.:—

	Soda Creosote.	Second Running.	Wood Creosote Oil.
Water .....	4.05	3.57	2.79
Indifferent oil (distilling under 534° F.) .....	1.17	14.96	13.46
Crude creosote .....	19.38	14.42	17.00
Heavy oil (distilling above 534° F.) .....	42.42	22.35	26.55
Wax (paraffin resin) .....	14.88	27.61	26.55
Coke .....	9.24	6.87	5.78
Loss .....	8.56	10.23	8.75
	100.00	100.00	100.00

The second running examined was an average sample taken from 16 cords of wood in ordinary operation; the soda creosote and wood creosote oil were taken from the working tanks of the creosoting works. The analysis was made by distilling the oils to dryness in a cylindrical wrought-iron still (substantially the same as that represented in Dr. Lunge's "Distillation of Coal Tar") having a charging capacity of 9 gallons (231 cubic inches).

Temperatures were taken at every per cent. by volume of the distillate.

The indifferent oil is the light oil which remains after treating the fraction distilling under 534° F. with a 1.21 solution of caustic soda. It is unaffected by the latter. The crude creosote was separated by practically the same process as followed by Sir Frederick Abel with dead oil, the fractions below and above 534° F. both being treated with the caustic soda solution.

The loss is mostly due to a non-condensable gas, and in this respect the distillation differs markedly from that of dead oil of coal tar, the loss in the latter case being comparatively small. The loss in the wood creosote oil is due to the high temperature necessary to bring the heavy fractions over from the still, some of the oils being decomposed at the temperature of distillation.

Some idea of the uniformity of wood creosote oil may be obtained from oil produced under different conditions from that given above. The oil in question was obtained from South Carolina wood as distilled in a one-cord retort at the Charleston works and, not having received any addition of soda creosote, compared to the second running of the North Carolina oil. Determinations were made in a 50-gallon still, and the following results were obtained from an average of many analyses:—

	Per Cent. by Weight.
Water .....	3.97
Indifferent oil .....	15.53
Crude creosote .....	14.32
Heavy oil and wax .....	42.76
Coke .....	6.99
Loss .....	16.43
	100.00

The distillates at 600° F. were 45.48 and 37.40 per cent. by volume respectively. The above results show a considerable loss of heavy oil and wax (due probably to the still treatment), but otherwise the results are close when we

consider the different conditions under which the tests were made and the usual inaccuracies connected with this kind of analysis.

The creosoting plant proper, wherein the wood creosote oil described is used, does not differ essentially from the works where dead oil is exclusively used.

There are four creosoting cylinders, 66, 75, 88, and 100 feet long, the diameter of each being six feet. The process consists in submitting the wood to heat, vacuum, and oil pressure, each one of these steps requiring the most thorough execution, especially when green wood is under treatment. The first operation is but an artificial seasoning effected by passing superheated steam through closed pipes in the cylinder and thus drying the wood by a gradually applied radiant heat.

Simultaneous with the drying operation the vacuum pump is put on and kept at 26 inches of mercury until the wood is considered to be in a condition favourable to a thorough impregnation with oil.

The heat and vacuum process varies with the kind and condition of the wood to be treated, but is seldom kept up for a greater period than 24 hours. In this operation the albuminous constituents of the wood are coagulated and the moisture removed, partly in the vaporous and partly in the liquid condition. When the drying process is complete, the oil, heated to 120° F., is pumped into the cylinder, the vacuum pump running until the cylinder is charged to the finish. The pressure pump is now used to fill the remaining space and exert the pressure necessary for a complete impregnation.

One of the most important advantages of the creosoting process is in rendering the less durable sap woods more permanent, when exposed to deteriorating influences, than the best heart wood. The harder grades of wood need the treatment less and, in fact, are less perfectly treated. Practically the oil cannot be injected into heart wood.

The more sap wood in a piece of timber the better suited is the latter for creosoting purposes and the more likely to resist the sea-worms. The pecuniary saving resulting from the treatment of sap wood is well presented in Dr. Heinzerling's "Die Conservirung des Holzes." The wood more generally treated in the Southern States for piles to be used in salt water is the *Pinus taeda* or *loblolly* of the mills. A loblolly pile, 9 in. x 16 in. x 33 in. recently creosoted with dead oil, showed oil  $5\frac{1}{2}$  in. deep at the mid-section of the stick. A piece of good heart wood similar to the above would probably show oil not more than one-eighth of an inch. In considering the properties of the oils used in creosoting work it is well to examine the views now held regarding creosote oils. As already mentioned, antiseptic salts first tried, failed with timber exposed to sea-water. The cause of the failure is attributed to the solubility of the salts or their albuminoid compounds.

Dead oil soon replaced them and on account of its comparative insolubility has found extended application in marine work.

The use of dead oil, and especially the heavier grades of this oil, has been an acknowledgment, on the part of engineers, of the efficacy of the mechanical as distinguished from the chemical or physiological properties of wood-preserving agents.

The insoluble and non-volatile constituents of the oils exert a physical action in excluding air and water, and in retaining mechanically the bodies which would otherwise be dissolved. The principle here involved is exerting a strong influence upon the selection of dead oils at the present time, the heavy (non-volatile) insoluble oils meeting with the most favour.

The quantity of oil lost by volatility from creosoted timber is much greater than is commonly supposed, and since the oil which has been lost can be of no service to the timber when used, the question of evaporation of oil from treated timber becomes a matter of commercial importance.

The cost of creosoted timber depends, primarily, upon the quantity of oil put into the wood, and the value of the treated material depends upon the quantity of the valuable constituents which remain after the timber has been subjected to the usual conditions of volatility and solubility.

The element of volatility affects not only all treated timber used on land but also that which is destined for immersion in salt water.

Timber after coming from the cylinders is left exposed at the works, on the vessel or at the place where it is to be used. After it has been put down, the portion between high and low water is exposed to conditions favourable to the evaporation of the more volatile constituents.

The tests made on the volatility of the wood creosote oil and dead oil were carried on throughout by treating samples of *Pinus terda* which had been deprived of their moisture by heating in a (steam) dry kiln at a temperature of 180° F. for five days.

The first determination of loss by evaporation was made by a heated immersion of the wood samples in the respective oils, two samples of wood being used for each oil and all of them having been cut from the same stick so as to insure uniformity.

The four oils used were as follows:—

(1.) Wood creosote oil from working tank and a good average of the oil used in practice.

(2.) Dead oil from one of the New York dead oil companies. Represented as a heavy dead oil with a large percentage of naphthalene, and an average representing 500 barrel quantities.

(3.) Dead oil from the same company as (2.). Represented as a light oil with a small percentage of naphthalene. An average like (2.).

(4.) Dead oil from another New York company. Represented as an average as sold for creosoting work.

The above samples of dead oil, containing about 50 galls. each, were heated by means of a tight steam coil, and after melting and mixing to secure uniformity they were analysed under identically the same conditions as the wood creosote oil.

The percentages by weight at 50° F. were as follows:—

Number of sample.....	1.	2.	3.	4.
Water mechanically contained..	2.08	0.59	0.24	0.42
Water of decomposition.....	0.71	..	..	..
Indifferent oil .....	13.16	21.62	42.47	48.59
Crude creosote.....	17.00	..	..	..
Tar acids .....	..	9.72	15.34	16.63
Heavy oil.....	26.55	..	..	..
Wax (paraffin resin) .....	25.97	..	..	..
Solid bodies (naphthalene, &c.) .	..	67.50	40.23	27.77
Coke.....	5.78	0.28	1.49	6.42
Loss.....	8.75	0.29	0.23	0.26
	100.00	100.00	100.00	100.00

And by volume:

Distillate under 534° F. ....	26.00	77.00	61.00	78.00
Distillate under 600° F. ....	35.75	86.00	78.50	90.00
Residue at 600° F. ....	64.25	14.00	21.50	10.00

The high percentage of coke in (4) was due to an insufficient heat at the end of the distillation and decreases the percentage of solids for this sample.

The operation of treatment was carried out by immersing two prismatic blocks (as described) 4 in. × 4 in. × 13½ in. in an average sample of each oil. The data involved may be condensed as follows:—

Number of sample	1.	2.	3.	4.
Colour of oil .....	Black	Green	Green	Green.
Temperature of oil	135° F.	131° F.	134° F.	145° F.
Oil absorbed per cubic foot.....	11.87 lb.	11.93 lb.	11.96 lb.	11.62 lb.

Time of absorption, minutes .....	270	50	97	126
Superfices of two blocks, sq. in. ....	192	187	485	490
Volume of two blocks, cubic in.	422.9	18.6	115.3	422.9
Specific gravity of wood .....	0.619	0.618	0.581	0.656
Colour of treated wood .....	Black	Olive green	Light brown	Light brown

After removing the samples from the bath, and wiping off the excess of oil, they were exposed to evaporation in a sheltered room, but with free access of air. The samples were suspended side by side, and the weights taken daily during the first 10 weeks, and weekly thereafter. During the first 19 days each set lost weight, in consequence of the natural volatility of the oil, but after this date losses or gains were found to occur according as the temperature and humidity of the atmosphere varied.

The weekly course of evaporation at the end of 30 weeks showing:—

Number of sample.....	1.	2.	3.	4.
Percentage of loss of the oil absorbed	15.32	32.57	30.71	43.82
Ratio of loss by weight .....	1.00	2.12	2.00	2.88
Ratio of loss per unit of surface .....	1.00	2.13	2.01	2.81

respectively. Commercially speaking, these figures are very significant. From them it is seen that in order to have 12 lb. of oil per cubic foot remaining in the timber after 30 weeks of similar exposure, the quantity of oil which must be injected will be, in the place of 12 lb., the following quantities:—

Number of oil.....	1.	2.	3.	4.
Lb. per cubic foot.....	14.17	17.80	17.32	21.35

Not an inconsiderable quantity when it is considered that the quantity of oil injected practically determines the cost of the treated timber.

The preceding determinations were supplemented by others carried out under the conditions involved in the usual creosoting process—heat, vacuum, and oil pressure.

The vacuum was carried to 27 inches of mercury and the pressure to 200 lb. per square inch or less as the oil treated with required.

The samples, as before, were obtained from several of the New York dead oil companies, and were prepared in the manner already described. The wood creosote oil was the same as that already used. The three dead oil samples, numbered here 6, 7, and 8, showed the following percentages, by weight, at 50° F. when analysed in the same manner as the preceding samples:—

Number .....	6.	7.	8.
Water.....	0.43	1.31	1.90
Indifferent oil .....	47.51	18.60	31.68
Tar acids .....	16.39	9.30	6.03
Solid bodies (naphthalene) .	32.83	69.03	58.80
Coke .....	1.16	1.57	1.44
Loss .....	1.08	3.19	0.47
	100.00	100.00	100.00

And by volume:

Distillate under 534° F. ....	68.00	85.00	39.00
Distillate under 600° F. ....	91.66	92.00	52.00
Residue at 600° F. ....	9.00	8.00	48.00



The data in this case was :

Number of sample .....	5.	6.	7.	8.
Oil injected per cubic foot .....	11'3	10'8	10'0	10'6
Superfices of each two blocks ...	496	496	496	496
Volume of each two blocks.....	432	432	432	432
Specific gravity of the wood ....	0'619	0'605	0'567	0'588

These samples were exposed to a somewhat higher (summer) temperature than the preceding, but otherwise in the same manner. After an exposure of eleven weeks the ratio of loss calculated to the unit of surface was found to be as follows :—

Number of sample .....	5.	6.	7.	8.
Ratio of loss .....	1'00	2'38	2'64	1'62

the wood creosote oil again being taken as unity on account of showing the least loss.

The relative volatility of the corresponding constituents of the wood creosote oil and dead oil was obtained by injecting samples of wood with each constituent and determining the loss in weight from week to week. The samples of wood taken were the same in size, and the exposure was simultaneous with that of the crude oils themselves.

The actual losses in ounces after 11 weeks of exposure are as follows :—

No. of Oil.	Constituent used.	Specific Gravity of Wood.	Oil Injected.	Ratio of Loss Per Unit Surface.
9.	Indifferent oil, dead oil	0'560	8'2	6'30
10.	Indifferent oil, wood oil	0'626	7'6	7'44
11.	Refined phenols.....	0'647	10'6	8'36
12.	Refined creosote .....	0'611	10'9	6'41
13.	Crude phenols .....	0'592	11'2	7'41
14.	Crude creosote .....	0'620	11'3	3'35
15.	Naphthalene.....	0'642	9'7	6'44
16.	Paraffin resin .....	0'544	11'6	1'00
17.	Heavy oil .....	0'600	9'5	1'62

Further comparison on the volatility of the crude oils and their constituents were made by exposing equal weights of each simultaneously in open vessels, the surface of exposure being the same in all cases. Such an exposure, depending as it does upon the viscosity of the oil and other considerations, does not represent the conditions at play in practice, but confirms the general results which would be indicated by the analysis of the oils and which were found to be true on exposing the treated blocks.

In comparing the crude oils the wood creosote oil is taken as unity, and for the constituents the wax or paraffin resin is similarly taken. The ratio of loss was found to be :—

No. of oil .....	5.	6.	7.	8.	9.
Ratio.....	1'00	1'84	2'01	1'09	8'80

No. of oil .....	10.	13.	14.	15.	16.	17.
Ratio.....	47'53	15'73	9'26	15'66	1'00	6'00

The absolute percentage of loss by such an exposure of 12 weeks was found to be 12'1 and 1'5 for the wood creosote oil and paraffin resin respectively.

By an examination of the daily variation in weight of the four wood samples, 1, 2, 3, and 4, treated by simple immersion, it was found that the dead oils not only showed

the losses referred to, but also were found to have sustained the greatest gains during days of great humidity. These results, found by comparing the variation for each day with the temperature and relative humidity for that day, were found to be quite uniform. This was further confirmed by exposing the four samples in the open air to the action of rain, dew, and sun-heat. This greater susceptibility to moisture, on account of evaporation, offers greater opportunities for the entrance of the germs of destruction, and thus in part destroys one of the main purposes of the creosoting process—the rendering of wood impervious to air and water. In considering the data here presented it is obviously necessary to compare the losses by evaporation in each constituent with the quantity of that constituent in the crude oil as shown by the analysis.

No absolutely uniform relationship of the evaporative tendency in the different samples could be looked for, since there are probably modifying circumstances at work in the case of the volatility of semi-solid bodies in wood which would not come into play in ordinary cases of evaporation.

In the case of the comparative solubilities of these oils, which the writer is at present investigating, these disturbing conditions again come into play and considerable light has yet to be thrown upon the subject.

Comparative tests as to the immunity of these oils and their constituents from the sea-worms are now being made by exposing the samples here described at places on the North Carolina coast, where ordinary timber is destroyed very quickly. The samples exposed were those treated by immersion (1 to 4), those treated by pressure (5 to 17), and another complete series of crude oils and constituents which have not been exposed to evaporation.

Works, in which timber treated with wood creosote oil is exposed, are to be found on the Atlantic, Gulf and Pacific coasts of the United States, and at the eastern terminus of the Panama, and of the Nicaragua canal.

The oldest authentic record of timber treated with this oil and exposed where the teredo and limoria rapidly destroy untreated timber, is six years. The writer has recently examined a locality at the mouth of the Cape Fear river, where piles treated with wood creosote oil have withstood the attacks of the worms for five years. The latter is three miles from sea, and so infested with the worm that common one-inch plank is destroyed in three months and unbarked piles in two years. It is considered here that the wharves, though not sustaining any great weight, need to have the piles removed every two years. In one case, two piles treated with wood creosote, and standing close together, after having been driven five years are still perfectly sound. The uncreosoted tide gauge nailed to the piles has long since been cut off to high-water mark.

The wharf used for the United States Custom house was built of both creosoted (about 50 in number) and uncreosoted piles. All of the former examined by the writer were sound, while the untreated piles, in every case, showed the presence of the worm.

That timber treated with the wood product has been entered by the worm is an unfortunate fact, though the writer has never found either the teredo or limoria in any of the timber he has examined. Samples sent to the laboratory and showing evidence of the worm have, as in similar cases with dead oil, been evidence of the efficacy of the oil, for in all such cases the injured wood was nearly white and showed the absence of oil, while pieces taken at the same time from adjacent piles were uninjured.

The failures of creosoted timber in America (whichever oil may have been used) is for the most part traceable to the use of heart wood or wood directly from the river.

An insufficient quantity of oil is also a great source of failure.

Time has not yet allowed of a comparison of the relative values of the two oils considered here, but from the writer's observations the advantages of each may here be condensed.

The dead oil shows a smaller percentage of water, a larger percentage of solidifiable constituents, and is handled more easily in the creosoting cylinder.

The wood creosote oil is free from ammonia (which is considered to hasten decay), is more uniform in its

constitution and less volatile and soluble when exposed to air and water.

The efficacy of the latter oil has been well established where used, but the comparative values of the two oils can be determined only by time.

#### DISCUSSION.

The CHAIRMAN said that it was always gratifying to receive communications from members not in direct and immediate contact with the home sections of the Society. It afforded an additional proof of the interest which the British Society of Chemical Industry created among technologists elsewhere, and especially across the Atlantic. A considerable proportion of the members of the Society were in America, and it was a great compliment to the Society that so enterprising a people should value the Society's Journal so highly. With regard to the communication itself, it was to be regretted that they had not before them the tables referred to. He was personally sorry for that, because he wanted to ask some questions relating to them. He assumed that the conclusion drawn by the author from his comparison of wood creosote with coal-tar creosote was in favour of the former; but whether he had substantiated that conclusion was another question. The scientific interest of the comparison was very considerable, but it had not the same practical interest in this country, because we had not here the facilities for getting the wood creosote so cheaply. In that connexion he hoped that Mr. Clark had made comparisons between the life of wood preserved by creosote and by other methods now in vogue. In a book published as long ago as 1875 by Messrs. Spon, Mr. Britton, a gentleman in the Surveyor's Department of the Metropolitan Board of Works, had attempted to cover the whole field of the preservation of timber, especially from dry rot. If his memory served him, preference was given to certain metallic salts, and chiefly to perchloride of mercury. A process for such treatment of timber, patented by Kyan, was at one time very successful, but was ultimately dropped on account of expense. But it was remarkable that in connexion with the very purpose referred to by the author of the communication, namely, the preservation of timber exposed to the action of water, there had been a very noticeable revival of the employment of metallic salts of various kinds. The subject was therefore again occupying the attention of constructive engineers; and if Mr. Clark's comparisons included not only wood creosote and coal-tar creosote but metallic salts also, they would be especially interesting and valuable, if only by reason of their being brought down to a much more recent date than any hitherto existing.

Mr. A. H. ALLEN said that an exceedingly able—indeed classical—paper on the subject of creosoting timber had been read before the Institute of Civil Engineers a few years ago by Mr. S. B. Boulton, and that paper exhausted the subject up to its date. He agreed with Mr. Boulton that the use of phenoloid bodies, such as existed in wood-tar or creosote from other sources, was not all that was required, because they were not insoluble, and were, therefore, apt to be washed out in the course of time. He thought that much better results were to be obtained by absolutely filling the pores of timber with insoluble substances. That was the reason for using the thick coal-tar oils rich in naphthalene which were so extensively employed at present. He himself was the co-inventor of a process for creosoting timber, which never came to maturity, because on inquiry he found that there was no real demand for such processes. Engineers were quite willing to use the process, even on a large scale, but only at the inventor's expense; and they required from 7 to 10 years to form an opinion of its merits. Timber exposed between wind and water deteriorated very rapidly, and in that case they had to deal with worms, not dry rot. The *Teredo navalis* was a worm with a saw-like arrangement at one end, by means of which he tore the timber into sawdust and passed it through his body. No doubt he possessed digestive organs of some sort, but he (Mr. Allen) was informed on high authority that the main cavity in the *Teredo* was not a digestive canal of the ordinary kind, being apparently used chiefly as a convenient means of passing the sawdust out at the other end. They were thus dealing with conditions very different from those which

ordinarily existed. If one could find a substance which would give the *Teredo* chronic indigestion, that would be the most valuable agent to employ. If the agent used were soluble, it was washed out by the water; if completely insoluble, it had no effect on the *Teredo*. So at present they were on the horns of a dilemma. It was very difficult to improve on the processes at present in use, because engineers were unwilling to go to any expense in testing new methods. In fact, they would very much rather replace timber as occasion required than treat it by any process which had to be paid for.

Dr. C. R. ALDER WRIGHT had begun to hope, from some remarks made by the editor in reading the communication that the products of the distillation of the *Pinus sylvestris* were going to be instrumental in abolishing some of the evils that arose from the consumption of alcohol. They were told by Dr. Ward Richardson that methylic alcohol might be taken with impunity as compared with the ethylic fluid; and from the glowing description of the wood alcohol on the table he was in hopes that if it were obtainable in sufficient quantity and of uniformly good quality, it might become a most important social agent. But a taste of the sample had dissipated the anticipations he had formed. It had a most diabolical flavour, and he did not envy the man who should acquire a taste for it. With regard to the variety of products obtainable from the *Pinus sylvestris*, he thought that they merited very considerable attention from British wood distillers, as showing them that woods other than those they usually dealt with yielded products of very great commercial importance. From that point of view, if from no other, the paper would be very valuable to the Society.

Mr. WATSON SMITH said, with regard to the evaporating powers of dead oil and wood creosote respectively, Mr. Clark had given some data which showed that the former evaporated more per unit of surface than did the latter. That was quite natural, and was due to the fact that naphthalene, which was largely contained in coal-tar dead oil, was a body which, though possessing a high boiling point, evaporated or volatilised at very low temperatures. On the other hand, wood creosote would contain such bodies as guaiacol and creosol, and those bodies, while having high boiling points, were not so peculiarly volatile at comparatively low temperatures. On the other hand, naphthalene was thus volatile. To enforce the argument as to volatility, he had only to remind them that naphthalene had been used to adulterate camphor, a substance of eminent volatility. Then again, the guaiacol and creosol in wood creosote were associated with paraffin, a substance which had a still higher boiling point than naphthalene, but which was practically non-volatile at ordinary temperatures. All these circumstances would tend to account for the superior action of wood creosote as regarded evaporation. The author appeared to have tried many experiments with antiseptic salts, but found that they did not answer, as they were too easily washed out of the wood. Dr. C. R. Alder Wright had referred to the unpalatable flavour of the wood naphtha; that might be due to a small quantity of terpenes, which whilst efficiently denaturing the alcohol, could not affect it injuriously for the varnish-maker, to refer to one industry only.

Dr. FRANKLIN S. CLARK sent the following reply:—

The views expressed by the Chairman, Mr. Thomas Tyrer, in reference to the metallic salts, so far as their antiseptic properties are concerned, agree, I believe, with the experience of preserving wood not exposed to water. The trial of zinc chloride for preserving railroad ties on the German railroads is favourably reported upon, and may be cited as an instance corroborating the above-mentioned views. Where wood is much exposed to the solvent action of water, as in the case of marine works, the salts are quickly washed out and their antiseptic power has but a short time to exert itself. No instance has come under my notice where these salts have preserved wood against the sea-worms in American waters.

The views presented by Mr. Allen bring up the question of the value of the antiseptic action of different agents compared to their mechanical effect. This is a very important phase of the subject, but was not taken up in the paper, the latter aiming to present the mechanical action of the two

oils when injected into wood. The antiseptic action of the constituents of these oils is certainly an important consideration in creosoting, but it is still an open question among the coal tar authorities how much the preservation of wood is due to the germicidal and how much to the physical action of the agents used. The comparative values of the two oils can only be determined by actual trial under similar conditions.

The high boiling point of naphthalene does not indicate its permanency under atmospheric temperatures, as will be shown by the comparative determinations of the paper. The relative degrees of evaporation of water and oil of turpentine at ordinary temperatures shows that the boiling point alone is not a criterion of permanency.

As to the solubility of the comparative constituents of the two oils, it is found that the heavy oil and paraffin resin, the constituents of the wood oil corresponding to the naphthalene of dead oil, are, like naphthalene, practically insoluble in water.

The comparative solubility of the phenoloid constituents of each is somewhat indicated by examining the solubilities of carbolic acid, cresylic acid, &c., compared to guaiacol, cresol, &c., the constituents of wood creosote. This comparison would lead to the belief that the latter were less liable to wash out from treated wood.

The germicidal properties of these several constituents would throw much light upon their values for preserving wood, but these investigations have never yet been made.

The caustic taste of the wood alcohol, referred to by Dr. Alder Wright, is traced to the crude wood acid which forms the basis of the manufacture of wood alcohol.

In the distillation of hard wood, the oily products are very viscous and heavy, which fact explains the ready separation of acid and tar and the slight tendency of the acid to take the oily bodies in solution. In the case of the distillation of pine wood, however, the presence of turpentine-like bodies causes the specific gravity of the acid and oil to nearly approach each other, causing thereby, to a greater or less extent, a solution of some of the oily components of the tar. These constituents are difficult to remove from the acid by any commercial treatment, and give a biting taste to the resulting methyl alcohol.

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Notices of Papers and Communications for the Meetings to be sent to the Local Secretary.

*Meeting held on Wednesday, November 5th, 1890.*

MR. A. NORMAN TATE IN THE CHAIR.

OPENING ADDRESS BY THE CHAIRMAN, MR. A. NORMAN TATE, ON SOME AIDS TO THE FURTHER DEVELOPMENT OF CHEMICAL INDUSTRY.

(ABSTRACT.)

AMONGST other aids to which we may now look to assist in the development of chemical industry are those that may be termed educational, and the present movement towards technical education, which has been largely promoted by the passing of the Technical Instruction Act, ushers in a new

era of national educational effort that must greatly aid all our industries. Whether the Society as a society should take any definite action in this matter is a subject for consideration, but there are many ways in which individual members can assist.

There are two modes of teaching in connexion with technical instruction, viz., hand work (of course including all such manipulations as chemical laboratory work), and oral teaching, and we have to be careful that there shall not be an excess of oral teaching, lest we perpetuate under the name of technical education some of the worst evils of ordinary instruction as now too often given.

We must once for all clearly understand that there is no possibility of teaching trades or manufactures except in the actual workshop or manufactory, and that any attempt to teach them in schools, colleges, or other teaching institutions must end in failure. And I would go further to say that no profession can be thoroughly well learnt without training amongst those who practice it.

It has often been said lately that what is really wanted is that all-round training of eye, hand, and general faculties that shall give facility in observation and manipulation in various directions, and the introduction of manual work into our board and other elementary schools must help greatly in this direction. And the introduction into such schools of the teaching of the elements of science should give that knowledge of general facts that underlies all industrial occupations, provided that it does not run in the direction of much of our present school teaching—mere statements of scientific facts, without ample accompaniment of practical illustration of their applications. It is desirable that similar teaching should be given in what are considered our higher class elementary schools, many of which are woefully deficient in scientific and practical training.

But, of course, all this early training is of a general character, affecting all occupations alike. It is when the pupil leaves ordinary school studies that more specific instruction should commence. Any instruction of this kind must be given to our working classes either when continuing partly at school or in evening classes. Now we have a large number of science and art schools and classes, and in many places good classes under the City and Guilds of London Institution, and these have already done immense service, but there is required more definite organisation—something on the lines of "organised science schools" mentioned in the Science and Art Directory for this year, at page 34, but with that curriculum modified to suit special places and circumstances.

There must follow a distinct advantage in working such schools somewhat on the lines of the Science and Art Department, inasmuch as it has shown itself so thoroughly in earnest in insisting upon practical teaching as against "cramming," and although it has had largely to rely upon written answers to questions to assess results, yet its system of examination has gradually become a really good test of sound practical teaching, and in many subjects its questions are models of searching investigation into the *practical* knowledge of the pupil as against ordinary examination "cram." And in organising modes of instruction it has insisted upon really efficient laboratory instruction, not only in chemistry but in physics, biology, and other branches of science, and has, by its careful inspections, largely prevented "slipshod" work.

It is desirable that the attention of workpeople should be more directed to such schools. The majority of students now enter them with the object of gaining some intellectual, pleasant occupation for their leisure time, and not because they think they will gain knowledge that will assist them in their ordinary avocations; and we may all do more than we are doing in calling attention to these schools and helping them by all means in our power.

In these evening schools the ordinary school course must be followed up by more advanced teaching, which, as regards chemical industries, does not mean teaching in chemistry and physics only, with sufficient mathematics to work out problems set by an examiner. The lecture courses must be supplemented by laboratory work, or I believe it would be far better if these ran concurrently, for in evening classes there is not too much time to spare, and thoroughly

good practical experiments with the theory of their action explained at the time would be of far more value than many of the separate lecture and practical courses now in vogue.

An enlargement of such a course as the alternative elementary chemistry course of the Science and Art Department is in the direction I mean, for this is infinitely superior in imparting sound chemical instruction to the usual lecture courses. Then other subjects should be taken, such as will give some good knowledge of the nature of materials used in the construction of buildings and plant, and there should be instruction in the elements of mechanics, in building construction, machine construction, and drawing; all these, however, dependent upon the special occupation of the student and the time at his disposal. A study of these extra subjects is, I consider, most essential, especially for those who have any responsible charge in connexion with our chemical industries, for it has been far too frequently shown that a chemist who is a chemist only, however thorough his chemistry, is not the proper person to conduct chemical manufacturing work, for numerous are the chemical processes that have failed, although sound as regards their chemistry, because attempts have been made to work them with totally unsuitable plant, and with ignorance or disregard of circumstances that could not fail to materially modify the chemical reactions and prevent success.

This is why I recommend attention to the organising of subjects in science schools, so that the proper subsidiary subjects may be taken in connexion with the main subject. The City and Guilds of London Institute has done something in this direction.

Progressing from that teaching which forms the scientific groundwork, and coming to the more purely technological instruction, what is the training that can be given? Each locality of chemical industry should organise special courses bearing upon its own special industries, but such courses should not be mere lecture courses simply described by the aid of a few diagrams, models, or specimens. The processes described must be, as it were, turned inside out, and every possible detail that affects success or failure carefully explained.

But the sort of courses that I think would be of most service would be good practical expositions of general operations, such as would deal with the operations of fuel-burning, ebullition and evaporation, solution, crystallisation, fusion, &c. These could be made useful in connexion with the work of almost any manufactory, and would be very serviceable to those who cannot give time to follow out special courses, or who are not engaged in any special work.

Having organised such instruction for our workpeople, we may next endeavour to stimulate them to study by giving preference in their employment to those who show themselves specially apt, and by offering scholarships and such like rewards for those really hard working intelligent students who wish to go further in their studies in higher schools or colleges, and thus help them forward in the world. I think the Society of Chemical Industry might well consider present and new methods of instruction in chemical technology, and help good work by offering scholarships and by other practical aid and suggestions.

But now, leaving the consideration of the education of our workpeople, and coming to the education of those who are to take higher grades in our chemical industries. First of all, it is necessary to give attention to our ordinary middle-class schools, which frequently are very deficient in scientific and practical training, and then we have to consider how far we can organise schools for some such scientific and preliminary technological teaching as I suggest we should provide for our workpeople. And next we have to look to our higher colleges and universities, which should give the highest training possible, and more especially confine themselves to such work instead of overlapping the work of more elementary schools. That the more elementary teaching should be given in these higher colleges is probably now necessary, because where many of them exist, being of late establishment, the ground has not been well prepared for them, and students do not enter them sufficiently advanced for the higher branches of study.

For the more technological work something in every way more complete is necessary than what I have described. In

workmen's evening schools and others giving similar instruction, again there should not be too much of the lecture course without ample opportunity for full demonstration of all the principles involved in the processes; and I cannot help but think that even in these higher schools and colleges, instead of some of the courses on special processes, courses on general operations would often prove more useful.

But who is to give this advanced technological instruction? Unfortunately, teaching of technology is not usually considered advanced, as it is usually put down for study under quite subsidiary circumstances, although I consider it should be just the reverse. Mr. Levenstein, in a late address at the Technical School, Manchester, stated that we were short of teachers, and I quite agree with him. One of the greatest difficulties in teaching technology in this country is to get suitable teachers. There has been hitherto no inducement to really capable men to teach. The merest pittance has been awarded for such teaching, and in one or two of our largest colleges it is a scandal that the teaching which bears most upon useful practical work in connexion with our industries should be so poorly paid as compared with that of the men who do the more elementary work. Consequently, men capable of teaching, but with a bent for technology, have occupied themselves in the more practical manufacturing pursuits, in which remuneration has been more in proportion to their labours, and technological teaching has been left more especially to those whose training has been chiefly that of the school or college, with a smattering gained by an occasional walk through a manufactory or a more or less diligent study of technological works. If we are to alter this and get good men to teach we must see that such teaching is given a more honourable position than it has hitherto held, and one which its importance deserves. If we follow upon some such lines as those I have endeavoured to mark out, I cannot but think that this new era in technical instruction will prove of the greatest advantage to our industries.

#### INDUSTRIAL MUSEUMS.

But now I wish to speak of technical, trade, and commercial museums as aids to industries. Our ordinary museums are, I fear, too much neglected to be of much educational aid, and we are miserably deficient in industrial museums. Yet a well-arranged series of raw products, descriptions of their characters and of the processes by which they are converted into new materials, with specimens of these new materials, can prove of great help in the study of an industry, and an incitement to further work in designing new processes, looking for new material and making serviceable that which has hitherto lain idle. Such collections should be supplemented by carefully prepared descriptive handbooks and by frequent lectures illustrative of the nature and uses of the several specimens. There are numerous such museums on the Continent and in America. I have seen lately the Commercial Museum of Brussels and one in the neighbouring city of Antwerp, which latter is an outcome of the Antwerp Exhibition of 1885, and to this annual subsidies have been voted both by the municipal and provincial authorities; but one of the most interesting and instructive that I have seen, and one that may well be taken as a model, although of comparatively small size, is at Haarlem in Holland, and in this is an excellent exhibition of the products of the Dutch colonies, with illustrations of the mode of their collection or production, and the uses to which they are applied. A collection of similar character should exist in this city of Liverpool, the largest port in the world. It would materially help on commerce and industries, and should contain raw and manufactured products imported and exported, with their technical histories and illustrations of the uses to which they are applied, and new materials with suggestions as to their possible uses. A large number of substances come to Liverpool year after year that are never made use of because they are never heard of beyond one or two brokers or merchants, who, not being able to examine properly into their characters and capabilities, relegate them to the category of useless articles, for, owing to want of training in scientific and technical knowledge that would be of service to them in their everyday business

many of our merchants and brokers are unable to deal, even in an elementary way, with substances a little different to those they are accustomed to.

And a museum that would be serviceable in a port like Liverpool would be, I believe, of even more value in centres of industrial activity.

Such museums would be fitting adjuncts to technical schools; and following on these there should be—

#### LABORATORIES OF RESEARCH

Specially arranged for the examination of raw products and manufacturing substances. In these new imports or native products might be subjected to tests to ascertain the purposes for which they are suited; and substances already known, but not in use, might be systematically examined to endeavour to find uses for them. A large number of new substances are brought into the different ports of this and other countries every year, that, properly used, could be made of commercial value, but now, for want of careful examination, remain noticed only for a short time as curiosities, and are then forgotten. Laboratories of research could also be made available for finding new uses for substances already employed, and probably frequently outlets would be found for commodities with which the market is overstocked. Supplementary to such institutions should come—

#### TECHNOLOGICAL LIBRARIES.

With ample supply of scientific and technological literature, although to give them a completeness much improvement is desirable in our technological literature, for many of our technological handbooks are but "hashes" of a few larger works, and these are often but of indifferent character. Probably the best example of technological literature that has been produced is our own Journal.

#### INVESTIGATION OF ANALYTICAL PROCESSES.

All the topics I have yet dealt with are more especially educational, but now I turn to something different to point out how greatly chemical industrial, and commercial transactions would be benefited by a thorough investigation of processes employed in the examination of raw and manufactured substances and material in process of conversion. Many processes, excellent in themselves, give, in the hands of different operators, results so far different as to lead to annoyance and inconvenience, and this frequently arises from some very slight difference in details of manipulation.

Other processes again vary in results according to variations in the substances examined, such as where the determination of the main constituent is modified by presence of impurities or small quantities of other substances, the action of these extraneous matters being often overlooked.

It would, I think, be very useful to bring before this Society papers comparing results on the examination of certain products and determination of certain constituents when treated by different analytical processes or by essentially the same process under somewhat different conditions.

It is most important for chemical transactions that differences in commercial analyses should not occur, as oftentimes they mean not only delay in settling transactions but considerable difference in money values, &c.

But whilst speaking of differences in analyses I cannot help referring to sampling operations, which are a more fruitful source of discrepancies and require most careful attention; indeed, to ensure accuracy in assessing the true value of a commercial article sold on test, the sampling operation is almost, if not quite, as important as the analytical.

I wished to have alluded to the use of the microscope in chemistry, as its employment is every day becoming more important, but must now close, and trust I may have an opportunity of dealing with that subject and other aids to chemical industries on a future occasion.

Dr. HURTER proposed and Mr. E. K. Muspratt seconded a vote of thanks to Mr. Norman Tate for his address, which was supported by Dr. Campbell Brown, and to which Mr. Tate replied.

## Newcastle Section.

Chairman: John Pattinson.

Vice-Chairman: T. W. Stuart.

#### Committee:

A. Allhusen.	T. W. Lovibond.
P. P. Bedson.	H. R. Procter.
G. T. France.	B. S. Procter.
G. Gatheral.	W. W. Procter.
C. H. Hils.	W. L. Rennoldson.
T. W. Hogg.	J. E. Stead.

Hon. Local Secretary and Treasurer:

Dr. J. T. Dunn, The School, Gateshead.

Notices of Papers and Communications for the Meetings to be sent to the Local Secretary.

Meeting held in the College of Science, Thursday,  
November 6th, 1890.

MR. JOHN PATTINSON IN THE CHAIR.

#### CHAIRMAN'S OPENING REMARKS.

ALLOW me to thank you for the honour you have conferred upon me in electing me Chairman of the Newcastle Section of the Society of Chemical Industry. Fifteen years ago I occupied a similar office in the Newcastle Chemical Society, which became amalgamated, very wisely, as I think, with this Society in 1882, and although you have on more than one occasion since then asked me to become your Chairman, I have always felt it my duty to endeavour instead to induce others closely identified with the chemical industries of the district to occupy this post, and thus to interest themselves in the welfare of the Society. The time has now arrived, however, when you again wish me to take this office, and you have done me the honour of electing me. I shall try to fulfil its duties to the best of my ability.

I do not intend troubling you with a formal inaugural address, but rather to imitate the example of the London Section and that of some other Sections in this respect. They have no inaugural addresses. I believe that it is desirable to get rid of the notion that Chairmen of Sections are expected to prepare an elaborate inaugural address. Such a notion is calculated, I think, to deter many gentlemen from accepting office who are in every way well fitted to act as Chairmen, but who may not have the time to prepare, or who think they have not matter of sufficient interest for, an elaborate address on chemical subjects.

There are, however, one or two subjects of special interest to most of our members of which I think mention should be made on this occasion. The first is the much-talked-of union of Leblanc soda manufacturers. The prospectus of a company to be called "The United Alkali Company, Limited," is just now being issued. Should this company be floated, nearly the whole of the Leblanc soda works in the United Kingdom, numbering about 50, will become the property of one large concern, and be managed by one and the same board of directors. Much has been written in the newspapers in condemnation of this project by some of the consumers of the produce of these chemical works and by other interested persons. There is, however, much that is exceptional and peculiar in the present position of the Leblanc soda makers which leads to the conclusion that, from their point of view at any rate, this union is a very wise and prudent step. We all know the circumstances. Carbonate of soda can be much more cheaply made by the ammonia-soda process than by the Leblanc process, and the ammonia-soda makers consequently command the market for this article. The price of alkali has thus been brought down so low that its manufacture by the Leblanc process has, to say the least, ceased to be remunerative, and the costly plant used in the Leblanc works would probably ere

now have been entirely disused were it not that the hydrochloric acid resulting from the Leblanc process (which acid at one time was regarded as a waste by-product) has always been and still is the cheapest source of chlorine for the making of bleaching powder and chlorate of potash. The by-product has thus become the chief manufacture and source of profit of the Leblanc soda makers, and the soda may now be considered the by-product. There have been during the past six or eight years various announcements that new and cheaper sources of chlorine had been discovered which would supersede the hydrochloric acid of the Leblanc process, and thus give the deathblow to the Leblanc soda makers, but in spite of all these prognostications, as a matter of fact the whole of the 150,000 tons per annum of bleaching powder made at the present day is made from the hydrochloric acid of the Leblanc process. Electrolytic bleaching in paper works, too, has not yet proved the success that it promised to be, so that the demand for bleaching powder is still undiminished. Unfortunately for the Leblanc manufacturers the Leblanc works in existence could make more bleaching powder than there was a demand for, and hence we have had the makers competing with each other until bleaching powder as well as soda was sold at an unremunerative price. Under these circumstances, towards the end of 1883 the bleaching powder makers formed a combination to restrict the make, and thus to secure a price which would enable them to carry on their works. This combination was for some time successful in its object, and it lasted until the end of 1889, when, owing to some of the makers proving disloyal to the arrangement, it ceased to exist. Should the proposed union be carried out, the United Alkali Company will then have complete control over the quantity produced and the prices at which sales are effected. In this respect the proposed united company will have an advantage over the recent bleaching powder combination. In other respects I do not see that the effect upon the trade of the country and upon the users of bleaching powder is likely to be very different from the effect of the bleaching powder combination existing from 1884 to 1889. Practically the present union of Leblanc soda makers is a bleaching powder combination without the possibility of disloyalty. We did not find then that the price of bleaching powder was raised to a price which seriously affected the paper makers and others, nor need we fear that the prices will now be raised exorbitantly high. The directors of the united company know as well as any other business men that very high prices can only result in bringing a host of competitors into the field, and the consumers of caustic soda and bleaching powder are thus safeguarded against any serious rise in prices over and above those necessary to carry on the manufacture so as to yield a reasonable amount of profit.

How will the proposed union affect the Society of Chemical Industry? Will it reduce the number of its members, or lessen the number of papers contributed to its meetings? I am inclined to think that it will not seriously affect us. Managers and chemists will still be wanted in each of the works, and there will still be the same opportunities as at present of their suggesting improvements in processes in the works and in the laboratories.

The other subject to which I wish to call your attention this evening is the question of black coal smoke. In spite of all the numerous contrivances for the prevention of smoke there is no doubt I think that on the banks of the Tyne the nuisance from black smoke has considerably increased in recent years. In most cases the stoking of furnaces, used only for the raising of steam, seems to be carried on utterly regardless of the quantity of smoke emitted or of the dirt and discomfort caused to those who have to live in the smoke-polluted neighbourhood. We sometimes pride ourselves upon the striking architectural and natural features of our city as seen in approaching it from the south, but in my intercourse with travellers through Newcastle-on-Tyne, I have found that the only feature noticed in most cases is the extreme smokiness of the place. This state of things should not exist. It is quite time that the attention of manufacturers and the general public should be called to the matter, and that an attempt should be made to lessen the evil. I am not one of those who think that, under all circumstances, it is an easy thing to entirely

prevent even the occasional formation of some smoke when burning Newcastle coal; but I am quite sure that with suitable appliances and with a little care very much of the black smoke now sent out from boiler fires can be prevented. I am glad to be able to announce that this question of smoke prevention has just recently been taken up by a number of gentlemen in various parts of the country who have formed themselves into a "Committee for Testing Smoke Preventing Appliances." Mr. A. E. Fletcher, the respected Chief Inspector of Alkali Works has taken great interest in the formation of this association, and he is the chairman of the executive committee. It is not the intention of the association to harass the manufacturer by legal proceedings. On the contrary the aim is to aid manufacturers by collecting information respecting the efficiency and general value to the coal consumer of smoke-preventing appliances and methods, and to employ disinterested experts to thoroughly test these appliances and methods when using various qualities of coal. The manufacturer will thus learn, on trustworthy authority, what smoke-preventing appliances suitable for his purposes exist, and he will be saved from the fate of many manufacturers who, on the representations of the inventors, have fitted up costly forms of apparatus which on trial have proved to be neither economical or efficacious. I have no doubt that the labours of the committee will assist very materially in obtaining an abatement of the smoke nuisance. The work they are undertaking will however take some time to complete, and much expense will necessarily be incurred. I trust the members of this Section and all manufacturers in this district will as far as possible give this project their sympathy and support.

We look to the members of this Section to interest themselves in providing papers and notes for our monthly meetings during the present session and thus to add to the progress and success of our Section of the Society.

## SOME NEW AND IMPROVED ANALYTICAL METHODS FOR ALKALI WORKS.

BY PROFESSOR G. LUNGE.

THE first edition of the "Alkali Makers' Pocket Book," edited by Dr. Hurter and the writer, has found so much favour with the chemical public that a second edition has been called for, which will be issued shortly. Although the great majority of the analytical methods prescribed in the first edition of our book have stood the test of five or six years' practical application in a large number of laboratories, it stands to reason that this space of time has not passed by without bringing under our notice the desirability of improving some of the methods and tables then given, and adding new ones for wants which have made themselves felt in the meantime. Since, according to the plan of the "Pocket Book," we can give there merely the practical prescriptions for carrying out the tests, and these in the fewest possible words, it seems appropriate to choose another channel for laying before our fellow workers the reasons why we have made a number of alterations and additions to the "Pocket Book," and giving sufficient details for proving the accuracy of the new methods proposed; and these notes will, I hope, possess some interest also for those chemists who do not consult the "Pocket Book."

The bulk of the work to be described has been done by myself, with the help of some of my assistants; where suggestions have come to me from outsiders I shall duly mention them.

### 1.—ESTIMATION OF SULPHUR IN PYRITES.

This is merely mentioned to state the fact that the method described by me previously, and adopted in the "Pocket Book," has been over and over again proved to be equal in accuracy to the best otherwise known, and far superior to them in point of convenience, speed, and facility of execution. A somewhat serious attack was made upon it by Jannasch and Richards, but Professor Jannasch has



himself acknowledged (J. prakt. Chem. 40, 236) that their criticism was caused by a confusion of my older with my more recent method, which they had entirely overlooked, and that my new method gave, in his hands as well as in my own, the most accurate results. This has been conclusively proved by the comparison of methods made by two of my students, Messrs. Barbezat and Obregia (Zeits. f. angew. Chem. 1889, 473), and this matter may now be considered as settled.

For the testing of *burnt ore* (pyrites cinders) we now prefer the method of Watson (this Journal, 1888, 305) which was found to be preferable to all others tried in my laboratory.

## 2.—TESTING THE PYRITES BURNER GASES.

Wherever the burner gases are tested, it is probably always done by Reich's method, as described in the "Pocket Book," that is, aspirating the gas through a measured quantity of iodine solution, and ascertaining the volume of gas required for decolorising the iodine. This shows the quantity of sulphur dioxide present. Since, however, it has been proved by many observers that the gases contain sulphur trioxide, sometimes up to one-tenth of the total sulphur present, the above-described test must be declared inaccurate, and I propose, instead, estimating the *total acids* by passing the gases through a *caustic soda solution*. Very much depends in this case upon the indicator to be chosen.

The indicator must be affected exactly alike by  $\text{SO}_2$  and  $\text{SO}_3$ , otherwise the results will not give any useful clue to the working of the process.

Former investigators (especially R. J. Thomson, myself, and, much later, Blarez) have proved that litmus and most other ordinary indicators give indistinct results; methyl-orange shows the point of saturation for  $\text{SO}_2$  at the point when  $\text{NaHSO}_3$  is formed; only phenolphthalein sharply indicates the point when  $\text{Na}_2\text{SO}_3$  is formed, so that only this indicator can be used in the present case, since it is indispensable that the point of saturation be the same at whatever degree of oxidation the sulphur be present in the gases.

The next question was this: Whether the absorption of the sulphur acids by very dilute caustic soda solution is as easy as that of  $\text{SO}_2$  by iodine, and whether Reich's apparatus would have to be modified for this purpose. To decide this, in the first instance a number of laboratory experiments were made under my direction by Mr. Marchlewski, with mixtures of  $\text{SO}_2$  and air in varying proportions, by means of a specially constructed gas holder in which a fairly constant proportion of gases could be kept for some time. It was soon found that it would be desirable to modify the absorbing bottle so as to become more efficient, and this has been done by adopting the form shown in Fig. 1. The inlet tube for the gas is sealed at the bottom, and is perforated below the level of the liquid with many pin-holes, through which the gases issue in many minute bubbles, instead of one large bubble. Our bottle held 410 cc. and was charged with 230 cc. of water, 10 cc. of decinormal soda solution, and three drops of an alcoholic solution of phenolphthalein. Each time after opening the aspirator tap for a moment and passing a few cc. of gas through, the bottle was shaken up for half a minute. Especially towards the end this must not be neglected, and it must be carefully observed on a white background whether the last pink tint has vanished or not. Special tests were made in the twilight and with artificial light to approach the conditions of working in factories. In every case at the end of the operation a single drop of decinormal soda solution was added in order to try whether there was not an excess of  $\text{SO}_2$  present, which would prevent the liquid from turning pink again, but this never took place. A fresh experiment can be made at once in the same bottle by simply adding another 10 cc. of soda solution, and this can be repeated several times in succession.

In the following experiments the percentage of  $\text{SO}_2$  was calculated by the formula  $\frac{100 \times 11.14}{11.14 + n}$ , in which 11.14 represents the volume of  $\text{SO}_2$  retained by the 10 cc. of decinormal soda solution, and  $n$  the volume of the water run out of the aspirator, reduced to 0° and 760 mm. With

five different mixtures of gas (A—E) the following results were obtained:—

A.				
n.	1.	2.	3.	4.
271	corresponding to 3.90 per cent. $\text{SO}_2$ by volume.	271	3.94	" "

B.				
n.	1.	2.	3.	4.
262	corresponding to 4.07 per cent. $\text{SO}_2$ by volume.	263	4.06	" "

C.				
n.	1.	2.	3.	4.
236.5	corresponding to 4.49 per cent. $\text{SO}_2$ by volume.	238	4.47	" "
		237.5	4.49	" "

D.				
n.	1.	2.	3.	4.
131	corresponding to 7.83 per cent. $\text{SO}_2$ by volume.	133	7.73	" "
		130	7.81	" "

E.				
n.	1.	2.	3.	4.
122	corresponding to 8.44 per cent. $\text{SO}_2$ by volume.	124	8.24	" "

These experiments prove that the described method yields concordant and evidently acceptable results. It was, however, a further question whether this would be also confirmed by actual factory testings, or whether in these there would be an essential and permanent difference between the soda and the iodine method. Experiments in this direction have been made by Dr. Notzli at the Aussig Chemical Works, by aspirating burner gases by means of a T-tube at the same time through decinormal soda solution and decinormal iodine solution. In these first experiments no reading of the thermometer and barometer was taken for correcting the volume of gas aspirated, hence the percentages calculated are not exact, but the results are all the same comparable with one another.

The following observations were made with successive samples, of necessarily varying percentages, but those given on the same line (for soda and iodine respectively) were always taken by aspirating at the same moment.

### SERIES A.

	10 cc. Soda Solution.	10 cc. I Solution.
Water run out ....	cc. 134	cc. 154
" ....	136	148
" ....	146	160
" ....	158	170
" ....	169	183
Average .....	149	163
Corresponding to..	7.0	6.4 per cent. $\text{SO}_2$ by volume.

### SERIES B.

	10 cc. Soda Solution.	10 cc. I Solution.
Water run out ....	cc. 164	cc. 175
" ....	136	148
" ....	122	130
" ....	101	112
" ....	98	105
" ....	95	103
Average .....	119	128
Corresponding to..	8.6	8.0 per cent. $\text{SO}_2$ by volume.



## SERIES C.

	50 cc. Soda.	50 cc. I.
Water run out ....	cc. 614	cc. 684
Corresponding to..	8.0	7.5 per cent. SO <sub>2</sub>

It is perfectly clear that in every single case the total acidity, calculated as SO<sub>2</sub>, is superior to the quantity of real SO<sub>2</sub> as indicated by the iodine. But in order to obtain a sure quantitative foundation I caused Dr. Nötzli to make a special experiment in which the thermometer and barometer were observed, and in which the acids were all oxidised by means of iodine, and the total sulphuric acid gravimetrically estimated, so that we now possess all the elements of calculation. Burner gases were aspirated at the same time though 50 cc. decinormal NaOH and 50 cc. one-tenth normal I. The temperature in the aspirating bottle (which was placed close to a large gas-pipe) was 34° C., the barometer stood at 740 mm. In order to discharge the colour of phenolphthaleïn, 650 cc. water had to be run out; the absorbing liquid, after oxidation by I, yielded gravimetrically 0.1963 grm. SO<sub>3</sub>. In order to discharge the colour of the iodine 684 cc. of water was used; gravimetric analyses showed 0.2047 grm. SO<sub>3</sub>. Now we find:—

## A.

650 cc. of 34° and 740 mm. ....	= 532 cc. of 6° and 760 mm.
To this must be added the SO <sub>2</sub> absorbed, = 50	" "
Total original gas.....	588 " "
or $\frac{55.7}{588} = 9.47$ per cent. by volume of total acids calculated as SO <sub>2</sub>	

## B.

684 cc. of 34° and 740 mm. ....	= 560 cc. of 6° and 760 mm.
Add for SO <sub>2</sub> .....	56 " "
Total original gas.....	616 " "
or $\frac{55.7}{616} = 9.04$ per cent. of SO <sub>2</sub> really present.	
leaving	0.43 per cent. of SO <sub>2</sub> present as SO <sub>3</sub>
<u>9.47</u>	

If 588 cc. of original gas, after oxidising all SO<sub>2</sub>, yielded altogether 0.1963 SO<sub>3</sub>, 616 cc. of gas must yield  $\frac{616 \times 0.1963}{588} = 0.2056$ . We really found in the iodine solution 0.2047, which is a very satisfactory confirmation.

This calculation at the same time shows what great errors are often made in factories by neglecting the temperature and barometrical pressure. Even in the above estimations there is still a slight error, since the column of water in the aspirator (whose height I do not know) ought to be deducted from the barometrical pressure; but this error, which would appear to make the percentages slightly lower than they actually were, cannot have affected the total results to a sensible degree.

I may be allowed in this place to mention a plan for testing burner gases which has been in my mind for several years past, but for which I have not yet found the means of carrying out in practice. This is—*estimating the percentage of sulphur acids in burner gases by their specific gravity*. The gaseous mixture in question contains an almost constant proportion of nitrogen, a quantity of oxygen varying between narrow limits, and a certain quantity of the acids of sulphur, together with those of nitrogen which may (and indeed must) be neglected in this as well as in the preceding method of testing. Nor is it necessary to take notice of the difference in specific gravity between the vapours of SO<sub>2</sub> and SO<sub>3</sub>, for the method in question cannot replace an exact analytical estimation, and is only intended to facilitate the control of the vitriol-making process by the ease and speed of the observation, and by the possibility of arranging a *continuous graphical check of the pyrites-burning process*.

If we put atmospheric air = 1, the specific gravity of nitrogen is = 0.9714, that of oxygen, = 1.1056. Suppose we replace part of the oxygen by sulphur dioxide, whose specific gravity is = 2.247, the specific gravity of the mixture will be raised; for instance, with 8 per cent. gas:—

$$\begin{array}{rclcl} 79 \text{ N} & = & \frac{79}{100} \times & 0.9714 & = & 0.7675 \\ 13 \text{ O} & = & \frac{13}{100} \times & 1.1056 & = & 0.1437 \\ 8 \text{ SO}_2 & = & \frac{8}{100} \times & 2.247 & = & 0.1798 \\ & & & & & \hline & & & & & 1.0910 \end{array}$$

or with 6 per cent. gas:—

$$\begin{array}{rclcl} 79 \text{ N} & = & \frac{79}{100} \times & 0.9714 & = & 0.7675 \\ 15 \text{ O} & = & \frac{15}{100} \times & 1.1056 & = & 0.1658 \\ 6 \text{ SO}_2 & = & \frac{6}{100} \times & 2.247 & = & 0.1348 \\ & & & & & \hline & & & & & 1.0681 \end{array}$$

A difference of 2 per cent. volume of SO<sub>2</sub> is represented by a difference of 0.023 in specific gravity. That is, differences of 1 per cent. SO<sub>2</sub> will be indicated by the second, and such of 0.1 per cent. SO<sub>2</sub> by the third decimal place of the specific gravity.

It is some time since Mr. Friedrich Lux, of Ludwigshafen, constructed his gas-balance, which is now generally employed for instantly or continuously testing the specific gravity of ordinary illuminating gas; and it has also been employed with some like success in the analysis of that gas. For two years past I have tried to induce Mr. Lux so to modify his gas-balance that it could be used for acid gases; if I had such an instrument at my disposal, I am sure I could make it useable for testing burner or vitriol-chamber gases for SO<sub>2</sub>, and even arrange it for continuous registration of the results. But hitherto I have not been able to obtain a balance made of such material that it could be used for this purpose, and it remains to be seen whether Mr. Lux will be able to do that.

Another method for estimating the specific gravity of gases has quite recently been proposed by Friedrich C. G. Müller. It consists in passing a stream of the gas through a perpendicular tube, 4 ft. long, connected at the bottom with a sensitive pressure-gauge communicating at the other end with the atmospheric air. It stands to reason that the 4-ft. column of gas will exercise a different pressure on the pressure-gauge from that of atmospheric air, and this, by means of an empirical graduation, can be utilised for estimating the specific gravity to the third decimal. After prolonged correspondence with the inventor, concerning the application of this ingenious method to the testing of burner gases, I have come to the result that this should be possible, but so far I have found no time to work out a practical method and apparatus for this purpose.

## 3.—TESTING OF THE CHAMBER EXIT GASES.

The testing of the exit gases from vitriol chambers is decidedly capable of improvement in some particulars. In the first instance the well-known prescriptions of the English Alkali Makers' Association can be greatly simplified by introducing the above described process with caustic acid and phenolphthaleïn, and this has been already done with great practical success.

But I found it also necessary to revise the methods for estimating the *nitric oxide* in the exit gases, since many experiences point to the fact that far greater losses arise from this source than had been hitherto assumed.

For this purpose it was indispensable, first, to compare the different descriptions of absorbing apparatus in use, and select the best of them; secondly, to see whether any more accurate method than that employed—potassium permanganate as an absorbent for NO—could be found. I will here give only the general results of this investigation, in which I have been very ably assisted by Mr. Marchlewski.

*Concerning the best shape of absorbing apparatus.*—I excluded at once from our research the simple and rough forms, as well as those adapted only for re-weighing, but not for re-titration, such as Liebig's potash-bulbs. The

apparatus experimentally compared were: first, Mitscherlich's tubes, Fig. 2; second, Pettenkofer's tubes, as modified by myself, Fig. 3; third, Winkler's glass worms, which are

equally to be considered as modified Pettenkofer tubes; fourth, glass-bead tubes, Fig. 4; fifth, bulb tubes with 10 bulbs, of the shape shown in Fig. 5.

Fig. 1.

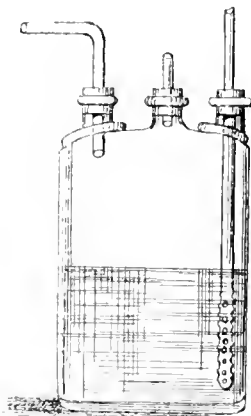


Fig. 3.

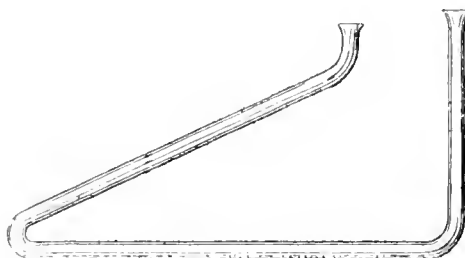


Fig. 4.

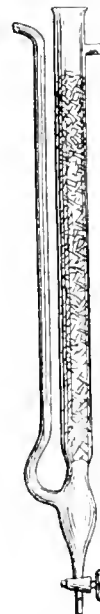


Fig. 6.



Fig. 2.

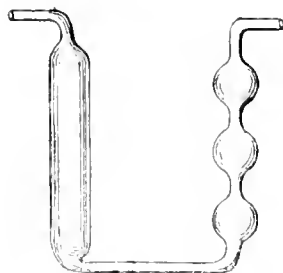
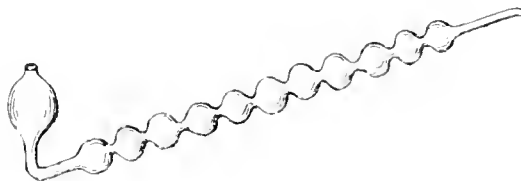


Fig. 5.



If the Mitscherlich tubes, we always placed three in succession, of the other apparatus two, and made a large number of experiments with all of them with the following results. The Mitscherlich tubes are most imperfect, even three of them in succession do not secure a complete absorption of the nitric oxide from a 1 per cent. mixture of that gas with 100 vols.  $\text{CO}_2$ . Since the tubes known as "Todd's tubes" are but slightly modified Mitscherlich bulbs, similar results must be looked for in their case. Much better was the absorption with the Pettenkofer-Lunge tube, indeed, decidedly superior to Winkler's glass worms, but even here a single apparatus did not retain all the  $\text{NO}$  from a 1 per cent. mixture. This was, however, the case both with the glass-bead tube, Fig. 4, and the ten-bulb tube, Fig. 5, one of which sufficed for completely absorbing the  $\text{NO}$  from a 1 per cent. mixture with  $\text{CO}_2$ . Of these two apparatus I ultimately decided for the ten-bulb tube, which for greater security may be enlarged to fifteen bulbs, because it is easier to employ and completely wash it for re-titration.

My research further extended to the question whether potassium permanganate could be replaced by a better absorbent for nitric oxide. Permanganate has the drawback that even strongly acid solutions, on passing the gases through, especially when hot, gives a precipitate of hydrated manganese dioxide. This causes no inaccuracy in testing if the tube is rinsed with part of the ferrous sulphate solution employed in re-titrating, when the precipitate will easily dissolve; the final result is exactly the same as in such cases where no manganese dioxide had been precipitated. But this precipitation is always a little inconvenient, and an absorbent free from this drawback would be, no doubt, preferred to potassium permanganate, especially if it were more sensitive than that reagent is to nitric oxide.

Hydrogen peroxide has been several times proposed for absorbing nitric oxide, and we thoroughly examined it in that respect, but it does not seem worth while to give any details of our experiments, for the result was most decisive. Hydrogen peroxide, both in an acid and an alkaline solution, is a bad absorbent for nitric oxide, and altogether to be rejected for the estimation of that substance.

Many years ago I had tried another kind of absorbent for nitric oxide, viz., concentrated nitric acid, or a mixture of this with strong sulphuric acid. This retains the nitric oxide by the formation of nitrous acid, which can then be estimated by means of potassium permanganate or by the nitrometer. As the stream of gases carries away a good deal of nitric acid, we must retain this by a solution of caustic potash or soda. We have now subjected this method to a thorough investigation, with the following result: The method is much better than absorption by hydrogen peroxide, but it is decidedly inferior to the use of potassium permanganate, both in point of accuracy and in that of speed and convenience of execution.

The final conclusion of that research was: *That, so far, no better mode of estimating the nitric acid contained in chamber exit gases is known than that of passing them through a titrated solution of potassium permanganate, strongly acidified with sulphuric acid, and contained in a 15-bulb tube. At the end of the operation an excess of titrated solution of ferrous sulphate is added, and the operation brought to an end by re-titrating with standard permanganate.*

I may be permitted to state on this occasion that for standardising a solution of potassium permanganate, as well as for the testing of manganese ore and bleaching powder, the gas-volumetric methods worked out by me, and founded on the use of hydrogen peroxide, offer great

convenience and saving of time, and are at all events most useful in checking the ordinary methods. It is hardly necessary to point out in what extraordinary degree all such methods are facilitated by the use of my *gas-volumeter*, which permits reading off the volumes of gases as corrected for temperature, pressure, and moisture, without any calculation or any observations of thermometer and barometer. This is equally the case with the nitrometric operations proper, for which I have constructed a special "reaction-vessel" (this Journal, 1890, 548), greatly facilitating and expediting the analysis of *nitrous vitriol*, *nitrate of soda*, and so forth.

#### 4.—SPECIFIC GRAVITY TABLES FOR SULPHURIC ACID.

New tables of the specific gravities of sulphuric acid of various strengths have been worked out with the greatest care by myself in conjunction with Mr. Isler, and have been reprinted in the Society's Journal, 1890, 501. Similar work is to be done for nitric and hydrochloric acids, but so far the considerable time required for it has not yet been found.

Soon after the first publication of our memoir there appeared in the Journal of the Chemical Society (vol. 57, 61—185), an extremely elaborate paper by Professor Pickering on the same subject; and Mr. H. D. Richmond has founded upon this a table of the specific gravities of concentrated sulphuric acid (this Journal, 1890, 479). Professor Pickering's results are not unessentially different from ours, and the enormous amount of his observations, and the seemingly stupendous accuracy of those, indicating *thousandths* of a per cent. of  $\text{H}_2\text{SO}_4$  in the acid, may possibly make them appear more trustworthy than ours to those who have not worked in the same field. But I cannot for one moment acknowledge this conclusion. Hundreds of tests made by myself with the greatest possible care, with calibrated vessels and corrected weights, with constant checkings and re-checkings, have proved to me that estimations of strong sulphuric acid are already *very* good, if they do not deviate from one another more than by one unit in the first decimal per cent., and that the *utmost* accuracy attainable, but most certainly not attained by the great majority of workers, is  $\pm 0.05$  per cent. If, therefore Professor Pickering maintains (loc. cit. 73) that his percentages are accurate to  $\pm 0.001$ , this *impossible* assertion is in my mind from the outset a reason for distrusting the accuracy of his figures altogether, and I believe that this impression will be conveyed to others as well who take the trouble of looking at the very tottering foundation of that seemingly stupendous accuracy. All his hundreds of observations are founded upon the analysis of a sample of pure sulphuric acid, supplied by Dr. Messel, from which all other strengths were prepared by adding weighed quantities of water, without any further analyses. The fundamental analyses were not made by Professor Pickering himself, but by Mr. Crompton, and no special description is made of the methods used, except that the estimations were made volumetrically, starting from pure silver. Now those estimations of Mr. Crompton's vary between 99.704 and 99.962 per cent., that is to the amount of 0.26 per cent., and yet Professor Pickering does not hesitate to assert that the percentage of his acids, made from the fundamental acid analysed by Mr. Crompton by way of dilution, and consequently unavoidably introducing some fresh errors, however slight, are accurately known to the third decimal per cent. It is simply impossible to admit the justice of this claim, but with it falls to the ground any claim to the superiority of his results over mine.

#### 5.—SODA MANUFACTURE.

The stimulus for working out the following methods was afforded by a suggestion on the part of Dr. Hurter, who pointed out that analytical methods for the purposes in question, adapted to the requirements of a works laboratory, were very desirable.

(a.) *Estimation of Free Lime in Black-Ash.*—It is well known that a certain quantity of free lime ought to exist in black-ash, in order to facilitate the lixiviating process. I

have found that the following process, an adaptation of a plan proposed by Winkler for the analysis of quicklime, yields very good results.

The sample of black-ash is treated in the ordinary manner, as prescribed in the "Pocket Book," for obtaining a solution; that is, 50 grms. are quickly, but thoroughly, ground up to a fine powder, which is put into a litre flask and covered with tepid distilled water, *freed from carbon dioxide by boiling*. The mixture is at once well shaken up, and the agitation is several times repeated during two hours. Without such speedy and thorough shaking, a hard cake is formed at the bottom which does not any more disintegrate. After two hours the flask is filled up to the mark, and special samples are taken out for the single tests. First of all the tests are made for free lime and, if required, for total lime (see below), and for these the contents of the flask are thoroughly shaken up each time before taking out a sample. When this has been done, the flask is allowed to rest and after settling the clear part of the contents is used for the ordinary black-ash tests. The samples intended for the estimation of free or of total lime are taken out with a pipette of the form shown in Fig. 6, which is not so easily stopped up by the muddy residue as the ordinary pipettes, with a long thin outlet tube. After taking out the sample, the outwardly adhering mud is washed off, the contents of the pipette are run into a beaker, the pipette is rinsed into the same, an excess of barium chloride solution is run in, a drop of phenolphthalein solution is added, and the liquid is titrated in the cold with one-fifth-normal oxalic acid till the pink colour has just disappeared. If care has been taken to thoroughly shake up the flask before each sample is taken out, the results agree perfectly well. By this method only the free lime is found, as the oxalic acid acts upon the carbonates only after saturating the free lime, and, when doing so and setting some  $\text{CO}_2$  at liberty, the red colour is discharged at once.

A special set of tests were made by adding a quantity of neutral cane-sugar solution, in order to see whether more lime would thus be indicated, owing to its solubility in that solution; but the results entirely agreed with the former ones, so that the sugar can be dispensed with.

(b.) *Estimation of Total Soda and Total Lime in Black-Ash.*—This estimation is desirable because it is a check on the proper mixing of the black-ash. Unfortunately, the most accurate methods for this purpose would not be of very much practical use, because the difficulty of getting a really representative average sample of a black-ash ball is almost insuperable, as Dr. Hurter has found in an extensive series of trials. All the more expeditious methods are in their place here.

As it is, at most works the soda contained in the solution of black-ash in the shape of  $\text{Na}_2\text{CO}_3$ ,  $\text{NaOH}$ ,  $\text{Na}_2\text{S}$  and  $\text{Na}_2\text{SO}_4$  is generally estimated, and the result is best reduced for our purpose to  $\text{Na}_2\text{SO}_4$ . The soda present in other soluble forms or in the insoluble state need not be taken into account, or else a certain quantity is allowed for it once for all. We shall thus get the *total soda*, and we have only to make a special test for *total lime*, which we do as follows:—Together with the samples taken out of free lime, we take out two or three samples of the well-agitated *muddy* liquid for our present purpose, exactly in the same way as prescribed above. Each sample is put into an Erlenmeyer flask, mixed with a few cc. of strong hydrochloric acid and boiled till all the gases have been expelled. A drop of methyl-orange is now added, and sodium carbonate solution is run in till the pink colour has just disappeared. Now 30 cc. of one-fifth-normal sodium carbonate solution is run in from a burette and the liquid is once more boiled to precipitate all  $\text{CaCO}_3$ . The small quantity of ferric oxide, alumina, magnesia, &c. equally precipitated, can be neglected here.

The liquid is washed into a 200 cc. flask, which is filled up to the mark, and 100 cc. of the clear portion is titrated with one-fifth-normal hydrochloric acid to find out the quantity of  $\text{Na}_2\text{CO}_3$  taken up by the lime present.

In four tests according to this method made by my assistant, Mr. Werner, the following quantities of one-fifth-normal acid were used (upon 30 cc. of sodium carbonate

solution):  $16.5 - 16.5 - 16.6 - 16.5$ , corresponding to  $30 - 16.5 = 13.5$   $\text{Na}_2\text{CO}_3$  used up =  $0.135$  gm.  $\text{CaCO}_3$ .

Only after all the samples intended for the tests (a) and (b) have been taken out, the original black-ash solution is allowed to settle down, and the samples for the estimations of sodium compounds are taken now. In the last-mentioned case the clear solution showed a litre of  $9.2 - 9.25 - 9.2$  cc. one-fifth-normal acid, equal to  $0.0975$   $\text{Na}_2\text{CO}_3$ , or originally  $0.130$   $\text{Na}_2\text{SO}_4$ , to which should be added the  $\text{Na}_2\text{SO}_4$  present as such, but not indicated by titration.

(c.) *Estimation of Available Soda in Alkali Waste.*—In alkali waste there is, apart from soda occurring in the insoluble form (which is but rarely estimated), always some soda present in a soluble shape; at the same time also, owing to the prolonged action of warm water on calcium sulphide, some calcium sulphhydrate or other soluble calcium compounds. Certainly such soluble compounds of calcium cannot exist at the same time with sodium carbonate; in presence of water they will interact and calcium carbonate will be precipitated, whilst sodium sulphhydrate, &c. will enter into solution. In the case of old alkali waste mud, calcium sulphhydrate, polysulphide, and thiosulphate will be in solution as well; even in the case of fresh waste possibly such calcium compounds, partially acting upon acids, may be in solution at the same time with sodium compounds indicated by acids, whilst on the other hand badly lixiviated waste will yield a solution containing an excess of  $\text{Na}_2\text{CO}_3$ , and consequently no soluble calcium salts at all.

These facts have been known all along, and it was always notorious that direct titration of an aqueous extract of alkali waste leads to entirely erroneous results. I have proved this directly in the "Chemische Industrie," 1881, 372, and in consequence of this a process was received into the "Pocket Book," consisting in evaporating the aqueous extract to dryness with addition of a little ammonium carbonate, igniting the residue, treating it with water and titrating the clear solution. This process is somewhat lengthy; what is worse, it is inaccurate, as pointed out to me by Dr. Hurter. The reason is probably that during evaporation some of the sodium sulphide is oxidised into thiosulphate, which of course escapes notice in the titration. Dr. Hurter communicated to me the method used at some Lancashire works, and consisting in treating the aqueous extract with carbonic acid, boiling the solution to decompose the calcium carbonate, and filtering and titrating the clear solution. I have examined this method with the following results (obtained by Mr. Zalscecki):—

Total alkalinity of the aqueous extract ( $\text{Na}_2\text{O} + \text{CaO}$ ) =  
 $45.3$  cc. one-fifth-normal acid.  
 $45.15$  " "  
 $45.2$  " "

After one hour's treatment with gaseous  $\text{CO}_2$  (less is not sufficient), boiling and filtering:—

$26.0$  cc. one-fifth-normal acid.  
 $26.2$  " "  
 $26.3$  " "  
 $26.15$  " "

After quarter of an hour's boiling with zinc carbonate and filtering:—

$26.1$  cc. of acid.  
 $25.95$  "  
 $26.05$  "  
 $26.5$  "

Hence the  $\text{CO}_2$  method yields quite consonant results, practically identical with those obtained even more quickly by zinc carbonate. Neither of these methods will yield absolutely true results, but such as suffice for purposes of comparing working results. The difficulty of getting a really average sample of waste is certainly one which can be hardly overcome.

(d.) *Estimation of the Available Soda in Commercial Caustic Soda.*—It is well known that caustic soda contains several sodium compounds which saturate acid on testing, and are hence indicated as available soda, viz., sodium hydrate, carbonate, silicate, and aluminate. Where it is important that the caustic should contain as little carbonate

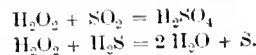
as possible, the  $\text{CO}_2$  is estimated and the  $\text{Na}_2\text{CO}_3$  is deducted from the total  $\text{Na}_2\text{O}$ . But there is a general, although but tacit, agreement to count the soda combined with silica and alumina as available soda, both because the quantities occurring in commercial caustic are exceedingly slight, and because the soda in these compounds is wellnigh all practical cases acts like the hydrate. This saves the somewhat tedious estimation of silica and alumina.

It does not seem as if this usage was to be attacked from any side; but Cross and Bevan (this Journal, 1889, 252) have drawn attention to the fact that when methyl-orange is employed as indicator, the test-acid saturates not merely the  $\text{Na}_2\text{O}$ , but also the  $\text{Al}_2\text{O}_3$  of the sodium aluminate, before the pink colour is produced, and this is a source of error. But apart from the fact that Cross and Bevan, as I have proved in the Zeits. f. angew. Chem. 1890, 300, erroneously contend that the change of colour takes place when the proportion  $2 \text{Al}_2\text{O}_3 : 5 \text{H}_2\text{SO}_4$  is reached (the real proportion being  $\text{Al}_2\text{O}_3 : 3 \text{H}_2\text{SO}_4$ ), I must also protest against their exaggeration of the real error pointed out by them, and their general condemnation of the use of methyl-orange for titrating caustic soda. From a large number of analyses, partly found in chemical publications, partly made by myself, I must conclude that the amount of alumina contained in ordinary commercial caustic soda is mostly quite inappreciable, and certainly never exceeds  $0.1$  per cent., so that it may be neglected in testing for alkali. The reason of this is, that in the manufacture of caustic the temperature in the end is kept so high that the compounds of soda with alumina and ferric oxide are split up again, and alumina and ferric oxide settle down at the bottom of the caustic pots. The "bottoms" are then laded out by themselves, and are mostly worked up again, but part of them is sold in that state, and as they contain about 2 per cent. of alumina, titration with methyl-orange would certainly give far too high a result. In testing caustic bottoms, therefore, methyl-orange must not be used, but otherwise there is no necessity for renouncing the use of this exceedingly convenient indicator.

#### 6.—SULPHUR RECOVERY. ESTIMATION OF SULPHURETTED HYDROGEN AND SULPHUR DIOXIDE IN THE SAME GASEOUS MIXTURE.

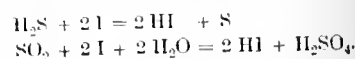
An analytical method for this purpose has become necessary, since in the Chance-Claus sulphur recovery process a gaseous mixture escapes containing small quantities of both  $\text{SO}_2$  and  $\text{H}_2\text{S}$ , together amounting to about 30 to 50 grms. S per cubic metre. Owing to the enormous amount of foreign gases, chiefly nitrogen, in spite of the presence of moisture no reaction ensues between  $\text{SO}_2$  and  $\text{H}_2\text{S}$ ; they must be dealt with by special absorbing apparatus, and hence also an analytical method for their estimation has become desirable.

Such a method had been devised by Mr. H. Crowther, chemist at Messrs. Chance Bros. He passes samples of the gases at the same time through solutions of hydrogen peroxide and of iodine. The former exerts the following reactions:—



That means:  $\text{SO}_2$  is converted into  $\text{H}_2\text{SO}_4$ , but  $\text{H}_2\text{S}$  does not form any acid, hence titrating the hydrogen peroxide by standard soda solution will indicate the  $\text{SO}_2$  alone.

On the other hand the reaction with iodine solution is in both cases such that each atom of S converts two atoms of I into HI:—



Thus the total sulphur is indicated, and  $\text{H}_2\text{S}$  is found by subtracting from it the S of  $\text{SO}_2$  found as above by means of  $\text{H}_2\text{O}_2$ .

Mr. Crowther's process has been introduced into all works where the Chance process is working, but complaints have reached me according to which the results are not accurate enough. In order to investigate this matter, I have, with the aid of Mr. Marchlewski, made a large number of experiments. It was found that in point of fact hydrogen peroxide

is not at all a good absorbent for  $\text{H}_2\text{S}$ , but at all events its acidity is not altered by that compound, whilst  $\text{SO}_2$  is easily and completely absorbed. This part of the process is therefore quite correct, indeed it has been employed for many years past by other chemists (especially Classen). The fault is in the other part, the estimation of total sulphur by iodine solution. We found that the gaseous current always carries away a certain quantity of iodine which makes the total sulphur and hence the  $\text{H}_2\text{S}$  to come out too high. But this can be avoided by placing behind the iodine an absorbing tube with caustic soda solution which retains the iodine, and whose contents are added to the iodine solution before re-titration. This addition then ought to be made at all events to the apparatus.

It is, however, preferable to proceed in a different manner, viz., to leave out the treatment with hydrogen peroxide, to pass the gases simply through a bulb-apparatus containing iodine solution, followed by another apparatus containing a little standard caustic soda solution. At the end of the operation the contents of both apparatus are united.

The mixture ought to possess an acid reaction; if not it is acidified with a known quantity of standard acid. The unused iodine is then estimated by means of thiosulphate; now methyl-orange is added and standard soda is run in till neutrality has been established. This process yields all data required for estimating  $\text{SO}_2$  and  $\text{H}_2\text{S}$ , since the latter liberates only its equivalent of  $\text{HI}$ , but the former, besides its equivalent of  $\text{HI}$ , also an equivalent quantity of  $\text{H}_2\text{SO}_4$ —



If we call the difference between ccm. of iodine solution originally employed and that of thiosulphate used in re-titrating =  $m$ ,—the originally employed ccm. decinormal soda solution (which must be  $< m$ ) =  $n$ , and the quantity of soda solution used in the end for establishing neutrality =  $o$ , the quantity of sulphur present is found by the following expressions:—

$$\text{S in } \text{H}_2\text{S and } \text{SO}_2 = 0.0016 \text{ } m$$

$$\text{S in } \text{SO}_2 \text{ alone} = 0.0016 (n + o - m).$$

In order to test this process practically, we passed a mixture of  $\text{SO}_2$  with very much atmospheric air through iodine and soda, as above described, and ascertained then how far the results obtained by titration coincided with those obtained by gravimetric estimation of the sulphuric acid formed.

The results were in three separate experiments:—

—	Iodometrical Process.	Gravimetric Process.	Difference.
<i>a.</i>	Grm. 0.07186	Grm. 0.0730	Grm. + 0.00114
<i>b.</i>	0.0776	0.07806	— 0.00046
<i>c.</i>	0.0799	0.06913	— 0.00077

Experiment *a* showed sufficient, *b* and *c* excellent agreement between the two methods. Hence the method now proposed, which is both more accurate and more expeditious than that formerly used, may be safely recommended to all chemists concerned.

One or other of the above mentioned absorbing methods, that is either hydrogen peroxide or iodine, can be employed in the methods proposed by Zilkowsky, Jannasch, and others, for estimating the  $\text{SO}_2$  escaping in the roasting of spent oxide of gas-works, or of pyrites itself. Those authors proposed bromine for this purpose, and estimated the sulphuric acid gravimetrically, after destroying the bromine present in excess; but the reagents now proposed save the lengthy manipulation last mentioned, and substitute a titration for a gravimetric estimation.

## Glasgow and Scottish Section.

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Notices of Papers and Communications for the Meetings to be sent to the Local Secretary.

### SESSION 1890-91.

1891.

Jan. 5th (Glasgow).—Mr. H. Ballantyne. "On the Effect of Exposure, under Certain Conditions, upon some Constants of Oils."

Feb. 2nd (Edinburgh).—Dr. J. B. Readman. "The Manufacture of Phosphorus: Part III."

Mar. 2nd (Glasgow):—

(1.) Dr. E. J. Mills. "Destructive Distillation: Part II."

(2.) Messrs. R. T. Thomson and H. Ballantyne. "On the Revision of Constants Employed in the Analysis of Fats and Oils: Part II."

Meeting held in the Societies' Rooms, 207, Bath Street, Glasgow, on Monday, November 3rd, 1890.

MR. C. A. FAWSITT IN THE CHAIR.

THE CHAIRMAN said he was sorry to inform the members that their Chairman, Mr. Stanford, had been ordered abroad through ill-health, and would not be able to take any share in the work of the Society for some time. He was sure they were all very sorry at this circumstance, and would unite with him in the desire that the time was not far distant when Mr. Stanford would be able to return home restored to health.


At the beginning of another session he would remind them that the success of the meetings depended upon the members themselves. He hoped they would vie with each other in producing papers of merit, and not only so, but that they would influence their friends, whether members of the Society or not, to come forward and read papers. One part of the meetings, of which he would like to see more advantage taken, was the discussion. Although the paper contributed was ever so valuable few remarks were elicited. This he hoped would be remedied in part at least, by the removal of the meeting place to this smaller room, where they would feel more at home. Regarding the night of meeting, it had been changed from Tuesday to Monday, the latter being considered the most suitable.

## A MECHANICAL RETORT WITH CONTINUOUS FEED AND DISCHARGE.

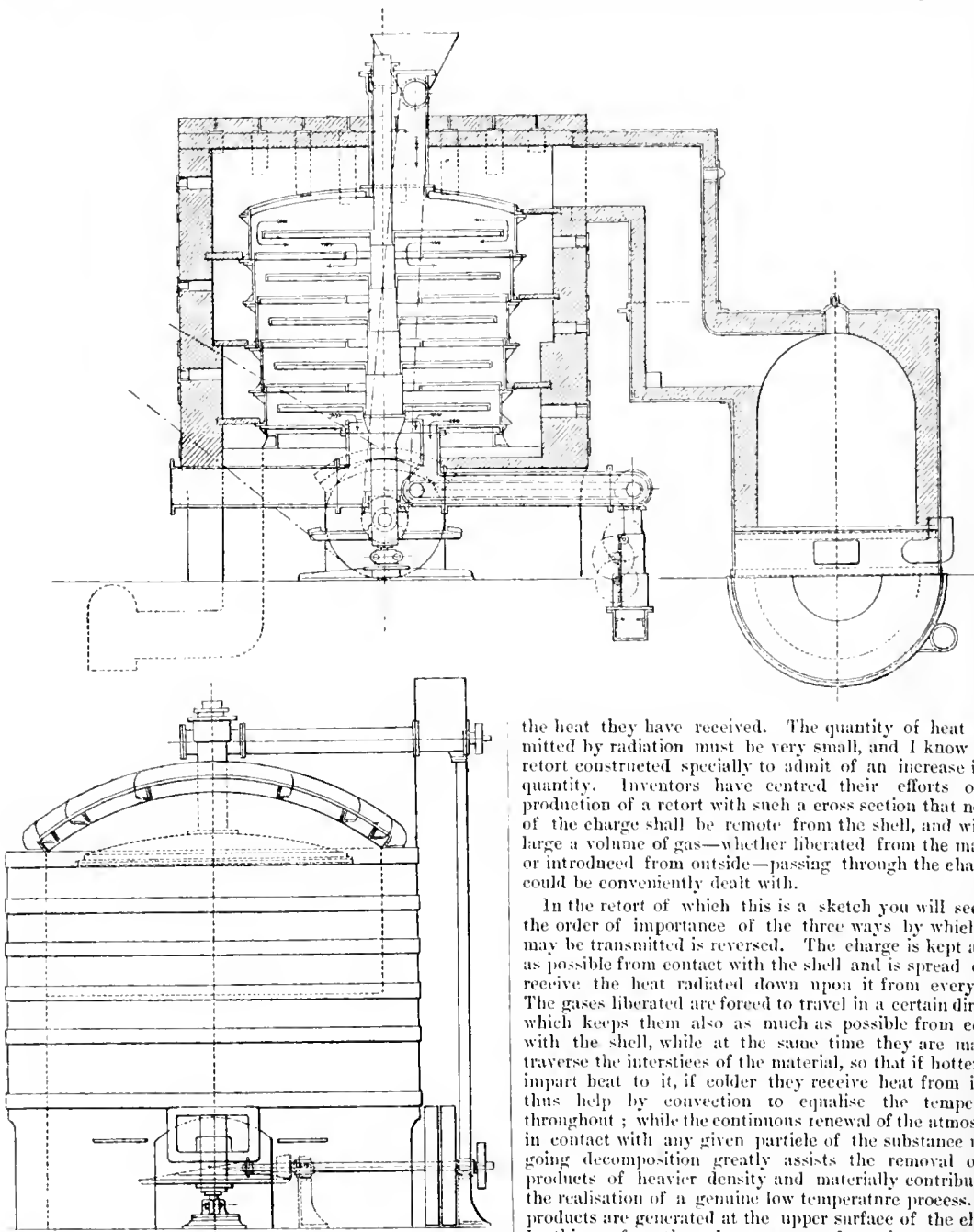
BY R. HAIG.

THIS retort, the patent of which belongs to the Mechanical Retorts Company, Limited, of Paisley, possesses, apart from its mechanical principle, a feature which should, I think, render it interesting to you from a scientific point of view. I refer to the manner in which it utilises the heat supplied to it.

A retort, in the ordinary conception of the term, is a vessel usually much longer than broad, and which may have its length disposed in a vertical, inclined, or horizontal

direction. The cross section may be round, square, -shaped, or oval. A furnace heats the outside, and the charge more or less completely occupies the inside. Heat passes by conduction through the shell of the retort to the contents, and upon the texture of these as well as on the temperature of the fire depends in a great measure the rapidity with which the charge is worked off.

The substances distilled are of low conductivity, and if they are charged into the retort in a state of fine division, or if they crumble during the distillation, the heat takes much longer to penetrate to the centre of the charge than when, owing to the state of coarser division of the material, there is more interstitial space. This, I think, is owing to the fact



the heat they have received. The quantity of heat transmitted by radiation must be very small, and I know of no retort constructed specially to admit of an increase in this quantity. Inventors have centred their efforts on the production of a retort with such a cross section that no part of the charge shall be remote from the shell, and with as large a volume of gas—whether liberated from the material or introduced from outside—passing through the charge as could be conveniently dealt with.

In the retort of which this is a sketch you will see that the order of importance of the three ways by which heat may be transmitted is reversed. The charge is kept as free as possible from contact with the shell and is spread out to receive the heat radiated down upon it from every side. The gases liberated are forced to travel in a certain direction which keeps them also as much as possible from contact with the shell, while at the same time they are made to traverse the interstices of the material, so that if hotter they impart heat to it, if colder they receive heat from it, and thus help by convection to equalise the temperature throughout; while the continuous renewal of the atmosphere in contact with any given particle of the substance undergoing decomposition greatly assists the removal of the products of heavier density and materially contributes to the realisation of a genuine low temperature process. The products are generated at the upper surface of the charge; by this surface they also escape, and are in no danger of having to penetrate a mass of material much hotter than themselves, as would be the case with gases generated in the middle of a charge heated by conduction from all round.

that in the latter case the gases evolved from that portion of the charge adjacent to the hot shell are free to travel throughout the rest of the mass, and by convection distribute

It is only during, say one-tenth of the period of distillation that the charge is in contact with a part of the shell directly heated by the furnace gases, and those furnace gases are at this point on their way to the chimney, and being almost spent can impart but little heat, while the charge having finished nine-tenths of its course through the higher or radiation-heating part of the retort is almost completely decomposed and in the condition least suitable for absorbing conduction heat; it may therefore be said that this plays no part in the distillation effected by our retort.

I think the idea of a mechanical retort probably first suggested itself to someone who distilled small materials, and who found the strain on the shell of the retort, owing to the high temperature at the end of an operation, very destructive.

It was natural to suppose that some arrangement by which the charge could be stirred up and each particle brought in its turn within the influence of the heated shell would obviate this disadvantage. With some materials the difficulties to be overcome were not very serious, and there are many retorts with mechanism inside or outside of them, which appears very simple and little likely to be deranged by the heat, which have been patented; yet when it came to treating a substance difficult of manipulation when hot and of a delicate constitution chemically, the users of those retorts appear to have lost confidence in them.

I have seen a horizontal cylindrical retort, capable of being rotated about its axis and consequently with all its mechanism outside, which, after a dozen charges had a skin of coke three or four inches thick adhering to the inside of the shell, caused not by the decomposition of gaseous hydrocarbons like the carbon coke in a gas retort, but by the crumbling and melting of the substance with which it was charged, and which was deposited layer by layer as the retort revolved. As this deposit increased in thickness the fire required to be urged, the distillate was less good, more coal was burned, the time to exhaust a charge greatly increased, and the chipping which was done after a thorough cool down was effected with considerable difficulty.

If this retort had been fitted with internal mechanism of the ordinary screw or chain type the result would not have been greatly different, and I think the defect lies in allowing the substance to rest on a surface heated from underneath by the furnace, so that decomposition begins below, and the superincumbent charge increases the tendency to adhere not only by its weight but also by preventing the free evolution of the vapours.

Our retort, as you see by the drawing, consists of a vertical cylinder, with a diameter of eight feet at the bottom, which is increased by three inches at regular intervals till it is nine feet wide at the top and seven feet six inches high. Both top and bottom are closed by discs, each having a large hole at the centre. Projecting upwards from the top disc and downwards from the bottom disc are sleeves which pass through the flues and brickwork to the open air, and which are fitted at their extreme ends with stuffing glands, in which the centre shaft, driven from below by gearing, revolves. The sleeves are much wider than is necessary for the passage of this shaft. It is through the upper one that the substance to be distilled is charged, the products being removed by the lower one. The centre shaft, like the shell, is also stepped, but instead of increasing towards the upper end it diminishes in diameter. The steps or shoulders are arranged at different levels from those on the shell, so that the discs which rest on them are exactly midway between those which rest on the shell. The discs rest alternately on the shaft and on the shell; those on the shaft revolve with it, those on the shell are stationary. The discs on the shaft do not quite reach to the shell, but are smaller in diameter by about a foot, the discs on the shell have an opening in their centres leaving an annular space round the shaft. From snugs cast on the under side of each plate are hung scrapers or deflectors which act like ploughs. Those hung from a fixed disc but working on a revolving disc, face towards the shell. Those hung from a revolving disc but working on a fixed disc, face towards the shaft. All are arranged to deflect the material on the respective discs, outwards from the shaft

and inwards from the shell. The material is put into the hopper on top either from hutchies on an over-head railway or by means of a bucket elevator from the ground, and is thence forced by a screw to the branch on the upper sleeve; here it falls down alongside the shaft on to a moving plate near its centre. As this plate moves slowly round the material forms a ring upon it. This ring is interrupted by a deflector hung from the top cover which pushes the material aside as fast as it comes round, and makes a larger ring while another small ring is being laid down. The large ring is again expanded into one still larger by another deflector, and so on till the outermost deflector pushes it over the edge of the disc on to the fixed disc below. As this deflector is itself fixed, the material falls on to the fixed disc at a fixed spot, and if left alone would pile up there, but certain deflectors hung from the edge of the moving disc and which (for simplicity, I will say) do not quite reach down to the fixed disc, are continually knocking the top off this heap and extending the material in a ribbon close to the shell. Here rings continue to be formed as before, but each succeeding one is lessened in diameter by a deflector hung from a moving disc, till all the material is pushed over the edge of the opening in the middle of the fixed disc. Here it arrives on the centre of a moving plate as at the first, and the cycle of movements outwards and inwards is repeated with each pair of discs till the material arrives at the bottom of the retort. It then falls down a shoot in the bottom sleeve to the chain conveyor, and is carried out over the large bevel wheel to the double valve apparatus by which it is discharged into a waggon or otherwise removed.

By the arrangement of deflectors described, you will notice that the whole charge is not kept in continual motion, but that only a little of it is being moved at any one time. Less power is therefore necessary for driving, and the deflectors move out of the way and leave the newly turned up substance fully exposed. The vapours generated or liberated travel in the same direction as the material, and pass to the condenser by the larger opening in the bottom sleeve. The heat, which may be supplied by a gas producer or furnace, according as one or other is suitable for burning the coal of the district, is first applied to the top of the retort, which is covered by an arch of special construction, and from thence passes by a zigzag course on both sides of the retort to the bottom, below which the flue gases escape to the chimney. You see that where the fire is hottest, the cold material is introduced, and when the material begins to decompose, the flue temperature is moderated, but always remains hotter than the material, and imparts heat to it. This might not, at first sight, appear a good arrangement for ensuring economy in coal consumption, for the flue gases, being above the decomposing temperature of the charge, convey a lot of heat to the chimney which might be used up in warming a fresh quantity of material to the distilling point; but it must be remembered that with the travel of the generated gases arranged as described, the temperature of volatilisation of the heavier products is lowered undoubtedly, and it is possible that that of decomposition may, by the favouring conditions, be so as well; so that, though the hottest fire were applied below the retort, and the flue gases more thoroughly cooled by passing over the cold material above, it is doubtful if any economy would be gained, and it is certain that the equality of temperature at present existing, and which is valuable, would be lost. The number of discs may be varied to suit the nature of the substance to be distilled, but I think need not exceed 12.

The retort at present in use at our Paisley works has only eight plates, and on a consumption of less than  $2\frac{1}{4}$  cwt. of coal per ton of wood, puts through an average quantity of 12 tons per day of 21 hours. The wood is principally birch in the form of shavings from the bobbin makers. The retort represented by the sketch in your hands was sent abroad to distil shale; it has 10 plates, and also puts through 12 tons per day, but the coal used, which was of very poor quality, amounted to about 4 cwt. on the ton of shale; with Scotch coal I am sure this figure would be much lower.

The shell of the retort is made of cast iron 1 in. thick, and the discs, which are strengthened by ribs cast on their



underside, have a thickness of  $\frac{3}{4}$  of an inch, the total weight of an eight-plate machine is about 20 tons, and the machine work necessary is trifling. The brickwork is simple, but I would like to draw your attention to the vault on the top, which is made so as to throw no strain on the side walls. Fireclay blocks with a cheek at each end are built into a "tee" iron curved to the circle of the outside of the vault and turned in at the ends, so that when the whole is lifted by a muzzle bolted to the web of the "tee" iron the bricks retain their position. Eight or ten of those arches of varying length when placed side by side cover the whole of the retort, and as the irons are not exposed to heat they bind the structure very firmly.

I regret I have been unable to show you a sketch embodying our most recent improvements, but the tracing handed round shows plainly the theoretical principles on which we work. The improvements I refer to are merely in the construction and enable us to have the castings made more cheaply. We now have the fixed and moving discs respectively made of one diameter, so that when shipped abroad one spare disc of each kind may be sent as a precaution against accident.

The discharging apparatus at the bottom of the retort is also simplified.

That the theory upon which the construction of the machine is based is a correct one is proved, not only by the small amount of coal used, but also by the excellent quality of the distillate produced. The wood used at our works, which, I believe, is stoved-dried before being cut up, can contain no superfluous moisture to increase the quantity of distillate, and this quantity, which amounts to 140 galls. per ton is, as far as I have been able to find out, much greater than that produced from air-dried billets commonly used in fixed retorts. The specific gravity of the liquor after settling is about 1.09 or 1.8 Tw., showing that it contains much hydrocarbon matter; it shows by test between 13 and 14 per cent. of glacial acetic acid. The yield of charcoal is 4 cwt., and the amount lost by difference as incondensable gas is only 2 cwt.

The retort which treated shale produced a full yield of oil of an excellent quality, much better than that from fixed retorts. The exhaust steam from the engine which drove it was, without being superheated, passed into this retort.

The power required for driving is very little, and by calculation from the breadth and speed of the belts amounts to three-horse power for the wood retort and five for the shale one.

We have treated two or three tons of seaweed with our machine and the iodine left in the residue was greatly in excess of that usually found. The quantity unfortunately was too small to afford a test of the capacity of the retort for this purpose.

#### DISCUSSION.

The CHAIRMAN said that the wood-distilling industry was one of great importance, and a paper connected with the subject was very acceptable. This industry had suffered much in recent years by competition from America and Germany. The Americans distilled an immense quantity of wood—mainly for the charcoal—and sent to this country acetate of lime and wood-spirit in such quantities as to make their manufacture here unremunerative. This retort, however, seemed as if it would help manufacturers to produce these articles more cheaply than could be done by the other retorts in use. If this was so, it would supply a want long felt by wood distillers. There was also a wide field before it for distilling shale, if it distilled economically, and the wear and tear were not excessive.

As far as he remembered, the original patent was in the name of a Mr. Lyle. Would Mr. Haig say whether this retort is a modification of that one or a new patent altogether? The wear and tear of these retorts had always appeared very heavy, and it would be an advantage if Mr. Haig could tell them what was the amount per ton of wood distilled. He should like to ask also what was the size of the shale treated, and how the temperature of this retort compared with that of the stationary retorts? Then he should like to know, too, what was the percentage of acetic acid, wood spirit, tar, &c. as compared with that which comes from other retorts?

Mr. Brown asked if Mr. Haig had ever tried this retort on alkaline liquors, and if not, how he thought it would do for that purpose?

M. J. PATTISON asked if the products contained more tar than those obtained by the old process, and if the quality of the charcoal produced was as good as, or better, than by the old one?

Dr. G. G. HENDERSON said that, with regard to the temperature of the retort, he gathered that the upper part was at a much higher temperature than the lower part. Could Mr. Haig give them the average difference of temperature between the upper and lower parts, and also could he inform them whether any experiments had been made as to the effect of varying differences of temperature between the top and bottom of the retort? He would also like to know if Mr. Haig could inform them as to the different percentage of the various products obtained at different temperatures, as compared with the ordinary retorts?

Mr. R. HAIG, in reply, said that at first the retort had been very difficult to work, though the actual wear of moving parts was nil. The trouble was caused by the faulty construction of the shell, which began to leak after a few months' work. This was now entirely obviated by having it made in rings instead of segments, so that no strains were thrown on the joints. With a tight shell there is no danger of anything going wrong.

The shale referred to was broken to the usual size for road metal.

The liquor produced from wood contained 13–14 per cent. of glacial acetic acid, which was allowed to be a very good yield.

The retort had never been tried for recovering waste soda lyes of paper makers, but if these were first concentrated to a proper consistency, it would certainly carbonise the organic matter they contained, and the soda might be lixiviated out of the residue. It would no doubt be an advantage to have the organic matter destroyed without exposing the soda to the sulphurous gases of the furnace.

The charcoal is discharged from the retort the moment it is spent, and, being produced at a low temperature, is very suitable for gunpowder making. Much of it is used for insulating the refrigerating chambers in which meat is brought to this country and stored on arrival; it is very light, and is unaffected by damp or dry rot. It is chiefly produced from birch, and this is known to yield more tar than some hard woods, but the retort produces so much more liquor per ton than is got by the old way that a slight increase in the tar which settles out is immaterial.

The question about the difference in results got by varying the difference of temperature which exists between the top and bottom of the mechanical retort was probably suggested to Dr. Henderson by the known arrangement of Young and Beilby's retort, which has practically two chambers, one kept at a higher temperature than the other, the one producing ammonia and the other oil, two distinct chemical operations. The retort described to-night, although to a certain extent divided by the discs, acts as a single chamber at a uniform temperature, but this temperature may be varied over all to suit the nature of the decomposition required. Wood and shale, which are worked for the distillates, require a low temperature, which is ensured by feeding the material thickly into the retort. The effect of a higher temperature may be readily shown by driving the retort faster and the feed slower, so that the charge passes through in a thin stream. Neither shale oil nor pyroigneous acid produced under such conditions is as good as that made in the first method of working, but it is conceivable that some substances might do better with a somewhat high temperature. The usual practice is to have redness just visible in the second flue from the top. The actual temperatures of the evolved or flue gases have not been measured, but it is possible to preserve great uniformity in these by changing the speed of the engine which drives the retort to suit variations in the condition of the fire.

## Obituary.

### EDWARD CHAMBERS NICHOLSON, F.C.S.

A MEMBER OF THE SOCIETY OF CHEMICAL INDUSTRY.

In the year 1845 the Royal College of Chemistry had been but comparatively recently founded by Professor A. W. Hofmann. In that year two of the Professor's first students were appointed by him to assist him in the pursuit of his earlier brilliant researches in organic chemistry. One of these was Edward Chambers Nicholson, the other was Frederick Augustus Abel, now Sir Frederick Abel. After a few years Nicholson became associated with the conduct of a small chemical manufacturing business, which, under his guidance and chemical supervision, speedily developed, and at length acquired, through his work, a very high reputation for the production of rare chemical compounds upon a manufacturing scale. Directly after the foundation to the coal-tar colour industry had been laid by the discoveries of W. H. Perkin, Nicholson produced upon a large scale and of a high degree of purity certain tinctorial coal-tar derivatives which speedily acquired great commercial importance, and thus brought fame as well as profit to his firm. Turning now to a series of lectures on "Coal-tar colours and on recent improvements and progress in dyeing and calico printing, embodying copious notes taken at the International Exhibition of 1862," by the late Dr. E. Crace Calvert, delivered in Manchester, we read "Most persons must have admired in the recent exhibition the beautiful crowns of acetate of rosaniline exhibited by Messrs. Simpson, Maule, and Nicholson, which presented in reflected light the green metallic lustre of cantharides' wings." Also further on we read "Roseine was first introduced into the trade early in 1860 by Messrs. Simpson, Maule, and Nicholson." Dr. Calvert later on refers to the once much-used "*Bleu de Lyon*," at first largely imported into this country, but, adds Calvert, "now manufactured by Messrs. Simpson, Maule, and Nicholson." It is indeed refreshing to note throughout the lectures how, through the influence of this great technological chemist, the firm, which he was raising to a position of eminence, not to speak of affluence, held its own, and more than held its own in the race with the best continental firms representing the coal-tar colour industry.

After a career as manufacturing chemist as brief as it was brilliant, Mr. Nicholson retired from business, and it has been a source of much regret to those acquainted with his exceptional powers as an investigator and originator of manufacturing processes, that he thought best to abandon the active pursuit of chemical science in the full tide of success, and when his continued labours could not have failed to be of important benefit to the chemical industries.

Edward Chambers Nicholson died on the 23rd of October last, after a brief illness, at the age of 63 years. It is not too much to say that to his remarkable talent for developing into successful manufacturing processes, intricate laboratory methods for the preparation of new and rare products, England chiefly owed the high position she held in the industrial world in the early days of the coal-tar colour industry. Nor should, in this connexion, the scientific enthusiasm or inspiration be forgotten, due to the teachings and ennobling influence of that master of scientific instruction and original investigation, A. W. Hofmann, an influence both felt and responded to by so many of his pupils during his directorship of the Royal College of Chemistry and subsequently in their later careers.—W. S.

## Journal and Patent\* Literature.

### I.—GENERAL PLANT, APPARATUS, AND MACHINERY.

*Lubricating Oils.* A. C. J. Charlier. The Engineer, 1890, 305—306.

See under XII., page 1043.

#### PATENTS.

*Improvements in Filters.* G. MacDonald, London. Eng. Pat. 14,972, September 6, 1889. 8d.

In this invention the filtering medium in the form of a compressed block is enclosed in a receptacle open at the top and perforated at the bottom, and is covered by a cap or bell, an annular space being left between the bell-cover and the receptacle. A connected series of these receptacles is placed in a portable vessel or cistern. The water to be filtered rises in the annular space, and flowing over the rim of the receptacle, passes through the filtering medium and perforated bottom, which communicates with the outlet pipe. By preference, the filtering medium is the "carbide material" described in Eng. Pat. 18,177 of 1888, and known as magnetic oxide of iron. Several modifications of the apparatus are shown on the drawing accompanying the specification.—E. S.

*An Improved Cooling Apparatus for Brewing and Domestic Purposes.* C. Huelser. From N. Reif, Hanover, Germany. Eng. Pat. 16,079, October 12, 1889. 8d.

See under XVII., page 1056.

*Improvements in Apparatus for Distilling and Concentrating Solutions.* J. Price, Liverpool. Eng. Pat. 16,520, October 19, 1889. 8d.

THE object is to construct the apparatus used in distillation and concentration so as to obtain the desired effects with great economy of fuel, and with little or no deposit of solid matter on the evaporating surfaces, and is effected by securing rapid flow of the liquid and maintaining it in contact with the heating surfaces. This is done by using metallic heating-tubes containing other tubes of non-conducting material, and having between the two tubes deflecting plates arranged preferably in a spiral form with an uniform or increasing pitch, so as to cause the liquid to flow continually in contact with the heating surfaces. The liquid to be treated is made to flow through the central tube, to return through the space between the two tubes (or *vice versa*). Suitable connections are fitted for the admission of the heating medium—for the liquid to be treated—for its transference to and from the several vessels, and also for the removal of the condensed water.—E. S.

*Improvements in Apparatus for Heating, Evaporating, and Condensing, applicable as a Feed-water Heater, a Distiller, or a Condenser.* W. W. Bengough, Forest Gate. Eng. Pat. 18,324, November 16, 1889. 11d.

THE apparatus belongs to that class where steam, used for heating and other purposes, is caused to pass through coiled pipes arranged within a suitable vessel, provided with inlet

\* Any of these specifications may be obtained by post, by remitting the cost price, plus postage, to Mr. H. Reader Luck, Comptroller of the Patent Office, Southampton Buildings, Chancery Lane, London, W.C. The amount of postage may be calculated as follows:—

If the price does not exceed 8d. ....	1d.
Above 8d., and not exceeding 1s. 6d. ....	1d.
" 1s. 6d., " " 2s. 4d. ....	1½d.
" 2s. 4d., " " 3s. 4d. ....	2d.

and outlet branches. In this invention, one or more sections of coiled pipe are used, each in the form of a double volute. The two volute coils of each section are in one piece, and may be made of solid-drawn copper tubing. Where several sections are employed, the outer ends of the uppermost and lowermost section are placed in connexion with steam inlet and outlet pipes. By this construction a large heating or cooling surface can be provided in a small space. The coiled pipe is arranged in a metallic vessel provided with inlet and outlet branches, and is connected with the vessel, so as to be easily disconnected if required. Suitable valves are provided for controlling the admission of steam to the coiled pipes, and of water and vapour, and their exit from the vessel. For details of construction and mode of working, the three sheets of drawings attached to the specification must be consulted.—E. S.

*An Improved Method or Process of and Apparatus for the Lixivation of Various Materials.* H. H. Lake. From "La Société Anonyme des Parfumes Naturels de Cannes," Paris, France. Eng. Pat. 18,528, November 19, 1889. 8d.

This invention relates to improvements in the method and apparatus described in Eng. Pat. 10,280 of 1889 (this Journal, 1890, 325), and is intended to dispense with manual labour, making the apparatus automatic by providing each extractor, either internally or externally according to circumstances, with a float and valve, which have the effect of permitting the passage of the solvent into each extractor either at the entrance or at the discharge, the passage of the gas from one extractor to the other being at the same time stopped automatically.—E. S.

*An Improved Cement used for Covering Boilers, Steam Pipes or Vessels containing Steam or Heated Water.* W. H. Loundsdale, Blackburn. Eng. Pat. 19,906, December 11, 1889. 4d.

The mixture consists of 4 cwt. of ground cork, 1 cwt. of paper pulp, 2 cwt. of charcoal, 3 cwt. of common clay, 3 cwt. of fireclay, 3 cwt. of cowhair or other animal hair or flax, 1 cwt. of soot, 3 cwt. of water, and may be applied as a covering to boilers, &c. in the usual manner.—B. B.

*Improved Process and Apparatus for Cooling Liquids by Means of Air.* J. Klein, Frankenthal, Germany. Eng. Pat. 11,246, July 18, 1890. 11d.

The improved apparatus devised for the above purpose consists mainly of a round or polygonal upright shaft or chamber in which water admitted from the top is brought into intimate contact with a current of air, produced by a fan, moving up in the opposite direction. Inside the shaft a number of plates, preferably of wood, and connected in two or more series, are so arranged that the plates of one series cross those of the other at a suitable angle. The liquid to be cooled is conveyed to the plates, and on reaching them is distributed into thin and even layers, and is brought into contact with the blast moving in the opposite direction. The air thus absorbs a portion of the heat and the liquid reaches the reservoir in a considerably cooled condition. (See also this Journal, 1890, 849.)—E. S.

*Improvements in Evaporating Apparatus.* S. M. Lillie, Philadelphia, U.S.A. Eng. Pat. 11,686, July 25, 1890, By Internat. Conv. December 26, 1889. 8d.

The invention relates to "Intransit Evaporating Apparatus," in which the liquid is evaporated while flowing in comparatively thin films over the heated evaporating surfaces. As regards a single pan or "effect" it consists of a battery of horizontal evaporating tubes, contained in an evaporating chamber, which tubes are closed at one end and at the other are connected with steam supply conduits, from which their interiors are supplied with steam. Above the evaporating tubes in the chamber is a spraying contrivance, fed with the liquid to be evaporated, and by

which it is distributed over the surface of the tubes, and then flows down over the same to the floor of the evaporating chamber. Means are provided to allow the escape of any gases that may collect in the tubes and other parts of the apparatus; and also for the concentrated liquid. For details of the mode of working it will be necessary to consult the drawings attached to the specification. There are 12 claims.—E. S.

*Improvements in Filters and Filtering Apparatus.* R. W. James, London. From E. J. Tobin and T. Davidson, Sydney, New South Wales. Eng. Pat. 13,029, August 19, 1890. 6d.

THE improvements are specially devised to provide increased effective capacity of the filtering medium, while the filter itself may be decreased in size. The invention is equally applicable to domestic and other filtering apparatus.

For a domestic filter a chamber is divided into two parts by a central partition preferably formed of limestone firmly cemented in it, and having an aperture through it allowing of communication between the upper and lower parts containing the unfiltered and filtered liquid. The filtering medium is made by cutting a deep groove or an annulus in the walls of a cylinder, also preferably made of limestone, the ends of the groove being firmly closed. On the foot of the cylinder is fixed a valve with a passage to the groove, and the cylinder is seated upon the partition with the valve entering the communicating aperture, and forming a tight joint. The liquid having filtered through the exterior and interior walls of the cylinder into the groove passes through the passage and valve into the lower part of the chamber, whence it is drawn off by a tap.—E. S.

## II.—FUEL, GAS, AND LIGHT.

*Commercial Condition of Persia.* Board of Trade J. November 1890, 604.

See under X., page 1040.

### PATENTS.

*Improvements in and Connected with the Manufacture of Illuminating Gas from Coal and analogous Material.* J. H. Fergusson, Liverpool. Eng. Pat. 15,947, October 10, 1889. 8d.

THESE improvements are in connection with the arrangements for re-heating or fixing illuminating gas after distillation. The re-heating ducts or fixing retorts are arranged in the upper part of a bench of distilling retorts, but separated therefrom by openings regulated by dampers, &c., for the entrance of the waste heating gases; the ducts may be disposed above the arch of a bench of retorts, and a suitable space formed round them through which the whole or part of the waste gases is made to pass on the way to the flue, the temperature being regulated to 1,000°—1,300° F. The fixing retorts may be single or U-shaped ducts, or they may have an artificially cooled pipe interposed in their course so that the gases are heated, cooled, and re-heated; in any case an artificially cooled pipe is provided on the duct outlet. When such a pipe is interposed between two branches of a fixing retort, tar may be introduced at that point, or it may be introduced at any suitable point of the duct whatever its form. The illuminating gases may be led directly from the retorts through a short valved conduit into the fixing retorts without any condensation of the tarry matters. In this case the gases are heated for a considerable time in passing through a long duct to a temperature not exceeding 1,000°—1,200° F., the junction of the duct with the cooling pipes being shielded from the heat.

Several modifications are described and illustrated by three sheets of drawings.—S. B. A. A.

*Improvements in Apparatus for burning Hydrocarbon or other Oils for Lighting and Heating purposes.* G. Rose, Glasgow. Eng. Pat. 18,101, November 13, 1889. 8d.

THESE improvements are developments of the author's previous patents, Nos. 11,517, 16,497, 17,826, of 1888, and are mainly applicable to that class of apparatus in which steam generated from water supplied to a coil pipe or chamber heated by the flame is used to spray the oil in the burner or combustion box. An oil tank or reservoir has a small water-tank secured to its exterior, interior or bottom, a pipe from the latter tank feeds a small coil pipe enclosed in a metal casing and secured to the upper surface of a broad flange surrounding the nipple of an oil-well burner similar to that described in Eng. Pat. 11,517 of 1888. Oil flows by gravitation through a valve-controlled tube into the oil well of the burner and is there ignited, and as soon as the coil is sufficiently heated steam is forced through a pipe connected with the top of the coil into the steam chamber of the burner, and the oil is forced out through the nipple of the burner in a fine spray. A branch of the steam pipe conveys a portion of the steam to the top of the water tank to produce the pressure necessary for feeding the coil. A valve is described which automatically regulates the water-supply according to the pressure in the tank; the admission of steam to the tank may also be regulated by a valve, and the water-supply pipe may have a syphon bend formed upon it for the purpose of retaining a supply of water in the coil.

A number of modifications and various details are described at length in the specification.—S. B. A. A.

*Improvements in Apparatus for Generating and Burning Gas from Heavy Oils or Carbonaceous Fluids.* W. G. Robinson, Elland. Eng. Pat. 19,121, November 28, 1889. 8d.

THESE improvements relate to the construction of the gas generator and burner of portable and fixed lamps in which hydrocarbon vapours are consumed, and are especially adapted to the apparatus of Eng. Pat. 3696 of 1889. In the improved apparatus, the generator is composed of three or more tiers of tubes, each tier consisting of three short tubes joined in a triangle, and communicating by means of short vertical tubes with the adjacent tiers. For convenience of cleaning, openings secured by screwed stoppers are formed in two of the angles of each tier. The oil is supplied from a reservoir into the lowest tubes of the generator, and the vapours are withdrawn from the uppermost tubes and conveyed to the burner, which is situated directly under the generator, and supplies it with the necessary heat. Pressure may be automatically produced in the reservoir by connecting the vapour exit tube with the upper part of the reservoir. The argand or air-mixing portion of the burner is of a hollow spherical or ovoidal form, with contracted upper and lower orifices, this form preventing undue fluctuation of the light from varying pressure in the reservoir.—S. B. A. A.

*Improved Methods of and Apparatus for Burning Liquid Hydrocarbons.* P. Humbert, Boston, U.S.A. Eng. Pat. 12,843, August 15, 1890. 8d.

LIQUID hydrocarbons, preferably crude petroleum, are converted into a mixture of methane and other gases by treating their vapours with atmospheric oxygen or with the gases from the decomposition of water by heat. The apparatus used consists of a volatilising coil provided with oil and air supply-pipes and with an exit pipe for the vapours connected with a burner placed at some distance below the coil. The coil and burner are enclosed by a cylindrical fireclay or iron shell, and two concentric cylinders successively decreasing in height are placed between the burner and the outer cylinder, and are separated by air passages. Gas issuing from the burner is thus supplied with air at different points, and complete combustion ensues. The coil may be protected from the direct action of the flames

by an internal shell partially closed at the top and provided or not with properly-regulated openings into the coil chamber. For supplying "decomposed water," two chambers or pipes divided into several communicating compartments, and filled with asbestos, are fixed between the volatilising coil and the burner, the water supply is connected with the lower end, and the gases led from the upper end into the volatilising coil.—S. B. A. A.

*Improvements in the Process of and Apparatus for Manufacturing Gas and for the Utilisation of the same.* J. T. Key, London. Eng. Pat. 12,860, August 16, 1890. 8d.

A VERTICAL gas-producer, preferably cylindrical over the middle portion and approximately conical towards the top and bottom, is fitted at the top with a supply hopper of rectangular section, and is fed with fuel by a longitudinally-grooved or recessed drum revolving in the lower end of the hopper. The producer has another rectangular opening at the bottom, bordered internally with side plates, against which the coke, ash or clinker is crushed, before withdrawal by a hollow longitudinally and transversely corrugated perforated drum, which revolves in the lower opening, and has its lower end immersed in water contained in a closed cistern beneath the producer. One wall of the cistern is inclined, and a portion of the water surface exposed, in order to facilitate the removal of the coke, but access of air to the drum is prevented by a curtain wall dipping into the water. The two drums are geared together to ensure corresponding uniformity in the rate of fuel supply, and of the coke discharge, and both drums have hollow axles. Air or steam is admitted into the producer through the hollow axle and perforations of the lower drum.

An improved atmospheric burner, especially suited for steam boilers heated by producer-gas, consists of a short central tube for the hot gas, which widens out and opens into a wider tube surrounding it and attached to it by stays. A core inserted and similarly attached to the other end of the outer tube, reaches almost to the open internal end of the gas passage. Air is supplied into the annular passage, so that the gas and air leave the core end of the burner and enter the flues (of a steam boiler) in concentric currents and not in intimate admixture; the heat of combustion is thus distributed along the whole length of the flues. Perforated firebrick blocks may be placed at intervals within the flues to mix the currents of air and gas more intimately, and to facilitate the absorption of the heat. The details are illustrated by two sheets of drawings.—S. B. A. A.

*Improvements in the Manufacture and Production of Artificial Fuel.* F. Lennard and E. W. Harding, Greenwich. Eng. Pat. 13,033, August 19, 1890. 4d.

SEVEN parts by measure of powdered coal or coal and coke or breeze, two parts of green or spent tan, and one part of coal-tar pitch are thoroughly ground and incorporated in a mechanical mixer, rendered plastic by treatment with superheated steam (340° F.) in a pug mill and compressed in perforated blocks. The proportion of tan may be varied from five parts to one part, and the proportion of pitch from three parts to one-half part. It is alleged that by the addition of tan the ignition of the fuel is sufficiently retarded to ensure perfect combustion.—S. B. A. A.

### III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

*Report on Carbolic Acid and Acids not Specified.*  
Report of the Central Laboratory of the Italian Customs Department, 1886—1889, 302—306.

THE tariff report distinguishes between carbolic acid which contains not more than 20 per cent. of light oil, distilling up to a temperature of 310°, classified as carbolic acid; 2nd, that which contains from 20—30 per cent. of light oil, classified as heavy mineral oil; and 3rd, that which contains above 30 per cent., classified as crude mineral oils (see Report on Oils). For the purpose of analysis, according to these definitions, the direction add to 100 grms. of the sample twice its volume of a 10 per cent. caustic soda solution; the light oil, which separates after some time, is then fractionally distilled.

The report classifies under carbolic acid both the crystallised acid (*Acidum carbolicum crystallisatum*) and the liquid acid (*Acidum carbolicum liquefactum*) under the title of pure carbolic acid.

*Benzoic Acid.*—A distinction is drawn in the tariff between "benzoic acid or flowers of benzoïn," classified as perfumery, and "benzoic acid of every other sort," classified as acids not specified. The compilers remark that such a distinction is only possible when the acid from various sources has not undergone refinement sufficient to make it absolutely pure. It is proposed to distinguish between benzoic acid from gum benzoïn and that obtained from urine, phthalic acid, &c., as follows—1st, a small portion of the sample is sublimed, when the former yields no residue, while the latter gives a small residue of lime; 2nd, when heated with potash, the acid from urine gives off ammonia; and 3rd, in the acid obtained from trichloro-toluene, traces of chlorine can be recognised by the copper oxide test (Jacobsen, *Archiv. der Pharm.* 1884, 366).—V. H. V.

*Report on Bitumen, Pitch, &c.* Report of the Central Laboratory of the Italian Customs Department, 1886—1889, 412—416.

THE tariff mentions "solid mineral tar derived from the distillation of fossil carbon and mineral oils, i.e., black, liquid residues from the same source, and pitchy residues, which do not serve for illuminating purposes or for the extraction of heavy oils." These are defined as "thick pitchy residues of a very deep colour and penetrating odour which remain in the retorts after distillation, and which serve as substitutes for tar and solid bitumen." These are subject to the duty of solid bitumens: liquid pitches, however, which can be worked for the heavy oils, are classed as "liquid mineral tar, derived from the distillation of fossil carbon and mineral oils," and are subject to the duty on heavy oils (q.v.). The harder varieties of pitch used for asphalts and artificial bitumens or for the manufacture of briquettes defined as "combustible agglomerates," are free of duty.

Vegetable tars mentioned in the report under the title of natural gums, resins, &c., are free of duty, while tars prepared for medicinal purposes are classified under the title of compounded medicines.

Residues obtained in the distillations of resins are classed as solid bitumens and subject to a duty of 50 lira per quintal; natural bitumens such as those of Judea, &c., are placed under the same heading, but bituminous earths such as those of Val de Travers are classed as earths, stones, &c. In order then to decide between natural and artificial bitumens the Direction adopted the methods of Durand-Claye (*Annales des ponts et Chaussées*, 1879, 122).

Bituminous schists, "which serve to increase the illuminating power of coal gas" are classed as solid bitumens; in this list boghead coal is included. Natural carbons such as fossil carbon, lignite, and wood carbon which have undergone processes, which would not prevent their employment for purposes other than combustibles, are classified under different headings in the report.—V. H. V.

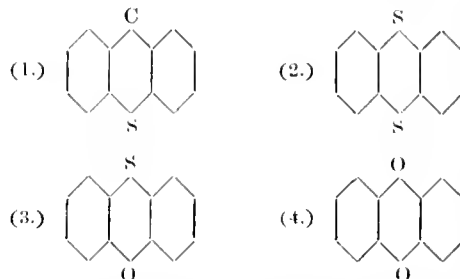
### IV.—COLOURING MATTERS AND DYES.

*A Method for the Production of Aromatic Sulphides and of Thioxanthone.* T. H. Ziegler. *Ber.* 23, 2469—2472.

RESEARCHES in colouring matters have proved that most of them can be derived from a system of two or more rings.

Antraquinone, xanthone, acridine, phenazine, phenoxazine, and thiodiphenylamine form the foundations of a great many colouring matters distinguished by their fastness and beauty.

The author tried to prepare bodies of the following constitution:—



Beckmann has long ago prepared a derivative of a body constituted according to formula No. 1 by the action of sulphuric anhydride on benzophenone; but thioxanthone could not be obtained from it.

Leuckart and Sandmeyer indicated simultaneously how thiophenols could be prepared. By the action of diazo compounds on these bodies, however, no azo bodies, but sulphides are obtained. Diazobenzene chloride and sodium thiophenolates form, for instance, phenyl-sulphide. The sulphides from diazo-*o*-toluene, diazo-*p*-toluene, diazo- $\beta$ -naphthalene and thiophenol form oils distilling from 290° to 310° C. Phenyl- $\alpha$ -naphthylsulphide crystallises in thin plates, melting at 49° C.

*p*-diazoacetanilide combines with thiophenol and forms *p*-acetamidophenylsulphide melting at 144° C., which, on saponification, yields the amidodiphenylsulphide crystallising from alcohol in needles, melting at 97° C. The preparation of thioxanthone is performed in three steps. By acting with diazoanthranilic acid on sodium thiophenolate diazothiophenyl ether is formed. On treatment with alkalis the sodium salt, diphenylsulphide-*o*-carbonic acid is obtained. The free acid is insoluble in water, but easily soluble in benzene, alcohol, and glacial acid, crystallising from benzene in white plates, melting at 165.5° C. It is soluble in concentrated sulphuric acid with a magnificent light green fluorescence, forming the ketone after a few minutes' heating. On pouring the sulphuric acid solution into an excess of cold water, thioxanthone is precipitated. It is soluble in hot glacial acetic acid, carbon bisulphide, and benzene, and crystallises in yellow needles, melting at 203°—205° C. On distillation or sublimation and crystallisation from benzene, white crystals are obtained, melting at 207° C., and boiling from 371°—373° C. under a barometric pressure of 7.5 mm.—A. L.

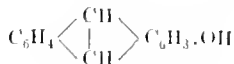
*The Action of Sulphur on Organic Compounds and the Synthesis of Tetraphenylthiophen.* T. H. Ziegler. *Ber.* 23, 2472—2476.

THE action of sulphur on diphenylmethane originated the idea of examining benzene derivatives containing a methyl or methylene group. Two mols. of phenylacetic acid were heated with one mol. of sulphur. At 220° C. evolution of sulphuretted hydrogen begins, which ceases after six hours, the temperature having gradually risen to 260° C. The product of the reaction is a thick fluid, easily soluble in benzene and carbon bisulphide, though little in alcohol. After purification, white microscopic needles were obtained, melting at 181°—182° C. They are tetraphenylthiophen. The same body can also be prepared by the action of

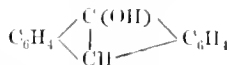
sulphur on dehydroxybenzoïn. It is very stable towards chemical agents; on treatment with concentrated nitric acid, however, a tetranitro-compound is formed. The stability of the hydrogen atoms in the benzene ring towards sulphur is very remarkable. Mixtures of benzophenone and azobenzene with sulphur are not acted upon even on boiling them. Triphenylmethane behaves in a like manner, no sulphuretted hydrogen being evolved, even on heating it with sulphur to 300° C. In other cases it is easily movable, as the formation of the *o*-hydroxythiophenols, of thiodiphenylamine, and of other bodies proves. In all these reactions the replacement of the benzene hydrogen is due, according to the author's views, to the presence of a basic group. Of special interest is the action of sulphur on toluidine, and experiments prove that Plüztzinger and Gattermann's views of the constitution of dehydrothiitoluidine and of the primuline base are correct.—A. L.

*On Meso-anthramine.* F. Goldmann, Ber. **23**, 2522—2526.

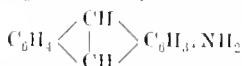
Of the monohydroxy derivatives of anthracene two are known: Anthrol—



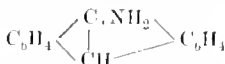
and Anthranol—



Of the corresponding amines only anthramine—



has been described. The author has prepared its isomeride, which he calls *Meso-anthramine*—



by heating finely-powdered anthranol with 20 times its weight of a concentrated aqueous solution of ammonia containing 25 per cent. of ammonia for 12 hours at 200° C. The product of the reaction consisted of yellow crystals and a yellow solution. The crystals were dried on porous plates and dissolved in alcohol. An alcoholic solution of hydrochloric acid was added and the hydrochloride of the new base precipitated. Ammonia precipitates the free base, which is purified by solution in cold alcohol and precipitation with water. It forms golden plates, decomposing at 115° C. It is very unstable and darkens even in the desiccator, is insoluble in water, soluble in alcohol, ether, chloroform, and benzene, with a yellow colour and green fluorescence. On oxidising it with a solution of chromic acid in glacial acetic acid *anthraquinone* is produced. Diazobenzenesulphonic acid produces with the alcoholic solution of meso-anthramine-hydrochloride a beautiful magenta colouration, from which a red colouring matter, probably an azo dyestuff, separates. It is insoluble in alcohol and water, but dissolves in dilute ammonia.

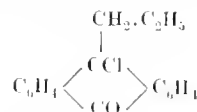
*Acetylmeso-anthramine.*  $\text{C}_{14}\text{H}_{13}.\text{NH}.\text{C}_2\text{H}_3\text{O}$ , obtained by the action of cold acetic anhydride on the base, forms colourless needles melting at 273°—274° C.

*Diacetylmeso-anthramine.*  $\text{C}_{14}\text{H}_9\text{N}(\text{C}_2\text{H}_3\text{O})_2$ , was obtained by boiling the monoacetyl compound for two hours with acetic anhydride. It forms colourless plates melting at 159° C.

*Meso-anthraminehydride.*  $\text{C}_{14}\text{H}_{11}\text{NH}_2$ , was prepared by boiling an alcoholic solution of meso-anthramine with sodium amalgam and precipitating the solution, after the reaction is finished, with water. The new base forms colourless needles melting at 92° C. and soluble in alcohol, benzene and ether. It forms salts with acids. On boiling the aqueous solution of the hydrochloride of the hydrated base it decomposes and forms anthracene and ammonium chloride,  $\text{C}_{14}\text{H}_{11}\text{NH}_2\text{HCl} = \text{C}_{14}\text{H}_{10} + \text{NH}_4\text{Cl}$ .—A. L.

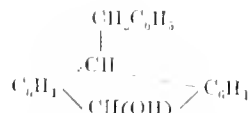
*Benzylloxanthranol.* C. Bach. Ber. **23**, 2527—2530.

*Benzylloxanthranol chloride*—

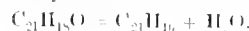


obtained by the action of phosphorus pentachloride on dry benzylloxanthrol forms colourless plates melting from 95°—102° C.

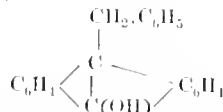
*Benzylidihydroanthranol*—



was obtained from benzylloxanthranol by reduction with zinc dust and ammonia. It forms yellowish needles decomposing at 130—140° C. On boiling it with acetic acid it decomposes into benzylanthracene and water—

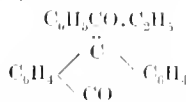


*Benzylanthranol*—



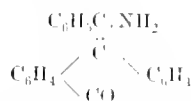
was prepared by treating bromobenzylene anthrone, dissolved in alcohol, with the calculated amount of sodium amalgam. Yellow needles melting at 183—184° C., soluble in alcohol, ether and glacial acetic acid were obtained.

*Ethylhydroxybenzylene anthrone*—



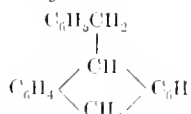
is formed when bromobenzylene anthrone is treated with sodium ethylate. It forms yellow plates melting and decomposing at 173°—175° C., soluble in ether, alcohol, benzene and glacial acetic acid.

*Amidobenzylene anthrone*—



is obtained by treating a solution of bromobenzyleneanthrone in benzene with dry ammonia. It is an orange amorphous body, melting at 150—152° C.

*Benzylanthracenedihydride*—



is the final product of the reduction of benzylanthracene. It crystallises in prismatic needles, soluble in alcohol, and melts at 110—111° C.—A. L.

*On Triphenylamine Derivatives.* R. Herz. Ber. **23**, 2536—2542.

*Nitrotriphenylamine* is prepared by treating a solution of triphenylamine in glacial acetic acid with a mixture of nitric and glacial acetic acid (3 : 5), finally heating to 45—50°. It crystallises from acetic acid in golden yellow needles

melting at  $139^{\circ}$ – $140^{\circ}$ , and dissolves readily in the usual solvents. With concentrated sulphuric acid a deep blue colour is produced. By reduction with tin and hydrochloric acid, the tin double salt of *Amidotriphenylamine* may be prepared. The free base readily oxidises in the air, being coloured a reddish-violet. The acetyl derivative is also described.

*Dinitrotriphenylamine* is obtained in a similar manner to the mono-nitro derivative by treating with a somewhat stronger mixture (equal parts) of glacial acetic and nitric acid and afterwards heating to  $55^{\circ}$ – $60^{\circ}$ . After recrystallising from acetic acid, fine yellow needles melting at  $206^{\circ}$ – $207^{\circ}$  are obtained. Concentrated sulphuric acid colours it violet-blue. By reduction with tin and hydrochloric acid *Diamidotriphenylamine* is readily prepared. After recrystallising from very dilute alcohol pointed needles separate which melt at  $187^{\circ}$ , being decomposed at the same time. The diacetyl derivative is prepared in the usual manner.

*Trinitrotriphenylamine* is prepared in the same way as the dinitro derivative, the temperature being finally raised to  $100^{\circ}$ . It separates at once in bronzy yellow needles and the yield is quantitative. The melting point after boiling in glacial acetic acid, in which the compound is almost insoluble, rises to  $280^{\circ}$ . Heydrich some years ago states that by oxidising a glacial acetic acid solution of the hydrochloride of triamidotriphenylamine with chloranil, he obtained a safranine reaction. The author could not arrive at the same result, always obtaining instead deep blue to violet induline-like colourations. However, on dissolving 0.0005 grm. of the base in 5 cc. concentrated sulphuric acid, containing a trace of fuming nitric acid, a deep green solution resulted, changing to dark blue on warming. On now adding water drop by drop to the cooled solution, the latter gradually assumed a brilliant red fluorescence which soon changed to violet and then magenta-red.

By the action of diphenylamine on pieryl chloride in toluene solution *Diphenylaminapieryl chloride* may be prepared. It crystallises from dilute alcohol in ruby-red needles melting at  $65^{\circ}$ – $66^{\circ}$ .

*Sodium triphenylaminetrisulphonate*.—Triphenylamine is gradually introduced into an excess of fuming sulphuric acid at  $60^{\circ}$ . The sodium salt is with difficulty soluble in dilute alcohol.

*Diphenylnaphthylamine* is prepared by adding 3.5 grms. of potassium to a boiling solution of 20 grms. of diphenylamine and 15 cc. of aniline. 20 grms. of  $\alpha$ -bromonaphthalene are now gradually introduced, and after boiling for a quarter of an hour the mixture is fractionated. When all aniline has distilled over, the pressure is reduced to about 80 mm. At  $230^{\circ}$ – $260^{\circ}$  some unchanged diphenylamine first distils over. The portion distilling at  $335^{\circ}$ – $340^{\circ}$  is dissolved in boiling acetone precipitated with alcohol and recrystallised from dilute alcohol, when silky needles are obtained, melting at  $142^{\circ}$ . Diphenylnaphthylamine dissolves in concentrated sulphuric acid with a green colour and on warming, in contradistinction to triphenylamine, a sulphonic acid is at once formed.—A. R.

*The Constitution of Dinitro- $\beta$ -Naphthol.* E. Loewe.  
Ber. 23, 2542–2546.

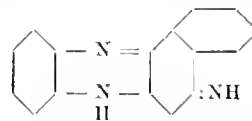
By oxidation of dinitro- $\beta$ -naphthol Graebe and Drews obtained  $\beta$ -nitrophthalic acid (1:2:4), which proves that in nitrating  $\beta$ -naphthol, one nitro-group enters into each nucleus of the latter. Moreover, the nitro-group which is contained in the non-hydroxylated nucleus must occupy the  $\beta$ -position, but which  $\beta$ -position is uncertain, and it has not been shown in what position the other nitro-group is in.

This research was undertaken with a view of ascertaining these facts. *Diamido- $\beta$ -naphthol* was first prepared by reduction with tin and hydrochloric acid, and after distilling the base, the hydrochloride was obtained in almost colourless needles by addition of concentrated hydrochloric acid. The base itself oxidises very readily in the air. The triacetyl and tribenzyl derivatives of this compound are also described. *Diamido- $\beta$ -naphthylamine* may be prepared by reducing the corresponding nitro derivative described by Graebe and

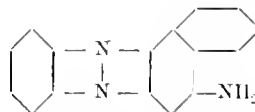
Drews. The latter was first obtained in a very pure state by extracting with xylene; it was then reduced with tin and hydrochloric acid, and the base after distillation converted into the hydrochloride with concentrated acid. For determining the position of the nitro-group in the hydroxylated nucleus of dinitro- $\beta$ -naphthol the author tried whether a condensation between diamido- $\beta$ -naphthylamine and phenanthrenequinone could be effected. A eurhodine was indeed obtained, soluble in hydrochloric acid with a reddish-brown colour, and in concentrated sulphuric acid with a violet colour, which turns brown on diluting with water. It sublimes readily and its ethereal solutions show a brilliant fluorescence. The formation of the body is a proof that in dinitro- $\beta$ -naphthol one nitro-group is in the ortho position to the hydroxyl group. A solution of the question which of the two  $\beta$ -positions in the non-hydroxylated nucleus is occupied by the other nitro-group had to be abandoned for the present, owing to the very small yields of diamidonaphthalene obtained from the above-mentioned dinitro- $\beta$ -naphthylamine by diazotising and reducing. The former base the author proposes to convert into a dichloronaphthalene by Sandmeyer's method, whence conclusions may eventually be drawn as to the position of the two nitro-groups in dinitro- $\beta$ -naphthylamine, and consequently also in dinitro- $\beta$ -naphthol.—A. R.

*Oxidation Products of Orthodiamines and Ortho-amidophenols.* O. Fischer and E. Hepp. Ber. 23, 2787–2789.

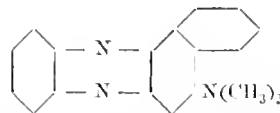
KERMAN (this Journal, 1890, 934) believes  $\alpha$ -amido- $\alpha$ -naphthylazine to be—



whilst the authors consider it as a true amido compound—



Experiments have proved that it can be diazotised and that on treatment with hydrochloric acid a phenol is formed. Furthermore, eurhodines have been prepared from  $\alpha$ -phenylenediamine and benzene-azo-ethyl-naphthylamine and benzylazodimethylnaphthylamine. The formation of the eurhodine—



proves that amidonaphthophenazine really does contain an amido group.

The reaction of  $\alpha$ -phenylenediamine on amines proceeds in different ways according to the nature of the amines. Amido-azo-naphthalenes take part in the reaction, whilst amido-azo-benzenes act mostly as dehydrogenating agents.  $\alpha$ -phenylenediamine and amido-azo-benzene hydrochloride form principally diamidophenazine whilst  $\alpha$ -amidophenol and amido-azo-benzene hydrochloride form triphenioxazine.

—A. L.

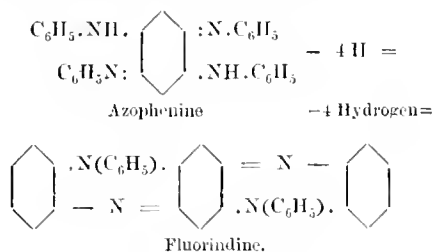
*Fluorindines.* O. Fischer and E. Hepp. Ber. 23, 2789–2793.

FLUORINDINES are produced by the action of heat on azophenines, by the action of  $\alpha$ -phenylenediamine hydrochloride on its red oxidation product. They are further contained in nearly all induline melts.

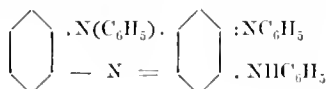
*Fluorindine from Azophenine* is formed by continuous treatment of azophenine. A violet colouring matter is formed as intermediate product. It is also obtained by



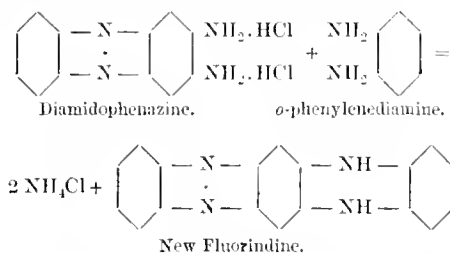
heating azophenine with 2—3 parts of zinc dust. It can be easily isolated, as it is nearly insoluble in alcohol, ether, benzene, toluene, and chloroform, but it imparts to alcohol a beautiful fluorescence. The red-violet solution appears in reflected light as fiery red, whilst addition of hydrochloric acid produces a greenish-blue solution with a brownish-red fluorescence. Fluorindine is a very stable body, resisting the action of concentrated hydrochloric acid even at 250° C. Its melting point could not be determined as it partly sublimes, a bluish-violet vapour being evolved. Analysis gave the formula  $C_{30}H_{20}N_4$ , and its constitution is probably explained in the following equation—



It is a weak base and therefore a weak colouring matter. The intermediate violet product is probably an induline,  $C_{30}H_{23}N_4$ , of the constitution—



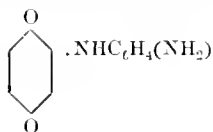
*Fluorindine from Diamidophenazine.*—The red oxidation product of *o*-phenylenediamine is diamidophenazine. If four parts of its hydrochloride are heated with three parts of *o*-phenylenediamine in an oil-bath for 10–15 minutes to 200°–210° C., a black powder is obtained, which is treated with boiling dilute sulphuric acid. The green sulphate of the new fluorindine remains as a greenish powder, which yields on boiling with alcoholic ammonia the free base as a brownish-violet powder, insoluble in nearly all solvents. By treating it for days together in an extraction apparatus with benzene-alcohol, plates are obtained of a greenish lustre, imparting to alcohol a beautiful yellowish-red fluorescence. Its formula is  $C_{18}H_{12}N_4$ , and its constitution is explained by the equation—



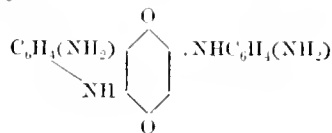
The authors call the new body *Homofluorindine*.—A. L.

*The Action of Quinones on o-Diamines and o-Nitro-aniline and Nitroparatoluidine.* J. Leicester. Ber. **23**, 2793—2798.

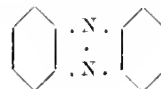
ANILINE acts on quinone, forming quinone-anilide. Ortho-diamines ought therefore to produce bodies of the type—



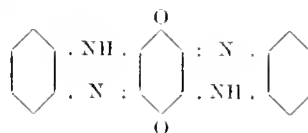
or of the type—



Perhaps the reaction might go further, and quinonephenazine

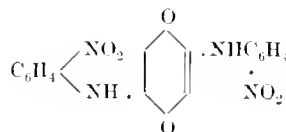


or quinonefluorindine—

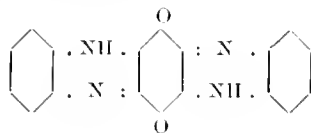


might be produced. On carrying out these experiments a mixture of substances was obtained, the purification and separation of which were very difficult. The experiments were therefore simplified by using orthonitro-amido compounds instead of *o*-diamido bases.

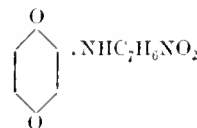
*Quinone-o-dinitro-anilide* was obtained by boiling 3 parts of quinone with 2.5 parts of *o*-nitro-aniline in a solution of glacial acetic acid for two hours. On cooling, red crystals separate from the solution, which after crystallisation from alcohol form red needles melting and decomposing at 305° C. They consist of



On reducing this compound with alcoholic ammonium sulphide at 100° C., colourless crystals are obtained, which, however, turn blue-black in the air. They were recrystallised from a mixture of methyl-alcohol and benzene, and form dark green crystals looking exactly like homofluorindine. They have the same blue colour and fluorescence in a solution of glacial acetic acid; they are, however, not identical with homofluorindine, but contain oxygen. The formula of the new body is—

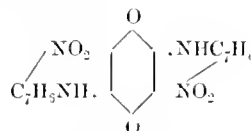


*Quinone-*o*-nitrotoluide*—

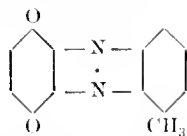


forms red crystals decomposing at about 300° C.

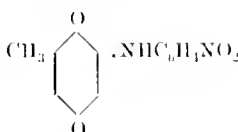
*Quinone-o-dinitrotoluide—*



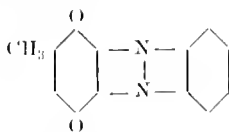
forms bronze-coloured plates decomposing at 140° C

*Quinone- $\alpha$ -methylphenazine—*

was obtained from the monitrotoluidoquinone by reduction with alcoholic ammonium sulphide at  $100^{\circ}\text{C}$ . It forms bronze-coloured crystalline powder, soluble in alcohol with a violet colour; its solution in glacial acetic acid is greenish blue, and shows a weak red fluorescence.

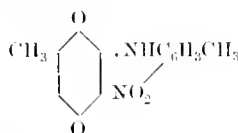
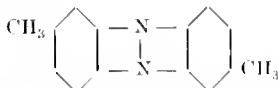
*Toluquinone- $\alpha$ -nitroanilide—*

obtained from toluquinone and  $\alpha$ -nitroaniline, forms red crystals decomposing at  $230^{\circ}\text{C}$ . On reduction it yields

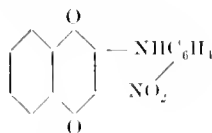
*Quinonephenotolazine—*

crystallising in red leaves.

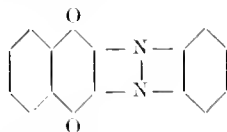
The author prepared further—

*Toluquinonenitrotoluide—**Quinonetolazine—*

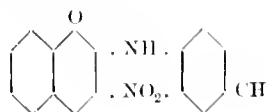
dark red leaves, soluble in alcohol and in glacial acetic acid.

 *$\alpha$ -Naphthoquinone- $\alpha$ -nitroanilide—*

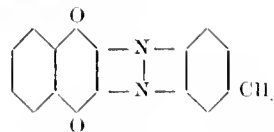
yellow crystals.

 *$\alpha$ -Naphthoquinonephenazine—*

green plates from alcohol.

 *$\alpha$ -Naphthoquinonenitrotoluide—*

orange needles.

 *$\alpha$ -Naphthoquinonetolazine—*

steel blue plates with a greenish lustre.—A. L.

*On the Constitution of the so-called Carbazines.* M. Freund and F. Kuh. Ber. **23**, 2821—2844.

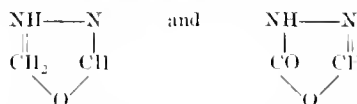
FURTHER researches on the action of phosgene gas on the acid derivatives of phenylhydrazine have disclosed the fact that the compounds originally described as derivatives of a hypothetical compound, carbazine—



must be regarded from a totally different point of view. It appears that the derivatives of phenylhydrazine are capable of reacting in two distinct ways, either in accordance with the usual formula,  $\text{C}_6\text{H}_5\text{—NH—NHCO}R$  or its tautomeric one,  $\text{C}_6\text{H}_5\text{NH—N}=\text{C(OH)R}$ . In the case of the action of phosgene gas the latter formula is considered the most likely by the authors, and to the hypothetical basis of the compounds produced they assign the formula—



Hantzsch having introduced the term “azole” for nuclei in which one  $\text{CH}_2$  group is replaced by a diad, the authors propose to call the last-mentioned compound “biazole,” whereas the two substances represented by the formulae—



are called respectively biazoline and biazolone. The reasons for coming to the above conclusions regarding the constitution of the compounds hitherto known as carbazines are:—

1. Only one molecule of phosgene gas reacts with diphenylcarbazine, the older view requiring two.
2. The behaviour of the acetyl derivatives towards phosphorus pentachloride prove the presence of only one, not two, movable oxygen atoms.
3. Certain of these bodies do not react with carbon oxysulphide which is not in accordance with the older views on the constitution of these substances. For the details of this research, which are unsuited for abstracting, the original article must be consulted.—A. R.

*Tetramethylbenzidine and its Dye-stuff Derivatives.* II. von Perger and F. Ulzer. Mittheil. des k. k. Tech. Gew.-Museums, 1890, 34—43.

THE authors tried to prepare tetramethylbenzidine by Michler and Pattinson's method, which consists in heating together under pressure benzidine, methyl iodide, and methyl alcohol, and decomposing with soda-lime the resultant methyl ammonium iodide. They found the yield, however, exceedingly small and the method consequently of no technical value. A better mode of preparation is based on a reaction observed by Michler and others (Ber. **12**, 1171, and **17**, 115), namely, the production of tetramethylbenzidine by heating dimethylaniline with sulphuric acid. In employing this reaction, the authors obtained the best results by the use of fuming sulphuric acid containing a small percentage of sulphuric anhydride. A 30 per cent.

yield of base was obtained when 25 parts of dimethylaniline were heated for eight hours at  $210^{\circ}$  with 85 parts of acid containing 4 per cent. of anhydride. In another experiment, using a larger proportion of acid, a 40 per cent. yield resulted, and, in this instance, 20 per cent. of unchanged dimethylaniline was recovered, so that technically a 50 per cent. yield may be said to be obtained. The base was separated from the acid mixture by pouring the latter into water, neutralising, removing the unchanged dimethylaniline by a current of steam, precipitating the tetramethylbenzidine as hydrochloride, clarifying a solution of this salt with animal charcoal, decomposing with ammonia, and purifying the free base by a single recrystallisation from alcohol, which renders the base pure enough for experimental purposes.

The dinitro-derivative (melting point  $188^{\circ}$ ) was prepared by adding the calculated amount of sodium nitrite to a hydrochloric acid solution of the base and crystallising from alcohol. Michler and Pattinson state that an orange-coloured compound is thus formed. The authors, however, find that a green nitroso-compound, melting at  $140^{\circ}$ , is first formed, and that this on exposure to the air is oxidised to the orange nitro-derivative. An isomeric dinitro-compound (melting point about  $167^{\circ}$ ) is found accompanying the last-mentioned compound. This the authors intend to further examine. Endeavours to effect the nitration with gaseous nitrous acid, as in the case of nitro-alizarin, and with nitric acid, were unsuccessful, it being found impossible to control the reaction.

Diamidotetramethylbenzidine was obtained by reducing the dinitro-compound above mentioned (melting point  $188^{\circ}$ ) with tin and hydrochloric acid, and was separated with ammonia, after the tin had been precipitated with hydrogen sulphide. Recrystallised from alcohol it closely resembles in appearance tetramethylbenzidine, and melts, as observed by Michler and Pattinson, at  $168^{\circ}$ . The yield is almost the theoretical. From the diamido-base so obtained, a range of tetrazo-dyes was prepared by diazotising and combining the tetrazotetramethyldiamidodiphenyl solution formed, with the following naphthol- and naphthylamine-sulphonic acids, the results being tabulated below:—

	Colour yielded by Dyestuff on Silk or Unmordanted Cotton.
1. Naphthionic acid ( $\text{NH}_2:\text{SO}_3\text{H} = 1:1$ ).....	Brown-yellow.
2. Naphthylamine sulphonic acid— ( $\text{NH}_2:\text{SO}_3\text{H} = 1:5$ ).....	Brown-yellow.
3. $\beta$ -naphthylamine sulphonic acid— ( $\text{NH}_2:\text{SO}_3\text{H} = 2:6$ ?—Brönmér's).....	Brown-yellow.
4. $\beta$ -naphthylamine- $\delta$ -sulphonic acid.....	Red.
5. $\beta$ -naphthylamine- $R$ -disulphonic acid.....	Pale brown-red.
6. $\beta$ -naphthol- $\beta$ -sulphonic acid— ( $\text{OH}:\text{SO}_3\text{H} = 2:6$ ).....	Chocolate-red.
7. $\beta$ -naphthol- $\alpha$ -sulphonic acid (Bayer's croceic acid).....	Dyeing power very feeble.
8. $\beta$ -naphthol- $R$ -disulphonic acid.....	..
9. $\beta$ -naphthol- $G$ -disulphonic acid.....	..

In view of the small yields of some of the intermediate products and consequent expensive nature of the diamido base, the dyes formed, to be technically available, would require to be of unusual intrinsic value, which it is evident they are not. It remains to be seen whether an isomeric diamidotetramethylbenzidine will yield more valuable dyes.

—E. B.

*On the Formation of Anthraquinone under peculiar Conditions.* L. H. Friedberg. J. Amer. Chem. Soc. **12**, 26—43.

LACBENZHEIMER'S phenanthrene-quinone reaction (Ber. **8**, 224) has been shown by Victor Meyer to be due to  $\alpha$ -methylthiophen,  $\text{C}_6\text{H}_4\cdot\text{S}\cdot\text{CH}_3$ , which is contained in all commercial toluenes. The reaction is carried out as follows:—A few drops of the toluene are added to a dilute solution of phenanthrene-quinone in glacial acetic acid. The mixture is cooled continually, and drops of concentrated sulphuric acid are allowed to run into it. After a few minutes water is added, when a colouring substance is precipitated, which on shaking with ether dissolves with a beautiful blue tint. This colouring matter appears to have the formula  $\text{C}_{19}\text{H}_{12}\text{OS}$ . (V. Meyer, "Die Thiophengruppe," 40), being formed according to the equation—



It is a dark blue powder, which on pressure assumes a copper lustre. It is easily soluble in alcohol, benzene, chloroform, and carbon bisulphide, but is insoluble in water. When heated alone it does not undergo any change, but in presence of lead chromate, anthraquinone is formed. This pyrogenetic change is remarkable, and since the green colouring matter obtained by the action of phenanthrene-quinone upon thiophen does not yield it under analogous conditions, it appears likely that the methyl group of the thiophen has to do with the formation of the anthraquinone.

The author enters into a detailed theoretical explanation of the manner in which the anthraquinone might be formed, in which he assumes the intermediate formation of thiophen aldehyde from the blue colouring matter, which then loses  $\text{CO}_2$ ,  $\text{H}_2\text{S}$ , and  $\text{COS}$ , and undergoes certain molecular rearrangements to form anthraquinone. The suppositions are not supported by any experimental data, but the author intends to test their correctness by crucial experiments.

—C. A. K.

*Report on Colours.* Report of the Central Laboratory of the Italian Customs Department, 1886–1889, 371–378.

**I. Coal Tar Colours**, distinguished as "colours derived from coal-tar in the dry state, in paste or as liquids," are free of duty. Among extraneous substances added were found dextrin, sugar, magnesium and sodium sulphates, and sodium chloride, often to such a degree as to modify the tariff regulation; thus samples, declared as coal-tar colour, consisted principally of an aqueous solution of sugar, coloured with a trace of colouring matter; these were classed as syrups, earminic acid (q.v.). Solutions of caramel and coal-tar colours were classed with "confectionery and preserves with sugar and honey."

**Coloured Extracts from Dye-Woods** pay 12 lire per quintal. Samples were sent of vegetable colours to which coal-tar colours had been added either for the purpose of fraud or of beauty; these were classed under the former category and liable to duty. Samples were also sent of extract of logwood sophisticated with extract of chestnut; for their examination the method proposed by Houzeau (Compt. Rend. **68**, 716) was adopted.

**Colours in Cakes, Powder, and of every other kind** pay 12.50 lire per quintal; among exceptions are included certain coloured earths, ochres, natural and artificial, classed as "coloured earths," also certain metallic oxides such as ferric oxide, minium, &c., and certain metallic compounds, mercury sulphide, lead carbonate, &c., specially classed. Coloured lacquers are classed as colours in cakes.

Samples declared as a solid coal-tar colour were recognised as a mixture of barium and calcium sulphates, coloured with a coal-tar colour; another declared as chemical products not specified, was a mixture of lead chromate (lead vermillion) and barium sulphate, while a sample of lead chromate (chrome yellow) was declared as a coal-tar colour.

—V. H. V.

## PATENTS.

*Manufacture of Indulin-like Colouring Matters.* O. Imray, London. From "The Farbwerke Griesheim-am-Main, W. Noetzel and Co.," Germany. Eng. Pat. 17,204, October 30, 1889. 4d.

A process for obtaining reddish-violet, reddish-blue, and grey-black colouring matters by reacting on primary aromatic amines with the hydrochlorides of nitrosodimethylaniline or nitrosodiethylamine together with hydrochloric acid and at temperatures between 90° and 150° C. 30 parts of aniline are heated to 90° together with 15 parts of hydrochloric acid of 20° B. and 25 parts of nitrosodimethylaniline are gradually added. A violent reaction occurs, after which the melt is raised to 120° when it becomes thick and solidifies on cooling. To purify it, it is powdered and dissolved in dilute hydrochloric acid, from which solution the dyestuff is precipitated with salt.—T. A. L.

*Process for the Preparation of Orange-Yellow Colouring Matters.* O. Imray, London. From "The Actien Gesellschaft für Anilin Fabrikation," Berlin, Germany. Eng. Pat. 17,957, November 11, 1889. 4d.

AN extension of Eng. Pat. 15,296 of 1885 (this Journal, 1886, 595, and 1889, 701) and of Eng. Pat. 2213 of 1886 (this Journal, 1887, 138, and 1889, 701). The dyestuffs referred to in the previous patents are obtained by the action of diazotised benzidine or toluidine on  $\beta$ -naphthylamine disulphonic acid R and phenol. Owing to the presence of the free hydroxyl group in the dyestuff it is sensitive to alkalis, but this can be prevented, as described in the present patent, by methylating or ethylating the hydroxyl group by heating the colouring matter with ethyl bromide or methyl iodide in dilute alcohol and in presence of caustic soda. The reaction takes place when the mixture is boiled under an inverted condenser and when completed the alcohol is distilled off and the dyestuff precipitated with salt.—T. A. L.

*Improvements in the Manufacture and Production of Direct-Dyeing Yellow Colouring Matters.* B. Willeox, London. From "The Farbenfabriken vormals F. Bayer and Co." Elberfeld, Germany. Eng. Pat. 18,354, November 16, 1889. 6d.

THE dehydrothiotoluidine melting at 191° C. (obtained by the action of sulphur on *p*-toluidine) or its sulphonic acid when diazotised can be combined with another molecule of dehydrothiotoluidine or with dehydrothiotoluidine sulphonic acid in an acetic acid solution. The mixture is allowed to stand several days at 20°–30° C., and is then raised to 60°–80°, and kept at this temperature until a sample dissolves to a clear solution in soda. The whole is then boiled, neutralised with soda, and the colouring matter separated in the usual way. The so-called prinnuline base or its sulphonic acids, and the bodies obtained by the action of sulphur on *m*-xylydine or  $\psi$ -cumidine may also be employed in a similar manner. The products dye unmordanted cotton greenish yellow. (See also this Journal, 1890, 854.) —T. A. L.

*Improvements in the Manufacture or Production of Azo Colouring Matters.* B. Willeox, London. From "The Farbenfabriken vormals F. Bayer and Co.," Elberfeld, Germany. Eng. Pat. 18,517, November 19, 1889. 4d.

AN extension of Eng. Pat. 13,665 of 1889 (this Journal, 1890, 854). The dihydroxynaphthalene monosulphonic acid there described, when combined with diazo compounds preferably in an acetic acid solution, gives colouring matters of a bluer shade than those obtained from the naphthol sulphonic or carboxylic acids.—T. A. L.

*Improvements in and Connected with the Manufacture and Production of Direct-Dyeing Colouring Matters for Dyeing and Printing.* B. Willeox, London. From "The Farbenfabriken vormals F. Bayer and Co.," Elberfeld, Germany. Eng. Pat. 18,519, November 19, 1889. 6d.

THESE dyestuffs are produced by first combining one molecule of tetrazodiphenyl chloride or tetrazoditolyl chloride with one molecule of *o*- or *m*-cresol carboxylic acid by which means an intermediate compound is produced. This then combines with another molecule of a phenol or amine producing new colouring matters. 20 kilos. of benzidine sulphate are converted into tetrazodiphenyl chloride and run into 11 kilos. of *m*-cresol carboxylic acid dissolved in weak acetic acid. The solution is then made slightly alkaline with soda and after standing some time is added to an acetic acid solution of 25 kilos. of  $\beta$ -naphthylamine disulphonic acid R. After about 12 hours the mixture is heated to 80° C., and made alkaline with soda lye. The colouring matter thus obtained is salted out and separated in the usual way. Various other amines and phenols may be employed in place of the  $\beta$ -naphthylamine disulphonic acid mentioned.—T. A. L.

*Process for the Manufacture of Picric Acid from Crude Phenols.* W. P. Thompson, Liverpool. From E. de Lom de Berg, Brussels, Belgium. Eng. Pat. 18,904, November 25, 1889. 6d.

CRUDE phenol is treated with sulphuric acid, in proportion to the amount of crystallisable phenol contained, the sulphonic acid dissolved in water and then nitrated. The cresol is said to be unaffected by this treatment. When the reaction is completed the whole is concentrated to the consistency of a syrup and treated with sulphuric acid so as to convert any remaining cresol into its sulphonic acid, thus rendering this body very soluble. The picric acid remaining after the product of the reaction has been taken up with water is purified by recrystallisation. Another process is to conduct the nitration, after sulphonating as above, so that liquid nitrocresol results, which can then be converted into tri-nitrocresol, whilst the decanted liquor is further treated with nitric acid to form pure picric acid.—C. A. K.

ERRATUM.—In this Journal, 1890, 933, for "Cyanamides" throughout the abstract read "Cyanamines."

## V.—TEXTILES: COTTON, WOOL, SILK, Etc.

*A New Fibre in Lagos.* Board of Trade J. November 1890, 596.

THE Lagos Weekly Times for the 30th August publishes a minute by Sir Alfred Moloney, Governor of Lagos colony, on the bass fibre of the bamboo palm (*Raphia vinifera*) known in Yoruba as the *Igi Oguro*. This production promises to become an important and valuable addition to the exports from Lagos. The following information is extracted from the memorandum in question:—

The "African bass" is one of the commonest native fibres of this colony—used by every fisherman in the manufacture of his lines, and prepared from one of the most plentiful of the palm trees of the colony, the *Raphia vinifera*, or "bamboo" palm.

The "African bass" is in appearance a stiff and wiry fibre, varying in colour from dark brown to light red, dependent for its shades on duration of soaking. It is most readily obtained in lengths of from 3 ft. to 4 ft., beyond which length it is inconvenient to pack, and difficult to procure without injury to the tree. In diameter it varies from  $\frac{1}{16}$ th to  $\frac{1}{32}$ th of an inch, the latter of which may be

accepted as the limit of fineness to be admitted in a commercial sample for the European market. It is said to be used mainly in the manufacture of hard brushes for various domestic and manufacturing purposes. The demand appears to be very large, and the price is exceedingly satisfactory. The source of its supply in this colony alone may be said to be practically inexhaustible.

### PATENTS.

*Improvements in the Treatment of Vegetable Fibrous Material to obtain Fibre therefrom, and in Apparatus therefor.* G. W. Robertson, Glasgow; D. Black, Lenzie; and J. McGlashan, Edinburgh. Eng. Pat. 12,626, August 10, 1889. 8d.

THE bark (raw or green bark as well as "ribbons") of the fibre yielding *Urticaceæ*, viz., rhea, ramie, china grass, &c., is first soaked in cold or hot water, or in a weak solution of caustic soda or other alkali for 6–12 hours, until thoroughly softened. It is then passed through a machine, having heavy iron rollers, in order to break the outer skin, to remove impurities and the liquid, and to straighten the fibres. One roller travels faster than the other, whereby a slight draft on the fibres is produced. Then the material is wound in ribbons on bobbins or reels with hollow and perforated centres, each ribbon being laid between webs of wire, so that the liquids may have free access to every part of the material. A number of these bobbins is mounted on vertical spindles inside a keir or boiler, containing a solution of caustic soda or other alkali from 2°–4° Tw. strength, and boiled under steam pressure for an average of 12 hours, the bobbins being suitably rotated on their spindles. Then the alkaline liquid is run off and may be used in a dilute state for the preparatory bath mentioned above. After that the material is carefully washed with hot or cold water. Next the bobbins are unwound, the fibres passed through rollers, dried, conducted through a series of heckling machines, and then carded, combed, or otherwise treated preparatory to spinning.—H. S.

*Improved Apparatus for and Method of Extracting Oleaginous and other added Matters from Fibrous, Spun, Textile, and other analogous Substances.* E. Mansfield, London. Eng. Pat. 14,364, September 12, 1889. 8d.

AN arrangement of apparatus for subjecting dirty cotton waste or similar material to alternate squeezing and expansion whilst under the action of an alkaline liquid, for the purpose of removing grease and dirt.—K. E. M.

*Improvements in or Connected with the Manufacture of Carpets, Matting, Twine, and the like from Esparto Grass.* J. E. Richardson, Carlisle. Eng. Pat. 15,135, September 25, 1889. 4d.

ESPARTO grass is put into a tank and water added for maceration. Caustic soda or potash may be added, about 5 to 15 pounds to each ton of the material, to facilitate the maceration; in some special cases dilute mineral acids, added in the same proportions, serve the same purpose. The time of maceration varies considerably, viz., from 7 to 30 days, when water only is used, and from 3 to 14 days if the above chemicals are employed. When the maceration is complete, the chemicals and impurities have to be removed by thoroughly washing with water for a further period of one or several weeks. Then the grass is taken out and passed through rollers, care being taken that it does not become too dry. After this it is subjected to the action of wooden beaters which process partly disintegrates the fibre; it is then passed through grooved steel or iron rollers, and lastly through smooth rollers which complete the disintegration, then it is carded and spun into rope, twine, or yarn. The goods, when made, are passed through hot rollers to finish them.—H. S.

*Improvements in the Treatment of Textiles by Means of Ammoniac Copper for the Purpose of making them Waterproof and Uninflammable.* C. Baswitz, Berlin, Germany. Eng. Pat. 16,708, October 22, 1889. 6d.

See under XIH., page 1047.

*Improved Means and Apparatus for Ungumming and otherwise Treating Flax and other like Fibres, also Leaves and Plants.* E. Johnson, London. From T. Burrows, Lille, France. Eng. Pat. 17,137, October 29, 1889. 11d.

THE loose materials are placed in cages which are fitted in an air- and steam-tight shell or boiler, these cages having either a rotary or an up-and-down motion. The shell is half filled with water or other suitable liquid, and then steam is admitted, to obtain pressure at the upper part of the shell. By the alternate action of the liquid and of the steam, a complete ungumming or retting is effected in a short time, without injury to the fibres.—H. S.

*A New or Improved "Proofed" Fabric.* T. Stoward, London. Eng. Pat. 17,629, November 5, 1889. 4d.

FOR a long time past horseshair has been made into a fabric known as "hair-cloth" or "hair-repp" which is especially suited for cushions in railway carriages, &c., owing to its great wear-resisting properties. But it has one great drawback, viz., being of an open nature, it admits dust, vermin, &c. The patentee therefore "proofs" such fabrics on the underside, by any of the well-known methods of proofing, hereby obtaining a fabric which he states possesses many advantages and is suitable for various purposes.—H. S.

*An Improved Method of Treating Vegetable Fibres so that they will Spin more Easily and make Stronger Yarn, Thread, or Strand.* K. T. Sutherland and G. Esdaile, Manchester. Eng. Pat. 18,932, November 26, 1889. 4d.

THE fibres are put into a vat or bath containing a weak solution of carbonate of soda (hot or cold), worked there for three hours, washed, dried, and prepared for spinning. Fibres treated thus will "flock," and when artificially shortened, can be spun on cotton machinery. The spinning will be easier, and the yarn, thread, or strand stronger. (Compare this Journal, 1890, 856.)—H. S.

*Improvements in or relating to the Decortication of Rhea, Jute, and other Fibrous Vegetable Stems.* J. Longmore and W. L. Watson, London. Eng. Pats. 7389 and 7390, May 12, 1890. 8d. each.

THE chief parts of a machine for the above-named purpose are two rollers, the one serrated, the other smooth, and a cylindrical beater. These parts must be in as close proximity to one another as possible. The rollers bring the material under the action of the beater, the serrated roller being placed above the smooth one. A fixed bar upon which the stems of the fibrous materials are broken is placed between the rollers and the beater, and made to rest on the smooth roller which supports it against the blows of the beater. Its vertical surface is curved, to enable the beater to work quite close to it. The bar further serves the purpose of scraping off the material from the smooth roller. The material is both carried to the machine and removed from it by two travelling aprons, one of which rubs off from the smooth roller any particles, especially gummy fragments, which the bar may have failed to scrape off. The beater and the serrated roller can be made of segments, instead of one cylindrical piece, and it is advisable, to make the ribs or blades of the beater removable so that they may be easily changed when worn or otherwise damaged. The construction is varied by doing away with the bar and using instead of the smooth roller another grooved or serrated one.

In this case the beater acts directly against this roller. The stems to be treated are laid longitudinally on one of the travelling aprons, nipped by the rollers, and by them delivered to the beater. The rollers should be adjusted in such a way that the stems are neither dragged nor crushed between the rollers and the beater. By the action of the beater the woody interior of the stems is broken into fine pieces which are easily removed by shaking the stems. The great advantages of this machine are that the whole length of the stems is uniformly operated on, and that simplicity and cheapness of construction are combined with continuity and reliability of operation.—H. S.

*A New Vegetable Fibre for use in the Manufacture of Brushes.* W. S. Booth, Gloucester. Eng. Pat. 9781, June 24, 1890. 4d.

The petioles of the leaves of *Sabal palmetto* (cabbage palmetto) are taken, steeped in an aqueous alkaline solution until soft, and then combed or hackled, thereby separating the useful fibres from bark, fine fibres and other waste substances.—H. S.

*Improvements in Apparatus for Carbonising Material such as Wool.* A. Silberberg and C. Detering, Bedburg, Germany. Eng. Pat. 10,856, July 12, 1890. 6d.

The object of this invention is to avoid a great disadvantage connected with the usual carbonising apparatus, viz., the escape of acid vapours. For this purpose the carbonising drum is mounted on a hollow shaft, and this shaft is connected with a cylindrically-shaped retort for the evaporation of the acid, the latter being introduced by a supplying tube fitting into the centre of the front surface of the retort. All these parts revolve together with the carbonising drum. The heating apparatus is so arranged beneath the retort that the fire gases pass uniformly over the whole surface of the retort. The products of combustion escape into a smoke-flue above the retort. The end of the supplying tube is fitted with another tube at right angles to it, having a funnel at its end, in order to raise from a vessel underneath it, at each revolution, a certain quantity of acid which flows into the retort. To keep the latter closed, the funnel tube is bent like a gauge-tube.—H. S.

*A New or Improved Process of Obtaining and Bleaching Vegetable Fibres.* K. Trobach, Berlin, and G. J. Bruck, Leipzig, Germany. Eng. Pat. 12,689, August 13, 1890. 4d.

The ripe, dry vegetable fibres, particularly flax fibres, are first boiled in a soap solution, and afterwards in hot water, and then washed with cold water. The material is next steeped in a solution of potassium permanganate, whereby all incrusting substances, such as pectin, gum, colouring matters, &c., are oxidised and destroyed. The material is again washed in cold water, and the reddish-brown colour produced by the permanganate solution removed by placing the material for a short time in a bath of sulphurous acid solution, afterwards washing in cold water. By this treatment the fibres are completely freed from all incrusting substances, and only the straw is left with the fibres, and is removed in the usual way. The fibres thus obtained are long, pliant, and brilliantly white, and give silky, glossy yarns which are of greater strength than those produced by other methods.—H. S.

## VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

### PATENTS.

*A Process for Rendering Dry Alizarin Fit for Colouring and Printing Purposes.* C. Romen, Charlottenburg, Germany. Eng. Pat. 18,217, November 14, 1889. 6d.

The method by which dry alizarin (instead of the 20 per cent. paste usually employed) may be used for dyeing purposes, consists in dissolving the dry powder in an alkali and adding borax. The following quantities are given for dyeing 100 kilos., for which the bath must contain 2,000 litres of water. A solution is prepared of 2 kilos. of the dry powdered alizarin in 15–20 litres of water, 1.3 litres of soda lye of 38° B., and 300 grms. of borax. This is then added to the water and the alizarin is precipitated by adding 1.3 litres of hydrochloric acid of sp. gr. 1.15 and 400 grms. of acetic acid. The goods are then dyed in the ordinary manner. For printing purposes the process is a similar one.—T. A. L.

*A New or Improved Machine for "Blowing" or Steaming, Boiling, Cooling, Scouring, and Dyeing Woven Fabrics.* A. T. Clay, Rastrick. Eng. Pat. 19,377, December 3, 1889. 8d.

The material to be treated is wound upon a perforated cylinder which revolves in an air- and steam-tight box, and the air pumped out of the box through the hollow shaft of the cylinder. Then steam is introduced into the box through perforated pipes, and the pumping continued, thus drawing the steam through the fabric. The perforated cylinder is kept revolving during the whole process. The time of treatment varies according to the nature of the fabrics. It is stated that liquids may also be applied to the fabric in the same manner.—H. S.

*Improved Apparatus for Dyeing, Cleansing, Bleaching, and Drying Yarn in Cops, or in a similar condition.* C. Weber-Jacquel, Thann, Germany. Eng. Pat. 19,408, December 3, 1889. 8d.

It is well known to be an advantage for the dyeing, scouring, &c. of textile materials, especially of yarn cops, to force the reagents through the materials. The essential features of this invention are that the reagents used are not forced through the cops by pumps, and that the vat is not hermetically closed. This prevents the spoiling of the machinery as well as of the reagents, and enables a continuous control to be maintained over the whole process. The cops are carried by a spindle frame, placed in an open vat, and connected with a vessel which stands on a higher level, and can be closed hermetically. The liquid descends into the vat by its own gravity, or under the influence of compressed air, and is sucked back by means of an exhauster. This is repeated as often as necessary for the dyeing, scouring, &c. of the cops. The spindle frame consists of a series of concentric tubes which communicate with each other by radial tubes, the latter being connected with that supplying the reagent. The spindles carrying the cops consist of hollow perforated tapering tubes of any suitable material. The apparatus can also be used for gaseous reagents, in which case the vat has to be closed.—H. S.

*Improvement in the Process of Treating Textile Fabrics and Manufacturing Bagging from Jute Butts.* A. Gratz and B. Gratz, St. Louis, U.S.A. Eng. Pat. 11,005, July 15, 1890. 4d.

Two or three parts of borax, or soda, alum, sodium chloride, &c., are dissolved in seven or eight parts of water, and applied to bagging manufactured from jute butts, and to other fabrics, to bleach them. The fabrics, still damp, are then formed into bundles or rolls, and stored away in this

damp condition, when a chemical action takes place in the mass which effects the bleaching of the fabrics. It is stated that bagging from jute butts along with other fabrics are by this method better bleached than by any previously employed process, and that the fabrics are rendered fire-proof, softer and more pliable.—H. S.

*Improvements in Colour Printing.* H. E. Newton, London. From G. W. Turner, Brooklyn, U.S.A. Eng. Pat. 12,636, August 12, 1890. 8d.

The object of this invention is to provide an improved method for printing in colours, by which an impression in two or more colours may be taken upon paper or textile fabrics by one operation, and is in particular applicable to book and newspaper work, where speed is of paramount importance. The invention is especially adapted to web machines, but is also applicable to other classes of printing machines, with either curved or flat printing surfaces. To effect the object desired a printing form or plate is prepared having surfaces of different heights, the picture or other matter which is to receive a colour different from the body of the paper being preferably placed below the surface of the main portion of the plate. With this form an impression surface is used having corresponding faces of different heights arranged reversely to those of the printing form. The paper to be printed is fed in the usual manner, the impression being given by bringing the impression surface into printing contact with both the upper and lower printing surfaces by one operation. The inking of the main surface is effected in the usual manner. The ink for the depressed portion is supplied by a secondary roller carrying an inking pad corresponding in size and shape to the depressed portion of the printing form, and being so geared that it engages with the depressed portion of the form as the cylinders revolve. The depression necessary to avoid contact of the lower surface with the primary inking mechanism is so slight as to cause no injurious effect upon the paper in printing.—W. E. K.

*Improvements in the Process of Colouring or Decorating Paper and other Fabrics for Wall Coverings and for other Purposes.* T. Strahan, Chelsea, Mass., U.S.A. Eng. Pat. 12,900, August 16, 1890. 8d.

This invention consists in producing devices upon paper, &c. by discharging liquid or liquid colour against the surface of the fabric by means of any suitable atomising arrangement. The colour may be directly applied or may be produced by the chemical action of the liquid upon paper coated with a suitable reagent. The jets of the nozzles of the atomisers may also play either continuously or intermittently upon the paper according to the effects desired, and two or more colours may be employed. The paper to be coloured is carried slowly along in front of the nozzles of the atomisers and at a suitable distance.—W. E. K.

*Improvements in Bleaching.* H. Thies, Laaken, and E. Herzig, Rentlingen, Germany. Eng. Pat. 12,950, August 18, 1890. 8d.

This invention relates to the bleaching of cotton and other vegetable fibres, and has for its purpose the economy of time, working power, steam and chemicals. The calico (or other vegetable fibre) is first impregnated with an acid solution, containing, per litre, 10 grms. of sulphuric acid, 16 grms. of hydrochloric acid, and 0.5 gm. of hydrofluoric acid. After lying for four hours it is steamed for half a minute and then passed into a washing machine containing a  $\frac{1}{2}$  per cent. solution of soda at a temperature of 50° C., and afterwards allowed to lie for 12 hours. The cloth is next washed in a washing machine with water containing chloride of magnesium or other alkaline earth compound, whereby the alkaline earth is deposited on the fibre by the action of the alkali contained in the cloth. The excess of wash-water is removed by squeezing-rollers, and the cloth brought into a specially-constructed scalding vessel or kier. Below the false bottom of the kier is a receptacle or chamber, and connected with this is a pump by means of

which the liquor may be drawn out of the receptacle and forced through the tubes of a boiler and then into the kier again at its top. Into this lower receptacle there also passes a perforated steam pipe, the steam passing into a second pipe which leads out into the air or to the condenser. The pressure in the lower receptacle is constantly varied by receiving or discharging steam, whereby the circulating liquid passes at a higher rate of speed through the fibrous material. With this accelerated circulation, moreover, the lye is concentrated by constant discharge of steam or water through the pipe into the air. When the kier has been filled with cloth and closed the contents are steamed for about  $1\frac{1}{2}$  hours at a temperature above 100° C. by means of the tube system contained in the tubular boiler. A charge of caustic soda is then introduced into the kier (to 1,000 kilos. of cotton 600 litres of solution prepared from 50 kilos. of soda ash, causticised with lime, and having dissolved in it 10 kilos. of resin), and this is circulated by means of the pump. By means of the tubular boiler the temperature of the circulating liquor is maintained at about 125° C., the operation being continued for about five hours. The cloth is then washed in the kier with boiling water, and finally "chemicked" and soured in the usual way. It is claimed that a cellulose of perfect chemical purity is thus obtained, which can be used not only for garments, but also for sanitary purposes and for the preparation of chemical products, as for instance gun-cotton.—W. E. K.

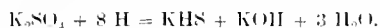
*Certain New and Useful Improvements in Dyeing Apparatus.* S. Pitt, Sutton. From A. Smith and Sons Carpet Company, Yonkers, U.S.A. Eng. Pat. 13,048, August 19, 1890. 11d.

This invention has for its object the reproduction by machinery of the operations of manual hand-dyeing. It consists of certain mechanism by means of which the hanks of yarn, hanging upon poles, may be shifted in the dye-liquor, and, if desired, trailed through the liquor; and when the entire invention is used the hanks of yarn hanging upon any one pole are not only shifted in the dye-liquor, but each pole of a series upon which hanks are hung in a dye-vat has the hanks upon it shifted in succession so that all the hanks of yarn which are being dyed at any one time in the same dye-vat are shifted in the dye-liquor the requisite number of times for the completion of the dyeing operation. For the mechanical devices by which this is effected the specification and accompanying 13 drawings must be consulted.—W. E. K.

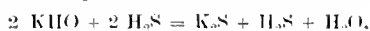
## VII.—ACIDS, ALKALIS, AND SALTS.

*The Reduction of Alkaline Sulphates by Hydrogen and Carbon.* Berthelot. Bull. Soc. Chim. [3], 4, 234—238.

It is usually stated in text-books that hydrogen reduces alkaline sulphates at a red heat, as, for example, in the following equation:  $\text{K}_2\text{SO}_4 + 8\text{H} = \text{K}_2\text{S} + 4\text{H}_2\text{O}$ . The author, however, finds that sulphuretted hydrogen always accompanies the aqueous vapour, and that the residue contains caustic alkali. In fact, the initial stage of the reaction is more correctly represented thus:—

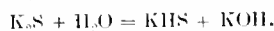


This reaction is exo-thermic. The reduction takes place at about 500° C. But at this temperature the potassium sulphhydrate produced is decomposed, under the influence of the external heat, into potassium sulphide and sulphuretted hydrogen:  $2\text{KHS} = \text{K}_2\text{S} + \text{H}_2\text{S}$ , 9.5 calories being absorbed per molecule of KHS. The formation of sulphuretted hydrogen is hastened by the current of aqueous vapour and excess of hydrogen. A portion only of this sulphuretted hydrogen passes on, the remainder reacting with the potassium hydrate forming sulphhydrate, which is, however, not stable at a high temperature.



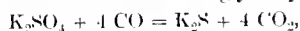


The heat liberated in this reaction is +12.8 calories. There is thus a tendency for all the alkaline metal to be converted into sulphide; but, on the other hand, the alkaline sulphide is decomposed by the aqueous vapour, an alkaline hydrate and sulphhydrate being regenerated with the liberation of +5.8 calories.



Between these various reactions a certain equilibrium is established, depending on the tendency of the bodies present to interact with evolution of heat, due to their internal energy; and on the decomposition of the sulphhydrate by the external heat. Meanwhile the production of water and sulphuretted hydrogen continues until all the alkaline sulphate is decomposed and all the sulphhydrate dissociated. The final result is a mixture of alkaline sulphide and hydrate, of which the relative proportions depend on the more or less prolonged contact of the aqueous vapour and sulphuretted hydrogen with the solid matter. A small quantity of alkaline polysulphide is also amongst the products of the reaction. Its formation is due to the decomposition of some of the sulphuretted hydrogen into sulphur and hydrogen. A part of this sulphur condenses in the upper portion of the tube; the remainder combines with the alkaline sulphide. As potassium sulphide is sensibly volatile at the temperature employed, a small quantity sublimes into the upper end of the tube, where it combines with the condensed sulphur, forming a reddish ring.

In studying the action of carbon on alkaline sulphates it is well to consider first the effect of carbon monoxide on these salts. The reaction is exceedingly simple,—



the heat liberated being +33.2 calories. In the case of sodium sulphate, +35.2 calories are disengaged. The temperature required to effect reduction is high, being about 700°–800° C. Purified dry charcoal acts on alkaline sulphates in a precisely similar manner and at the same temperature, carbon dioxide and an alkaline sulphide being formed. There is no doubt that carbon does not exert a direct reducing action on alkaline sulphates. Carbon monoxide is probably at first formed by the agency of the atmospheric oxygen present in the apparatus, or that contained in the charcoal. The carbon dioxide produced by the interaction of this carbon monoxide and the alkaline sulphate, reacts with the excess of carbon present, again forming carbon monoxide. The latter reduces a further portion of sulphate; and in this manner the action proceeds, the amount of gaseous carbon being increased during each cycle of reactions, so that a trace of carbon monoxide suffices to start the reduction.—H. T. P.

*Report on Acetic Acid.* Report of the Central Laboratory of the Italian Customs Department, 1886–1889, 307–313.

In order to protect the native industry of the manufacture of acetic acid from alcohol, the Italian Government raised the import duties recently (Decree, July 26th, 1888) on acetic and pyroligneous acids. The following duties are now in force: For liquid acetic acid, impure, 1 lira per quintal; for acid containing 10 per cent or less, 15 lire per quintal; from 10–50 per cent., 90 lire per quintal; above 60 per cent., 180 lire per quintal; and crystallised acetic acid, 200 lire per quintal (Journal, 8, 352). It is further laid down that “acetic acid is considered pure even though it is coloured artificially, though not containing substances of empyreumatic and bituminous odour characteristic of pyroligneous acid.”

The duty on crude pyroligneous acid is 2 lire per quintal, and it is defined as follows: “by crude pyroligneous and acetic acid are meant those which contain substances possessing empyreumatic and bituminous odours, arising from the distillation of wood, and of an acidity less than 50 per cent., calculated in terms of pure acetic acid.”

The Department points out that controversies frequently arise as to whether a product is to be classed as a *liquid* or *impure acid*, and that the high duties are an incentive to fraud, and that endeavours are and will be made to pass as pyroligneous acid concentrated acid of a considerable degree

of purity, such as are obtained after a second or third purification; these are often colourless, but are sometimes coloured artificially with caramel. As the regulations state distinctly that by an impure acid is meant crude pyroligneous acid, it obviously excludes acids obtained by the decomposition of purified acetates or by the method of Rothe, yet the laboratory have received samples of this nature. This point is insisted upon on account of a violent attack (Chem. Zeit. 13, 1473 and 1483) upon the Director of the laboratory for having classed as liquid acids, acids which were coloured and had a slight empyreumatic odour.

A table of analyses of samples received is appended to the report.—V. H. V.

#### *Report on Caustic Potash and Soda, Alkaline Carbonates.*

Report of the Central Laboratory of the Italian Customs Department, 1886–1889, 314–318.

The tariff distinguishes between pure and impure caustic soda and potash, the former paying 5 lire, the latter 0.50 lira per quintal; no such distinction is drawn in the case of sodium carbonates, whether natural or manufactured, but it is expressly laid down that “soda containing a quantity of sodium chloride greater than 60 per cent. is to be considered as sea-salt, and cannot, therefore, be introduced into the kingdom.” Determinations were made of the total alkalinities, the sodium hydrate, carbonate, chloride, and sulphate. Certain residues from the Leblanc process were classified as “concime” (refuse), in accordance with the report. Controversies arose regarding the classification of certain importations from France, as to whether they should be regarded as sodium carbonate, with a *differential* duty of 2 lire per quintal, or as impure caustic soda, subject to the *general* duty of 0.50 lira per quintal. As these contained a comparatively large proportion of carbonate (56–68 per cent.) and a small proportion of hydrate (3.5–10.5 per cent.), they were classed under the former heading.—V. H. V.

#### *Report on Potassium and Sodium Nitrates, Stassfurt Salts,*

yc. Report of the Central Laboratory of the Italian Customs Department, 1886–1889, 319–339.

ACCORDING to the tariff, every mixture of soluble salts containing a proportion of sodium chloride greater than 25 per cent. is to be considered as sea-salt, and its *importation is, therefore, prohibited*. Potassium chloride from Stassfurt is classed as manure; and from information supplied by the Syndicate out of 119 million kilogrammes of potassium chloride manufactured at Stassfurt in 1886, 4.2 million kilogrammes were sent to Italy. It is used in that country as manure, for the manufacture of nitre, and as a substitute for common salt in freezing mixtures. Carnallite, kainite, &c. are also classed as manures.

In cases of dispute concerning mixtures defined in the tariff as “salts for artificial sea baths,” the Direction determined the proportions of moisture, insoluble residue, sulphuric acid, total chlorine, magnesium, potassium, and sodium.

*Potassium and Sodium Nitrates.*—The former is subject to a duty of 3 lire per quintal, the latter is classed as “crude,” free of duty, and refined, with a duty of 3 lire. As a result of analyses by various chemists and of those performed in the laboratory, it was considered that refined sodium nitrate should contain 94–97 per cent. of the pure salt. Allusion is made to the frequent sophistication of sodium nitrate with chlorine. In both salts the proportions of water, insoluble residue, chloride, sulphate, and iodide were determined. It appears that several importations of salt and saline mixtures were confiscated, and the reports of analyses sent to the Minister.—V. H. V.

*Commercial Condition of Persia.* Board of Trade J. November 1890, 604.

See under X., page 1040.

## VIII.—GLASS, POTTERY, AND EARTHENWARE.

### PATENTS.

*An Improved Manufacture of Vitreous Material suitable for Artificial Marble, Stained Glass, and Analogous Objects.* F. R. Rostaing, L. A. Garehey, and N. E. Geille, Paris, France. Eng. Pat. 16,696, October 22, 1889. 6d.

THE waste glass of suitable size and of various colours and shapes is placed in moulds lined with silica or tale, &c., and the whole fired. A coherent mass is produced, which is dressed and cut, forming an irregularly-coloured block. The lower surface, *i.e.*, that which has rested on the mould, is rough from having been incompletely fused, and is left in this condition to enable the material to be more readily attached to the walls. Designs in relief, &c. can be obtained by pressure whilst the block is still plastic. If a suitable mould be prepared with movable partitions, then pieces of glass can be arranged in such a way that, upon firing, a stained glass window is produced, the necessity of using "leading," as in the ordinary process, being thus obviated.

—S. G. R.

*An Improvement in the Manufacture of Antique and other descriptions of Sheet Glass and Improved Apparatus in connection therewith.* J. G. Sowerby, Gateshead-on-Tyne. Eng. Pat. 17,565, November 5, 1889. 6d.

THE glass is blown into a jointed mould, furnished with a vertical slit. The cylinder thus produced is very thin at top, bottom and side at the opening of the slit, and can be easily cracked off at these points. It is then re-heated and flattened into sheets in the usual way. The cutting down, ordinarily effected with a diamond, is thus done away with. The specification is illustrated with drawings.

—S. G. R.

*Improvements in Etching upon Glass.* B. Z. Meth and H. Kreitner, Berlin, Germany. Eng. Pat. 12,155, August 2, 1890. 4d.

A SOLUTION is prepared by mixing together in proper proportions ammonium fluoride, sodium chloride and sodium carbonate, and then placing the mixture in a gutta-percha bottle containing fuming hydrofluoric acid and concentrated sulphuric acid. In a separate vessel made of lead, potassium fluoride is mixed with hydrochloric acid, and a little of this solution is added to the former, along with a small quantity of sodium silicate and ammonia. Some of the solution is dropped upon a rubber pad, and by means of a suitable rubber stamp bearing the design to be reproduced, is transferred to the glass vessel which is to be etched.

—S. G. R.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

*Magnesia in Portland Cement.* Dyckerhoff. Thonind. Zeit. 14, 452.

THE author has examined samples of cement containing proportions of magnesia ranging from 1.93 to 21.24 per cent. A normal Portland cement containing 1.93 per cent. of magnesia showed a regular increase in strength on keeping; the tensile strength of briquettes made of a mixture of 1 of cement to 3 of normal sand was 28.5—28.7

kilos per sq. cm. at 12—24 months. The remaining five samples when tested similarly all showed an increase in strength up to six months, the results being as follows:—

Per cent. MgO . . .	6.23	11.36	10.82	17.80	21.24
Tensile strength . .	26.5	25.5	22.5	22.5	15.0

The last three cements retrograded from this point more or less rapidly; the first two continued to increase in strength up to 12 months, and then began to go back. The speed of retrogression depends on the temperature at which the cement has been burnt. The sample containing 10.82 per cent. of magnesia, which was more lightly burnt than the others, diminished more rapidly in strength. Samples 1—3 showed cracks after three years, while the remaining two failed in this respect after two years. Pats made of sample 5 (21.24 per cent. of magnesia) unmixed with sand did not give signs of failure when subjected to the ordinary water-test until a year had elapsed. Besides these results with cements made of dolomitic material, the author found that strongly ignited magnesia added to a normal cement produced a powerful and prolonged expansion of the test pieces.—B. B.

*The Frost-Resisting Power of Natural and Artificial Building Stones.* Bauschinger. Thonind. Zeit. 14, 467.

EXPERIMENTS have been made with 21 different kinds of natural building stones, 3—6 test pieces of each being used. Their tensile strength, dry and wet, their capacity for absorbing water, their alteration in volume, tensile strength and behaviour towards water after freezing and thawing 25 times, and their specific gravity, were determined. Out of this number of samples, ranging from limestone to sandstone, only six were found to resist repeated freezing, *viz.*, one of dolomite, one of diorite, and four sandstones. Four other samples were found to resist freezing fairly, but not absolutely; but of 11 samples of artificial stone similarly tested, only three were found thoroughly unaffected, while eight proved fairly resistant.—B. B.

*Liability to Error in Mixing Cement with Wet and Dry Sand.* Candlot. Monit. Ceram. et Ver. 21, 144.

FERET has pointed out the possibility of serious mistakes in the proportions of mixtures of cement and sand due to a disregard of the condition of the sand—whether wet or dry. This is demonstrated by the following table:—

	Kilos.						
Quantity of water added to 100 kilos. of dry sand . . . . .	..	0.5	1	2	3	5	10
Weight of a cubic metre of moist sand	1,458	1,310	1,238	1,213	1,209	1,208	1,266
Weight of dry sand contained in 1 cubic metre of moist sand	1,458	1,304	1,226	1,189	1,174	1,151	1,119

It is obvious that the addition of comparatively little water largely increases the bulk of the sand,—out of all proportion to the amount used. For instance, 2 per cent. causes the weight of a cubic metre of the moistened sand to decrease 269 kilos., that is to say, 18.5 per cent. of the original weight. Such differences would cause great fluctuations, hitherto generally overlooked, in the composition of mortar mixtures intended to be of a certain strength.

—B. B.

*Corrosion of Zinc in Contact with Brick.* Iron, 36, 80.

UNDER some conditions, sheet zinc, when in direct contact with brickwork, appears to suffer to an appreciable extent from rapid corrosion. It has been found in the Berlin city

market halls that a portion of the zinc work supported upon brick walls was deeply pitted at a number of places, this being particularly the case where the metal was close to the bricks. Chemical examination of the bricks showed that they contained as much as 1.14 per cent. of soluble salts, capable of producing the destructive effect in question, and stimulated to more energetic action by moisture. The proportion of such salts, of course, varies with different kinds of bricks, while in some there may be nothing to induce any such corrosion.

*Report on Stones, Earths, Glasses, &c.* Report of the Central Laboratory of the Italian Customs Department, 1886—1889, 412—426.

POLISHED precious stones, such as rubies, emeralds, diamonds, pay 14 lire per hectogramme; but agates, opals, onyxes, pay 9 lire per kilogramme; while sham stones classified as glasses, crystals, smalt, pay 50 lire per quintal. Questions arose as to whether certain polishing materials made with powdered corundum were to be regarded as "polished precious stones" inasmuch as the report mentions corundum under this head; it was, however, decided that though corundum in the form of sapphires and rubies has an especial commercial value, yet, considering the common occurrence of this mineral, these materials should be classed as compounds of emery with bitumen, &c., and would thus be assessed as terra-cotta (Official Bulletin, 25, 883). It was also decided that infusorial earth came under the head of "fossil starch" and thus free of duty, and that powdered quartz, not specially mentioned in the report, should be classed as "natural sand."

Substances declared as non-metallic minerals such as native strontium and barium carbonates were considered to be rightly classified and not liable to the duties on chemical products not specified.

In order to decide whether materials declared as stones, earth, &c., were not in reality cements or hydraulic limes, and thus subject to a duty of 1.25 lire per quintal, the Direction determined (1) the specific gravity by the volumeter of Schumann, and (2) the amount of free alkali extracted from a given weight of the cement by a given volume of water according to the directions laid down by R. and W. Fresenius (Feichtinger, Technologie der Mörtelmaterialien, 185).

To judge of the hardening properties of mortar and cements, the criteria set forth by the College of Experts (Report, 1884, 139) were adopted.

#### PATENTS.

*Improvements in the Manufacture of Portland Cement from Hard Limestone.* W. T. Timewell, Bristol. Eng. Pat. 15,737, October 8, 1889. 4d.

HARD limestone such as the hard mountain limestone of Somersetshire and Devonshire is powdered sufficiently finely to allow of 75 per cent. passing through a sieve having 6,400 meshes to the square inch, mixed with "silicated clay" and water, ground, dried and burnt in the usual way. "Continuous running mixtures and the uncertainty of results incidental to the soft chalk processes" are said to be done away with, and "the production of a cement of high and regular quality" ensured.—B. B.

*Improvements in Resistant Coverings for Battery-Room Floors or Surfaces, Laboratory Floors, and like uses.* R. E. R. Crompton and B. H. Jenkinson, London. Eng. Pat. 17,623, November 5, 1889. 4d.

POWDERED slate is mixed with Trinidad asphaltum by the aid of heat, and the composition spread on the surface to be protected. Finely-powdered slate may afterwards be dusted on, and the floor finished by polishing. The process may also be used for the manufacture of vessels to hold acids and similar corrosive substances.—B. B.

*Improvements in the Manufacture of Building Material for Architectural and other Purposes, and of a Composition for use in Connexion therewith.* H. A. Bassett, Birmingham. Eng. Pat. 17,758, November 7, 1889. 6d.

TWO hundred parts of a viscous mixture of glue and water are mixed with 40 parts of oil such as rape or raw linseed and 12 of sodium carbonate, the whole being heated and agitated. In making such things as artificial stone 6½ parts of this mixture are taken and 16 parts of water and 9 of borax added thereto. One part of the resulting mixture is added to 6 parts of water, 1 part of white china clay, and enough plaster of Paris to give a stiff paste which can be moulded and polished.

For the preparation of plaster for walls, &c. 6½ parts are added to 16 of water, 96 of plaster of Paris, and 1 part of air-slaked lime, the mass dried and mixed with 9 parts of powdered borax; one part of the dried mass is mixed with 14½ parts of plaster of Paris, 40 parts of building sand, 1½ parts of wood dust and enough water to form a paste, which is applied as a first coat. The material for the second coat is made by using 10 of the dried composition, 112 of plaster of Paris, 140 of fine sharp sand, 10 of white china clay or powdered Bath stone, and enough water to form a fairly stiff paste. Similar compositions for other purposes may be used.—B. B.

*New or Improved Flexible Material applicable as a Substitute for Wood or Leather, and Process of Making the same.* W. P. Thompson, Liverpool. From C. Heepe, Boekenheim, Germany. Eng. Pat. 18,730, November 22, 1889. 8d.

See under XIV., page 1048.

*Improvements in the Treatment of Slag for a Variety of Important Uses, as Building Blocks, Paving Blocks, and the like.* S. G. T. C. Bryan, Birmingham, U.S.A. Eng. Pat. 8606, June 3, 1890. 6d.

TEX to 20 per cent. of anhydrous alumina is added to blast-furnace slag in the fused state, the mixture agitated and given a rotary motion by suitable mechanical means. It is claimed that the unfused impurities and the occluded gases are got rid of by this treatment, and that the produce is sufficiently homogeneous to be useful as a building material.

The moulds in which the treated slag is cast may be lined with wood, paper, or "carbon composition," the object of this device being to prevent "the contraction of the slag closing the cells near to and on the surface of the moulds," and to thereby increase the toughness of the product.—B. B.

*A Method of Colouring and Polishing Calcareous and other Stones, and producing Variegated Effects thereon in One or more Colours.* T. Breakell and J. W. Breakell, Manchester. Eng. Pat. 10,002, June 28, 1890. 4d.

THE process used may be understood by the following example:—To produce a red shade the stone is heated to about 200° F. and a mixture of "gum dragon and resin" applied to the surface, after which the stone is again heated for about two hours. The stone after cooling is cleaned and polished. The combination of resin with the colouring matter is claimed as novel. For brown shades boiled linseed oil in conjunction with tar and resin is also claimed. For variegated effects the stone is coloured as described above, then rubbed down until portions which have absorbed the colouring matter less deeply are exposed and treated with another colour in the same way as before, the parts already coloured absorbing but little of the new pigment. An imitation of marble may be produced by first painting or printing a pattern in a solution of "white gum lac" in naphtha, which serves to protect the stone from the colouring matter subsequently applied.—B. B.

## X.—METALLURGY.

*Titanium in Blast Furnaces.* A. J. Rossi. J. Amer. Chem. Soc. **12**, 91—117.

THE paper deals with the following questions:—(1.) Is titanium an element so obnoxious that ores, otherwise excellent, easily and cheaply obtained, should be rejected on account of its presence, even in small or limited quantities? (2.) Is it the excess of titanium which has caused the general prejudice there exists in America against the use of ores containing this element, and is this prejudice justifiable in all cases? (3.) Are there any limits within which the presence of titanium would pass unobserved, and would not influence the working of the furnace?

It is pointed out that titanium occurs to a varying extent in all the materials that enter the blast furnace, and a detailed summary of the percentage of the metal found in the ores used in the United States, Norway and Sweden, Russia, France, and England is given. A similar summary of the per cent. of titanium found in pig irons from many localities is also given. In the case of American pig this varies from 0.03 to 0.52 per cent. of  $TiO_2$ . A number of extracts from papers already published on the subject follows, and from an examination of the whole collated material the author draws his conclusions as follows:—

(1.) Titanic acid is not necessarily and always a cause of trouble in the blast furnace, even if present in large quantities.

(2.) In small quantities its presence appears to have been ignored for many years, and furnaces have run and do run to-day with ores rich in iron containing as much as 0.3 per cent. to about 1 per cent. of  $TiO_2$  in the charges without apparent trouble of any kind, these ores being smelted alone or in admixture with others more or less free from titanium.

(3.) When present in very notable quantities it has a tendency to render the slag pasty, and clog a furnace with titanium deposits; but with proper care and fluxes very little different from those ordinarily used, such ores may be smelted cleanly and profitably, though perhaps less economically than other ores.

(4.) Lean ores containing 40—48 per cent. of titanic acid have been successfully smelted, but at a relatively greater expenditure of combustibles. This refers especially to the smelting of Scandinavian ilmenite in charcoal furnaces, whilst it is pointed out that certain natural compounds of titanium, such as sphene, calcium silico-titanate, and kailhaute, which contain from 28—40 per cent. of  $TiO_2$  are perfectly fusible in a blast furnace. Reference is also made to an experiment of Professor Forbes, in which an ore containing 39.20 per cent. of  $TiO_2$  was smelted without admixture and without giving any trouble in the furnace. About 1—2 per cent. of the titanic acid remained in the pig, whilst the slag contained 36.18 per cent. of  $TiO_2$ , and approximated natural sphene in composition.

(5.) It cannot be maintained, in presence of the European and limited American practice with titaniferous ores, that inappreciable or very small quantities of titanium are objectionable or sure to cause trouble in the furnace; in fact the metallurgy of all kinds of titaniferous ores has been a success in Europe.

(6.) To decide what are the limits at which an ore containing titanic acid would require a modification in the ordinary charges used with ores free from it, could only be arrived at by direct experiment. Another practical question is to what extent the extra expenditure of combustibles, if any, would be compensated for by the special qualities claimed for the pig metal obtained? It is pointed out that the titaniferous iron ores are exceptionally poor in or free from phosphorus and sulphur (several analyses of American and other ores are given bearing out this point), and are therefore specially suited for the manufacture of Bessemer steel, so that the metal obtained from such ores has an increased value, apart from any particular beneficial effect due to the presence of titanium.

In conclusion, the author directs the attention of American metallurgists to their disregard of the value of titaniferous

ores, and states that hematite, &c. is often imported from great distances in cases where titaniferous ores are at hand more free from phosphorus and as rich and richer in iron than the imported ores.—C. A. K.

*Blast-Furnace Slags, and the Fusibility of Silicates.* A. J. Rossi. J. Amer. Chem. Soc. **12**, 189—214.

THE composition of blast-furnace slags affects their fusibility, and bears a close relation to the grade of iron obtained. Many circumstances may modify the results, and to say that a slag of a certain composition and fusibility will invariably correspond to a stated grade of iron is impossible.

Reviewing a number of tables, the results of experiments by Percy, Caron, and Berthier, and quoted in Vathaire, *Les Hauts Fourneaux*, the author points out that of the compounds which silica can form with lime and alumina, or alumina and magnesia, or magnesia and lime, as a general rule, the less basic they are the more fusible they become, until a certain limit of acidity is reached, and that amongst them, when the quantity of silica is above 40 per cent. and not over 60 per cent., the compound becomes as much more fusible as the percentage of silica is above the first figure, varying between 40 and 60, beyond which last limit the silicate appears to fuse again with more difficulty. The more complex a silicate becomes, the more fusible is it. If we consider that oxide of manganese, ferrous oxide, and alkalis are always met with in slags in proportions which may reach 5—7 per cent., that the alkalis, in very small quantities, increase considerably the fusibility of even the most refractory silicates, it can be better understood why injudicious charges of limestone do not necessarily produce injurious effects. A slag, for instance, which if based on a certain per cent. of alumina and lime would have been refractory, acquires a greater fusibility owing to the presence of manganous and ferrous oxides, alkalis and magnesia, derived from the limestone.

From a comparison of the equivalents of the various bases, the author points out the inconsistency of using lime in excess as a flux; it is the most expensive fluxing agent, and the one of which the largest quantity is needed. More of it is necessary than of magnesia; hence a *dolomite* is preferable to a *calcite*.

Analyses of slags are given, and the properties of the slags are discussed in explanation.

It is recommended, in order to be able to compare slags with one another directly, that all bases be calculated to their equivalent in lime. The type of a slag is thus at once known, together with its properties generally.—T. L. B.

*French Antimony Mines.* Moniteur des Produits Chimiques.

THE antimony mines of Montignat, in the Department of the Allier, which had been abandoned since the beginning of this century, are now again being worked successfully. The present exploiters are Messrs. Lassalle and Brulot, of Montluçon, and about 400 tons of the antimonial ore were extracted during the last 12 months. The Montignat ore was famous in the beginning of this century for its high quality. It is said to be quite free from arsenic, and to contain only traces of iron.

*Nickel Steel.* Engineering and Mining Journal, **50**, 355.

THE second stage of the Annapolis trial of armour plates resulted in still further establishing the superiority of the nickel-steel alloy for such defensive work. The tensile strength of the nickel-steel plate was such that though penetrated by the shots no cracks were developed in the plate. The all-steel plate was divided into quarters by four cracks running from the centre hole to the holes made near the four corners and thence to the outside of the plate. The Cammell compound plate may be said to have been entirely broken up. The nickel-steel alloy, of which the plate was composed, is said to contain less than four per cent. of nickel.

The question of a market for a largely increased production of nickel which was so much questioned last year may be said to be definitely settled by these trials, as in addition to the European demand a bill has been introduced and immediately passed, by the House of Representatives, at Washington, appropriating 1,000,000 dols. to enable the secretary of the navy to buy nickel ore and nickel matte for use in the production of steel armour for the new armoured cruisers.

*The Production of Platinum.* Board of Trade J.  
November 1890, 558.

It is announced from Mexico that M. Flavio Lobato, mining and chemical engineer, has discovered platinum in certain ores extracted from the mines of Las Yedras, in the State of Sinaloa. This news, if it is confirmed, will be of enormous importance to industry, more particularly as platinum is becoming scarcer, and realises an abnormal price.

The platinum mines, situated in the Oural mountains, contain, says the *Journal de la Chambre de Commerce de Constantinople*, important auriferous deposits; until recently gold in large quantities constituted the chief mineral working, and platinum was obtained at the same time without large supplementary expenditure. The richness in gold of the chief mines, Nisknee-Taglisk, and Gord-Blagodot has much diminished, and the preparation of platinum has become more difficult; such seems to be the explanation of the high price of platinum. A large number of miners have abandoned the mines, and have established themselves on the works of the Trans-Siberian railway.

*Discovery of Quicksilver in Austria.* Board of Trade J.  
November 1890, 573.

THE *Neue Freie Presse* reports the discovery of what promises to be a rich quicksilver mine at the little market town of Wippach in Carinthia. As long as 20 years ago children, while playing near the Wippach at low water, had been known to find the metal. Little attention seems however, to have been paid to the circumstance until recently, when the visit to the neighbourhood by a mining engineer afforded Chief Justice Dolowz an opportunity of drawing attention to the fact, which was subsequently verified by a number of well-known officials from Trieste, who, on visiting the spot, succeeded with little trouble in getting quicksilver. It is said that as the metal is to be found at a depth of only one metre, mining operations could be prosecuted on the surface. The discovery of quicksilver at so slight a depth is phenomenal from a geological point of view, the metal being usually found in old geological formations.

*Mines and Mining Interests in the State of Sonora.*  
Board of Trade J. November 1890, 582.

No copper mines are worked in Sonora, owing to the low price of that metal, and the owners of mines of that class have suspended all work on them. Veins of iron, antimony, and lead, which exist, remain undeveloped.

During the year cinnabar was discovered about 150 miles from Guaymas, in the district of Ureas, and ore has been found yielding 30 and 50 per cent. These deposits are being prospected, and have been bonded, it is stated, to an English company. The owners have, by a crude process, extracted 20 flasks of quicksilver from the ore. These deposits of cinnabar are the first that have been discovered or worked in this Consular district.

The plumbago mines, near the line of the Sonora railway, owned by an American company, are being worked. A small quantity of this mineral is shipped monthly to the United States.

*Commercial Condition of Persia.* Board of Trade J.  
November 1890, 604.

THE following particulars are taken from a report by the Dutch Consul-General at Teheran, and published by the Dutch Government (*Verzameling van Konsulaire en andere Verslagen*, 1890. *Teheran*):—

It is estimated that Western Europe exports to Persia every year goods having an average value of 4,000,000*l.* Persian exports are probably not so important, but in the absence of all statistics it is difficult to say. Persia is rich in natural products of all kinds. In elements of mineral wealth copper comes first; there are many copper mines in the north range of the Elburz near Teheran, as well as in the districts of Tabriz and Kasmin. Copper is the metal which is most used in the country, the inhabitants make almost all their utensils of it. The ore is very rich. Hitherto only a few mines have been worked; there are no rolling mills in the country, and the sheet copper has to be imported. The iron mines are at present of no value, although it is hoped that in connexion with a new concession from the Imperial Bank, some alteration will be made. Near Teheran there are some very rich iron mines, with an ore containing 70 per cent. little sulphur, and no phosphorus. Cobalt of very good quality is found at Kaschan, and arsenic at Kasuin, and at various places in Kurdistan; lead ore, containing manganese and silver, at Kerman and on the north side of the Elburz mountains; sulphur in various places. There is abundance of salt and saltpetre, as well as antimony, alum, borax (to the north-west of Kerman), potash, marble, coal, and asbestos, and on the road from Teheran to Tabriz quicksilver is found. Petroleum occurs in various places, and in particular at Schoester and Bender-Bashir. The Persian petroleum has a small specific weight. Coal is found in the north, in the south, and particularly to the west of Bender-Bashir. The quality of the Persian coal is not particularly good.

PATENTS.

*Improvements in Processes for treating Tinned or Galvanised Scrap Iron.* A. J. Campion and J. E. Temison-Woods, London. Eng. Pat. 14,462, September 13, 1889. 6*d.*

TINNED or galvanised scrap iron is pressed into cakes, and heated in a reverberatory furnace with nitrate or carbonate of sodium and oxide of manganese, with or without the addition of powdered felspar or granite. The iron is then withdrawn, and hammered or rolled. The metals in the slag can be recovered by any suitable means. The oxidation of the tin or zinc in the scrap can also be effected by blowing air through the heated metal. The strength and toughness of the iron can be increased by adding a small quantity of chrome ore to the charge. The nitrate of sodium and manganese oxide can be added by dipping the compressed metallic cakes into a hot saturated solution of the nitrate in which the manganese oxide is suspended.

—H. K. T.

*Improvements in the Extraction of Tin and other Products from the Refuse Slags and Débris of Tin-Smelting Furnaces.* A. J. Campion, London. Eng. Pat. 15,226, September 27, 1889. 6*d.*

TIN slags and refuse from tin-smelting furnaces are smelted with coal or coke and fluorspar, limestone, granite, or granite products such as felspar, quartz, &c., with the addition of rock salt or sodium sulphate for the purpose of forming a reducible silicate of tin. Slags from foreign ores which are rich in silica require the addition of alkali makers' "tank waste." The impure tin obtained is remelted, and nitrate of soda is added. Iron and arsenic are oxidised and form a slag, and tin, nickel, and cobalt are left. The two latter solidify, first forming "spieess," and the fluid tin remains on the top. The impurities in the metal can be also oxidised by blowing air through the mass. Impurities in "tin plate makers' waste" or "scruff" can be oxidised in the same way.—H. K. T.

*Improvements in the Treatment of Gold-bearing Quartz and other Minerals for facilitating their Reduction to Powder.* W. C. Taylor and T. N. Kirkham, London. Eng. Pat. 15,970, October 10, 1889. 6d.

THE ore is heated to redness in a reverberatory furnace, and is raked through openings in the side of the furnace into a tank of water. By this means the minerals are said to become so friable that they can be at once passed through crushing rollers.—H. K. T.

*A New Metallurgical Dry Process.* J. B. Alzagaray, Porto, Portugal. Eng. Pat. 16,776, October 24, 1889. 6d.

INSTEAD of converting minerals into fusible silicates to allow of their reduction by carbon, they are fused with carbon and alkaline salts, viz., common salt, sodium sulphate, or black ash, the temperature being carefully regulated so that only one metal is reduced at a time. A number of theoretical proportions of ingredients are given. The process is carried out in the Baxeres furnace (this Journal, 1890, 950).—H. K. T.

*A Method of Recovering Float Gold.* E. A. Cowper, London. Eng. Pat. 16,868, October 25, 1889. 6d.

WHEN crushed gold-bearing minerals are washed, small particles of gold float on the surface of the liquid and are lost. To cause the gold particles to sink, jets of steam are made to impinge on the surface of the liquid, the gold particles become wetted, and sink through the liquid.—H. K. T.

*Improvements in the Manufacture of Alloys of Iron and Aluminium and Alloys of Steel and Aluminium.* S. Pearson and J. H. Pratt, Birmingham. Eng. Pat. 18,048, November 12, 1889. 6d.

CALCINED IRONSTONE mixed with a considerable quantity of clay and the necessary amount of coke is charged into the blast furnace with fluorspar instead of lime. Cast iron containing aluminium is the result. Steel alloyed with aluminium can also be produced "in the blast furnace" by urging a more copious blast of air, whereby the carbon is oxidised and the percentage of it reduced to the necessary amount. It may also be obtained from the cast iron in a cupola or by other means.—A. W.

*Improvements in Rotating Furnaces for Roasting or Calcining Ores or other Substances.* S. Alley, Polmadie. Eng. Pat. 19,533, December 5, 1889. 11d.

THE rotating cylinder is made of iron segments bolted together with flanges, and in the longitudinal joints are fixed long narrow strips of iron projecting into the interior. These strips serve to hold in position the internal lining slabs of firebrick, which are preferably corrugated on the inside surface. The lower end of the inclined cylinder is connected with the fireplace and the receiving chamber, and the upper end is terminated by a smoke-box connected with the chimney, and a feeding apparatus. The ore to be calcined is moved along from a hopper by a rotating paddle into a feed pipe, which passes through the smoke-box a little distance into the cylinder, along which pipe it is conveyed by a revolving helix and thence into the furnace.—A. W.

*Improvements in the Manufacture of Ferro-Aluminium.* C. A. Faure, Paris, France. Eng. Pat. 19,588, December 5, 1889. 4d.

PERCHLORIDE of iron or other vapour rich in chlorine is passed over a mixture of bauxite and carbon heated to redness. The vapours and gases so formed are then led over scrap iron at a red heat, ferro-aluminium and chloride of iron (ferrous chloride) being formed. The latter is condensed and is subjected in a crucible, at a suitable temperature, to a current of air whereby oxide and perchloride of iron are produced. The perchloride of iron volatilises and can be used for a fresh operation.—H. K. T.

*Improvements in and Relating to a Process of and a Furnace for Treating Copper and its Alloys.* W. W. Keys, Bridgeport, U.S.A. Eng. Pat. 11,868, July 29, 1890. 8d.

THE patentee maintains that in order to obtain copper or its alloys of high specific gravity, great strength and minimum susceptibility to corrosion by hot acids, whether cast, rolled, or drawn, it is necessary to purify it and to eliminate the oxides, sub-oxides, and "other gaseous impurities as cause the metal when melted to flow sluggishly." &c. This is accomplished by bringing into contact with the molten metal the products of the incomplete combustion of hydrocarbon oils. Any suitable furnace can be employed, but the specification contains drawings of one in which the bed consists of a kind of shelf and a lower portion called the pot, the whole being heated by liquid or gaseous fuel. The copper is stacked on the shelf, melted and purified by the products of combustion as it trickles down into the pot, whence it is tapped.—A. W.

## XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

*Electrolytic Determination of Nitric Acid.* G. Vortmann. Ber. 23, 2796—2801.

See under XXIII., page 1066.

*Electrolytic Separations.* E. F. Smith and L. K. Frankel. Amer. Chem. J. 12, 428—435.

See under XXIII., page 1067.

## PATENTS.

*Improvements in Electric Batteries.* Sir W. Vavasour, Bart., London. Eng. Pat. 15,230, September 27, 1889. 1s. 8d.

IN this battery one of the cells is sealed hermetically, the electrode passing through the seal. Pressure is developed in the sealed cell during working and tends to force the electrolyte through the pores of the partition into the outer cell, whence it is allowed to flow away at a rate depending upon the output of the battery. The exciting solution is supplied also under pressure to the sealed cell at a suitable rate to make up for the lowering of level by percolation. Several different modes of applying this principle are fully described.—B. T.

*Improvements in Dynamo-Electric Machines for Electro-Plating and like Work.* W. B. Sayers, London. Eng. Pat. 15,700, October 7, 1889. 8d.

TO obtain a low E.M.F. for plating purposes from a dynamo running at a fair speed, the inventor cuts one or more slots in the pole pieces so as to weaken the magnetic field at these points, and puts on intermediate brushes so as to collect currents at one-half, one-third, or any desired fraction of the total dynamo E.M.F.—B. T.

*An Improved Apparatus for obtaining Metals of the Alkalis from Molten Chlorides.* L. Grabau, Hanover, Germany. Eng. Pat. 15,792, October 8, 1889. 8d.

THE difficulty hitherto has been to make the earthenware vessels used in this process last for a reasonable time, and it has been found that their destruction is not due to the heat employed but to the passage of the electric current through the cell; to obviate this the porcelain cell is made of a bell shape provided with a double wall, the top of which rises above the level of the molten chloride so as to

leave a clear gap between the chloride and the molten metal so that the current does not pass through the earthenware, but round underneath into the metal contained in the bell-shaped cell, the metal rising into this cell on account of its lower specific gravity. A delivery pipe leads away from the top of the cell to a receiver containing petroleum in an atmosphere of nitrogen or hydrogen.—B. T.

*Improvements in the Manufacture of Aluminium and other Metals.* D. Dixon, London. Eng. Pat. 16,794, October 21, 1889. 8d.

A MOLTEX halogen salt is decomposed electrolytically in the presence of a mixture of alumina and carbon, one of the elements of decomposition acting on the mixture producing an aluminium compound, which compound is then acted on by the other element of decomposition with the result of metallic aluminium. As an example magnesium chloride is decomposed into its elements, the chlorine reacts with the alumina and carbon at the anode, producing aluminium chloride and carbonic oxide, while the magnesium at the cathode decomposes the chloride so produced, forming magnesium chloride again and metallic aluminium. The process is continuous and can be conducted in one vessel with a porous diaphragm to keep the mixture surrounding the anode separate from the materials near the cathode.

—A. W.

*An Improved Construction of Plates for Secondary or other Voltaic Batteries.* H. T. Cheswright, Carcassonne, France. Eng. Pat. 19,080, November 27, 1889. 6d.

HOLES are punched through a lead plate in such a way as to force some of the metal out on the opposite side, forming a short tube with ragged edges; these holes are punched from each side so that both sides of the plate are roughened, and this roughness serves to retain the active material which is afterwards pasted on the plates. A flange is also cast round the edge of the plate to help to retain the active material.—B. T.

*Improvements in or connected with the Positive Elements (Negative Electrodes in charging) of Secondary Batteries, which Elements are Applicable also as Cathodes in Electrolysis.* The Mining and General Electric Lamp Company, Limited, and D. G. FitzGerald, London. Eng. Pat. 3731, March 8, 1890. 4d.

INSTEAD of using a leaden grid to support the spongy lead of the negative electrode, a grid of copper strip is made up, immersed in melted solder, and afterwards the apertures filled up with "lithanode material," such as a mixture of oxide and sulphate of lead. To effect the formation of the plate it is made the cathode in an electrolyte of magnesium sulphate, sodium sulphate, or potassium or sodium hydrate, or in a solution of dilute sulphuric acid.—B. T.

*Improvements in Primary Batteries and in Switching Apparatus connected therewith.* Sir C. S. Forbes, Bart., London. Eng. Pat. 4246, March 18, 1890. 8d.

FOR convenience of manipulation the cells of this two-fluid battery are placed in a frame mounted on trunnions, so that by tipping the frame the cells can all be emptied at one time. The positive plate is zinc immersed in a solution of calcium chloride or ammonium chloride or sodium chloride, while the carbon is in a solution of hydrochloric acid, sodium bichromate or chromic acid. A divided tank is placed over the cells and contains the two fluids for recharging; a switch is also fixed on each cell for enabling it to be either cut out of or put into circuit from a distance.—B. T.

*Improvements in Secondary Batteries.* M. B. Hollies and J. Warner, London. Eng. Pat. 8086, May 23, 1890. 6d.

A CONTAINING vessel of insulating material or of a conducting material lined with a non-conductor is divided into

liquid-tight compartments by plates of lead, and the compartments are filled with the electrolyte. The dividing plates are used as electrodes, one side of a plate serving as the positive plate and the other side as the negative.

—B. T.

*Improvements in Secondary Batteries.* T. M. Foote, Boston, Mass., U.S.A. Eng. Pat. 10,481, July 1, 1890. 8d.

A LEADEN tube preferably of rectangular section is filled with litharge or minium powder and bent or folded upon itself to form a plate of any desired size, adjacent sides of the tubes being burnt together, and the tubes themselves perforated to give the electrolyte access to the oxides of lead.—B. T.

*An Improved Connector for the Electrodes and Cells of Primary and Secondary Electrical Batteries.* A. J. Jarman, London. Eng. Pat. 10,515, July 7, 1890. 6d.

SCREW threads are cut upon the ends of a bar of suitable metal such as copper, and these ends are then bent round at right angles, and in the same plane, so that the ends are parallel and pointing in the same direction. The bar is then surrounded by a casting of lead leaving the ends projecting, these ends being united by a metallic connector held in position by suitable nuts working on the screwed ends, this connector serving as an additional stay to stiffen the arrangement, and also to lower the electrical resistance. The lead casting is then burnt on to the plates of the battery or attached in any suitable way.

—B. T.

*An Improved Apparatus for Producing Sheets of Metal by Electro-deposition.* M. G. Farmer, Eliot, Maine, U.S.A. Eng. Pat. 11,680, July 25, 1890. 6d.

A LONG cylinder of an alloy of lead and antimony is suspended upon a non-conducting roller and almost entirely immersed in a suitable electrolyte. The cylinder serves as the cathode, and is partially surrounded in the bath by a curved anode of the proper metal. A slightly zigzag line is painted with an insulating varnish on the cylinder parallel to its axis, and this serves to prevent a deposit of metal over the complete surface of the cylinder. Upon the passage of an electric current the cylinder is made to revolve slowly, and a metal sheet of any desired thickness is deposited upon the cylinder, broken only at the line of insulating varnish. This sheet can afterwards be stripped from the cylinder.—B. T.

## XII.—FATS, OILS, AND SOAP MANUFACTURE.

*Addition of Chlorine and of Halogen Acids to Oleic and Elaidic Acid.* S. Piotrowski. Ber. 23, 2531—2533.

OF the halogen addition products of oleic and elaidic acids only those have been previously described which are obtained by the action of bromine. Monochloro- and dichloro-stearic acid and monobromostearic acid are now described by the author.—T. L. B.

*Japan War.* O. Kleinstück. Chem. Zeit. 14, 1303—1304.

See under XXIII., page 1072.

*On the Determination of the Viscosity of Lubricating Oils.* A. Martens. Mitt. Königl. techn. Versuchs. 1890, 143—151.

ENGLER and Künkler, in describing their new apparatus for the determination of viscosity of lubricating oils (see Dingl. Polyt. J. 276, 42—47; this Journal, 1890, 651) make a



critical comparison between this new apparatus and the one used by the author. First, they state it as a disadvantage of the latter apparatus, that it takes a very long time to heat the oil in the air-bath, even if the oil is warm before. Secondly, in cases where the oil is heated outside the air-bath, and then brought into the apparatus, which has to be opened for the purpose, they assert that it is impossible to avoid changes of temperature. Thirdly, it is stated that many experiments show that the use of an undivided air-bath, as employed by the author, does not give uniform temperatures in the space surrounding the viscosimeter, unless the air-bath is of very large dimensions. In reply to these points the author says that to work with a uniform and high temperature throughout is neither required, nor is it a practical method, but that each oil should be tested at 8 to 10 different temperatures from 20° to 150° C., and the results obtained shown in a curve, presenting at a glance the properties of the tested oil. It is necessary only to keep the temperature equal during each single test, and the author has found that the use of an air-bath is the simplest and most convenient way for obtaining this result. The second objection is of no practical importance, but to pour the oil through an aperture instead of opening the apparatus is no doubt an improvement, and the author recommends that this plan should be adopted. It must be added that slight changes in the temperature of the oil do not affect the correctness of the test, if the temperatures are above 100° C., and a mean of the different temperatures observed be taken. The time occupied in pouring the oil into the apparatus is too short to alter its temperature, provided the surrounding air is not considerably colder. Against the third objection, the author gives tables and diagrams, which show that the temperatures in different parts of the air-bath at the same time differ only slightly, and that the curves which are obtained as described above, are quite satisfactory.

All possible errors are too small to be of practical importance, and the author is therefore of opinion that the apparatus, as used by himself, can be perfectly relied upon for all practical purposes, and has the great advantage of being simple in construction.—H. S.

*The Application of the Oleo-Refractometer to the Detection of Adulterations.* F. Jean. Bull. Soc. Chim. [3], 4, 105—108.

See under XXIII., page 1072.

*Lubricating Oils.* A. C. J. Charlier. The Engineer, 1890, 305—306.

AFTER a few preliminary remarks the writer explains that a good lubricating oil must have sufficient body to keep the surfaces between which it flows from coming into contact, hence the reason high gravity oils are now so much used on all classes of machinery; but at the same time everything depends on the class of work on which the oil is to be employed. If for light machinery, a very heavy oil would have a tendency to clog the bearings, thus impeding the speed; therefore for all ordinary machinery an oil ranging in gravity from '850 to '890 should be employed; whilst for heavy machinery, oils ranging from '900 to '980 should be chosen.

An important feature is to obtain oils having the greatest amount of fluidity consistent with body. Many oils are selected because they are apparently of high gravity, but such in reality is not always the case. The commonest paraffin burning oils can now be given an artificial gravity resembling castor oil, and yet immediately they become heated, even to a moderate temperature, they run off the bearings like water. Many of the Russian mineral oils now on the market have a gravity of about '920, are very highly refined and of a splendid colour, and yet when heat is applied they instantly become limpid, and are quite useless for the purpose for which they are required. But still it must be mentioned *en passant* that these Russian oils are very useful in making up compounds with oils not

affected by temperature, as they then supply body to an oil which, although an excellent lubricant, could not be used for heavy machinery on account of its low gravity.

Another point to be considered is to find an oil with a maximum capacity for receiving and distributing heat, together with a freedom from tendency to gum or oxidise; hence none of the drying oils should be used for lubricating purposes, and under this head users of lubricants must be warned against a process, which has lately been discovered, of adulterating lubricating oils with soap, by which is produced a very fine sparkling oil of gravity higher than castor oil, but at the same time utterly useless for lubrication. Users of lubricating oils often lay far too much stress on the colour of the oil, but as many methods are now known for giving oils, good or indifferent, any colour, either ruby, golden, or black, by the addition of the smallest percentage of the many aniline colours soluble in oils, the colour should no longer be taken as a test of quality; all that is required is that the oil should be bright and free from insoluble matter. It is essential that a lubricating oil should be neutral—that is, it should be neither acid nor alkaline. In the refining of many kinds of oil, mineral acids and alkaline earths are used, and, especially in the cheaper oils, sufficient care is seldom exercised in washing them free from all traces of acid; and then, again, it is well known that manufacturers often leave a large portion of alkali in the oils, as this greatly helps to make up their gravity.

The following are the more ready tests for the practical man:—With regard to the gravity, the oil is placed in a cylindrical glass vessel, and tested by the hydrometer. This method is very useful, because the gravity of the oil can be determined at any temperature. The glass vessel can be heated in a water-bath, and the gravity taken by placing the hydrometer in the oil at any moment. By this means it can be seen if the sample retains its original gravity when heated.

The "flashing point" of an oil is understood to mean the temperature at which the escaping vapour will momentarily ignite. The standard animal and vegetable lubricating oils, and all mineral oils of good body and high gravity, decompose or vaporise only at temperatures exceeding that of steam in ordinary engines, but nevertheless many of the lubricating oils now in the market are so worthless that it is very essential to know a ready means of ascertaining their flashing point. There are many good forms of testers, such as Abel's, Bernstein's, or Bailey's, but as they require very careful manipulation, the author suggests a simple apparatus, which can be worked very easily, and at the same time gives fairly accurate results. A small glass beaker is fitted into a larger one by means of a cork or india-rubber band; the former is to contain the oil, the latter serves as a water-bath. The smaller beaker is closed at the top by means of a cork, through which is inserted a thermometer dipping well into the oil, and a small piece of glass tubing tapering to a point, through which the vapour ascends. The oil should be on an exact level with the water in the outer vessel, so that the same pressure exists in both vessels; and when an oil to be tested is supposed to have a flashing point above that of boiling water, an oil-bath should be used in place of water. Everything being ready, the apparatus is stood on a sand-bath, under which is placed a spirit lamp, and gradually heated. A lighted taper is applied continually to the aperture through which the vapour from the oil is ascending, and immediately a small blue flame appears the temperature is noted, and the flashing point of the oil under examination thus determined. Any oil having a flashing point below 300° F. should be at once discarded as useless.

Another very important test is that for the viscosity or fluidity of an oil, and here a very simple and general test is to dip blotting paper in the oil and hold it up to drain; symmetrical drops indicate good fluidity, a spreading tendency viscosity. Again, an ordinary glass tube having a diameter of half an inch, tapering to a point at one end, and marked into divisions of 1 in., will answer all ordinary purposes for testing the viscosity of an oil. The oil to be tested is placed in this tube up to a given mark, and is then allowed to run out at the tapered end of the tube, and the time

taken is noted. This is compared with the time taken by standard oils previously ascertained in the same tube, and thus the viscosity of the oil can be determined. Sometimes it is necessary to take the viscosity at different temperatures, and in this case the glass tube can be fitted into an outer jacket containing water heated up to the required temperature. For all light machinery running at high speed, low gravity oils having a low fluidity should be looked for, whilst for heavy machinery of all kinds high gravity oils, with a moderate fluidity, should be employed.

An oil evaporating more than 5 per cent. in 12 hours, at a temperature of 140° F. is useless, as the evaporation creates a viscous residue, or leaves the bearings dry. To test for this, a weighed quantity of the oil should be placed in a porcelain dish and heated for the requisite time, then cooled and weighed and subtracted from the original quantity; the difference in weights constitutes the loss by evaporation.

The best lubricating oil is that which has the greatest adhesion to metallic surfaces, and the least cohesion in its own particles, and bearing this in mind there is no doubt but that mineral oils come first, sperm oil second, neat-foot oil third, and lard oil fourth; hence the finest mineral oils are best for light bearings and high velocities, and to give body to mineral oils fine sperm oil is used, or when greater tenacity is required, lard and neat-foot oils may replace sperm oil, while for the heaviest machinery mineral oils alone cannot be recommended, being deficient in body and of relatively low flashing point.

At the present time it is not so much a matter of quality which troubles the engineer; it is the question of cost. Lubricants answering in every way what is required of them can be obtained, but their cost is far too high for the ordinary consumer, and it therefore necessitates producers to search for compounds which will give to cheap oils the same properties as sperm, lard, &c., and be able to sell them at about half the price.

Mineral oils are the cheapest lubricants known, and yet they are often discarded for one reason, because they do not possess the same body as many of the vegetable and animal oils, and hence require a larger quantity to be used to do the same work. But though double the quantity of mineral oil is required to do the work done by other oils, yet as the price of the former is only one-quarter that of the latter, the mineral oils are still by far the most profitable to be used.

Oleate or stearate of alumina is now greatly used for giving a body to light gravity mineral oils. It is a very cheap compound, and can be added at a very small cost. The production of this "metal soap," as it is called, is well worthy of consideration, and can be well classed as a very successful result of research work recently done by several chemists in endeavouring to produce cheap lubricants. The high-class animal and vegetable oils in such high favour amongst engineers for their lubricating powers consist principally of olein and stearin, and these two substances, which give to these oils their high lubricating powers, can now be introduced into cheap mineral oils, thereby converting them into the finest lubricants at a cost far below that paid for castor, sperm and such like oils. The oleic and stearic acid is obtained in the usual way from any of the oils rich in olein and stearin, and is then precipitated with alum in the presence of caustic soda, thus forming oleate and stearate of alumina. A very small percentage of this metal soap dissolved with warming in light gravity mineral oils will increase their gravity some "50° or 60°," will greatly improve their lubricating properties, and after filtering through fine copper gauze, will give them a brilliancy not surpassed by any of the finest animal oils in the market, and at the same time not increase their cost of production more than 20s. per ton.

Some manufacturers use now a process of thickening their oils with ordinary soft soap, but this adulteration can be easily detected by adding glacial acetic acid and warming, when the soap will at once separate out. The writer recently examined a very fine-looking sample of lubricating oil, resembling in appearance castor oil, and found on estimation that as much as 60 per cent. of soap had been

introduced into the oil. Any oil found to contain such soap should be at once rejected.

Lardine and blown rape oil are now also largely used for giving body to lubricating oils. The former consists principally of cotton oil, which has been heated in the presence of a strong blast of air, somewhat similar to the process employed for thickening linseed oil, whilst the latter consists principally of colza oil treated in a similar manner. The presence of these "thickeners," as they are termed in the trade, cannot be said to visibly deteriorate the lubricating powers of the oil with which they are mixed, but they certainly do not improve it, and unfortunately they quickly become dull and cloudy, and in order to prevent this rosin oil must be used in conjunction with them. A small percentage of, say 1 per cent. of rosin oil, does not deteriorate the lubricating properties of an oil; but one containing above this percentage cannot be recommended for lubricating purposes. The presence of rosin oil can be detected in mineral oils by treating a small portion of the sample with an alcoholic solution of caustic soda, heating on a water-bath, evaporating nearly to dryness, adding a large quantity of distilled water, and boiling for some time, when a milky liquid will be formed; on the addition of more water and warming, the oil which separates, if free from rosin oil, should be clear, while a brown colour would denote the presence of rosin oil. As a confirmatory test, the aqueous liquid is drawn off from beneath and acidified with sulphuric acid; if no precipitate, the oil was all mineral, as a precipitate collecting, on warming, in brown viscous drops confirms the presence of rosin oil. But if instead of a milky liquid a semi-frosty mass is obtained after having added the distilled water to the evaporated solution, a fatty oil is evidently also present with the mineral and rosin oil; on adding more water the mineral oil will float to the top if present. The lower layer is then shaken up with amyl alcohol; when the alcohol separates as a brown layer, rosin oil is present. On further treatment of the aqueous solution with acid and warming, a separation of oleic acid, with its characteristic odour of fat, will show the presence of a fatty oil, either lard, neat-foot, or cotton oil.

For the lubrication of bearings of the largest size on which, either from the weight of the shaft or fly wheel, or from the drag of belts or ropes or from the formation of the bearing, very great pressure per square inch has to be resisted, castor oil up to the present appears to have no equal; and, in order to keep such bearings cool, it is very essential that this oil should not be adulterated. The adulterants to be looked for in castor oil are principally lard and colza oils, and they can be at once recognised by the density. The specific gravity of castor oil is .960; lard oil, .916; rape oil, .912; therefore, if the gravity is below .955, this alone indicates the presence of one of these oils. Again, another very characteristic test of castor oil is by the addition of zinc chloride. When pure it becomes yellow; when lard oil is present it becomes milky, and if colza be present a green colouration will be obtained.

Again, sperm oil is still very largely used by the leading cotton spinners, and the reason for this is that it leaves no stain and does not thicken by age or friction. Here again, on account of the high price of this oil, it is often adulterated, and generally with seal oil, cotton oil and mineral oils. These adulterants can also be easily detected with zinc chloride; if pure sperm oil a milky solution will be obtained, and if adulterated with any of the above-mentioned oils a brown colouration will be obtained.

Regarding the claims of tallow as a lubricant, there is no doubt but that, while in every case hydrocarbon oils are safer, and in the majority of cases are better and cheaper lubricants than tallow, even of the best quality, there is a number of cases in which engineers, although fully alive to the trouble and danger which are inseparably connected with the use of tallow, are constrained to use it, having failed to find any sufficient substitute. The great fault with tallow is that it corrodes metal, and often leaves a kind of deposit; and, again, it almost always contains free acids, as much as 25 per cent. of free fatty acid having been found in a sample of town tallow. Large proportions of free acid are apt to be due to the tallow being adulterated with wool-grease acids, or stearic acid from cotton-seed oil. Tallow,

again, frequently contains more or less water, infusible matters, and mineral impurities, and has been often purposely adulterated with starch, china clay, whiting, &c. For the detection of these mineral impurities, a small sample of the tallow should be dissolved in petroleum spirit, filtered, the residue washed with a little ether, and dried at a moderate temperature. On boiling the residue in water and adding iodine, a blue colouration will indicate the presence of starch; while effervescence of the residue on the addition of hydrochloric acid proves the presence of whiting.

**Report on Fired Oils.** Report of the Central Laboratory of the Italian Customs Department, 1886—1889, 65—83.

**FIXED** oils are divided in the tariff into two categories, viz., vegetable and animal oils. Of the former class fixed oils not specified are subject to a duty of 15 lire per quintal, exception being made of pure olive oil which pays 6 lire, cotton oil 15 lire, in addition to a surcharge of 14 lire, while palm and cocoa-nut oils, as also various kinds of oil which serve for medicinal preparations, are free of duty. Mixtures of olive oil with cotton-seed oil and other seeds are taxed as seed oils. Of the second class or animal oils, those of fish, when pure, are classified as medical substances not specified, if impure under No. 6, letter *b* of the general tariff; if not of fish, such as neatsfoot oil, they are assessed as fats of all sorts.

**Olive Oil.**—To ascertain the adulterations present in olive oil there are determined (i.) the specific gravity by means of the areometer of Pinchon which affords sufficiently accurate results according to the experiments of Del Torre (Conclusioni della Commissione Scientifica per gli olii, 1887) provided that a heavier oil has not been added to counterbalance the addition of a light oil; (ii.) the degree of hardness of the solidified oil by the apparatus designed by Serra-Carpi (*supra*); (iii.) the fusing point of the free fatty acids by the following method: the fatty acids, separated in the usual manner, are aspirated into a small bulb, which is bent round, then attached by wire to a thermometer, and the whole immersed in water. The temperatures both of fusion and of solidification are noted. A remark is made that the statements of Bach (*Chem. Zeit.* 7, 356) regarding these points are not sufficiently exact, and therefore they cannot be regarded as absolute criteria of the purity of the oil; (iv.) refractive index and dispersive power. A series of determinations have been made of these constants for various oils, values for some of which are given in the following table:—

	Refractive Index at 20°.	Dispersive Power referred to rays F and G.
Olive oil from various sources	1.4629 to 1.4637	*03914 to *04
Cotton oil.....	1.4667	*0387
Colza.....	1.4665	*0398
Sesame.....	1.4658	*0386
Earth-nut.....	1.4645	*03889

It is stated that these values approximate so closely to one another that they are not available for the detection of adulteration; (v.) the iodine number conducted according to the process of Hübl; it is considered that the value may vary between the extreme limits of 80—85, but more generally only from 80—83; (vi.) the saponification number or the quantity of potash expressed in milligrammes required to saponify 1 gm. of the oil (Köttstorfer, *Zeits. Anal. Chem.* 18, 199). The following values are given for various oils:—

Olive.....	191 —196
Poppy.....	192.8—194.6
Almond.....	187.9—195.4
Colza.....	177 —178.7
Sesame.....	190
Earth-nut.....	191.3—196.6
Cotton seed.....	191 —196.5

This determination is especially useful for mixtures of olive and colza oils; (vii.) Maumene's reaction on the rise of temperature produced by mixing 55 cc. of the oil with 10 cc. sulphuric acid of 66° B. Various samples of olive oil of undoubted purity produced a rise of 38° to 44.7°; for other oils Del Torre gives the following mean values:—

Cotton seed.....	69—70
Sesame.....	65
Flax.....	109
Earth-nut.....	58
Colza.....	59

In the opinion of the Direction, Maumene's reaction gives excellent results, provided that the operation is always conducted in the same manner, and very concentrated acid is used.

For the detection of various seed oils in mixtures, there are employed (1) the reaction of Hanchecorne, pointing out that the red colour produced in samples of rancid oil is easily distinguished from that produced by seed oils, a statement in opposition to the criticisms of Bizio (*R. Istituto Veneto*, 1885). (ii.) The reaction of Buillé, which is considered to afford a good proof of the addition of extraneous oils, provided that the minute precautions laid down by the author are exactly followed. (iii.) The reaction of Bechi for cotton-seed oil, which consists in adding to 10 cc. of the oil to be examined 1 cc. of the silver nitrate solution and 10 cc. of pure colza oil in amyl alcohol; the mixture is divided into two portions, of which one is warmed to 100° when a black colouration is produced if cotton-seed oil is present. The following prescriptions were laid down by the Scientific Commission: for the solutions, silver nitrate, 1 gm.; ethyl alcohol at 98 per cent., 200 cc.; ether, 40 cc.; and nitric acid, 0.1 gm.; and for the other, amyl alcohol, 100 cc.; pure colza oil, 15 cc. It is pointed out that excellent results are obtained, though in certain cases olive oil from Asia Minor and Corfu, made from fermented olives and kept for some time in stables, whereby ammonia was absorbed, gave the reaction of Bechi. Special tests are also quoted for the oils of colza, sesame, and earth-nut. The samples sent to the laboratory were found for the most part to agree with the stated declarations.

**Fish Oils** are classified in the tariff as impure fish oil, which includes the oils of porpoise, whale, and dog-fish, or as medicinal preparations, various rectified oils, and cod-liver oil. By a decree March 31st, 1890, the duties on impurities and adulterations of pure fish oil shall be determined by the Minister of Finance.

The work of Schädlér on the fish oils is alluded to (Benedikt *Analyse der Fette*, 236) and adopted by the Department. To recognise cod-liver oil a qualitative examination is made to detect the presence of the contained iodine; the method consists in saponifying the oil with caustic potash, evaporating the soap solution, and calcining the residue, in which the iodine is found in the usual manner. Samples declared as impure fish oil were recognised as pure fish oil, and in one case a sample of pure cod-liver oil was imported as a heavy mineral oil.—V. H. V.

## PATENTS.

**Improved Apparatus for and Method of Extracting Oleaginous and other Added Matters from Fibrous, Spun, Textile, and other analogous Substances.** E. Mansfield, London. Eng. Pat. 14,364, September 12, 1889. 8d.

See under V., page 1033.

**Improvements in Treating Crude Cotton-Seed Oil and Oils containing Resinous Matter and Free Fatty Acids to obtain Oil, Soap, and Resinous Colouring Matter.** R. Hunt, Liverpool. Eng. Pat. 17,870, November 9, 1889. 4d.

The oil to be treated is intimately mixed with water in the proportion of 2:1, by running both liquids in two streams into a rapidly revolving basin, where they are forced through a narrow slit and projected in thin films

against the surface of an outer vessel. The mixture, which now forms an emulsion, is allowed to stand for some hours, so that part of the water may be removed. To the supernatant emulsion half to  $1\frac{1}{2}$  per cent. of caustic soda and about 1 per cent. of common salt are added and the whole is thoroughly agitated and heated to  $110^{\circ}$  to  $160^{\circ}$  F. After settling, the liquor is treated with one-third to one-half its bulk of caustic soda solution of 4—5 Tw. containing one-half to 1 per cent. common salt at a temperature of at first  $110^{\circ}$ — $120^{\circ}$  F., then  $130^{\circ}$ — $160^{\circ}$  F. From the precipitate which settles out after both treatments with caustic soda the soap may be separated mechanically, the residue containing the resinous colouring matter.—K. E. M.

*Improvements in the Oxidation and Thickening of Oils for Use in the Manufacture of Paints, Varnishes, Linoleum, Floorcloth, or for other purposes to which Thickened or Oxidised Oils are applicable.* L. T. Thorne and "Brins Oxygen Company, Limited," London. Eng. Pat. 18,628, November 21, 1889. 8d.

Oils are oxidised for the purpose of bleaching or thickening in steam-jacketed vessels, by subjecting them to the action of pure or nearly pure oxygen in a finely divided state. Without the use of driers the process occupies two to seven hours; the presence of a very small quantity of driers shortens the time of oxidation. Consumption of oxygen per ton of oil varies from about 2,000 c. ft. to 4,000 c. ft. according to the degree of oxidation required. (See also Eng. Pat. 12,652 of 1886; this Journal, 1887, 443.)

—K. E. M.

*Improved Process for the Separation of an Extract called "Lanoresin," or Gum Resin, from the Waste Waters from Wool Washing and Combing Mills, and for the Production of the same in a Pure Condition.* C. Lahusen and C. Feiertlin, Delmenhorst, Germany. Eng. Pat. 18,642, November 21, 1889. 6d.

"LANORESIN" is a dark-coloured resin obtainable from the waste liquors of wool-washing mills. It is insoluble in benzene, ether, chloroform, carbon bisulphide, acetone, or strong alcohol, but is dissolved by water and dilute alcohol. To obtain it, the precipitate formed on the addition of a mineral acid to the waste liquor is soaped and mixed with 3—5 times its volume of benzene, heated and thoroughly agitated. During the agitation alcohol of about 95 per cent. is added to the amount of one-tenth the benzene; after standing some time the mixture separates into two layers, the lower one containing the "lanoresin." This is again treated in a similar manner to remove traces of grease, and finally the solvent distilled off.—F. H. L.

*An Improved Process of Filtering Oil.* C. Baker and W. E. Crane, Minneapolis, U.S.A. Eng. Pat. 4607, March 25, 1890. 8d.

THE improvement consists in passing the waste oil from bearings, &c., through a suitable vessel containing a porous filtering medium entirely submerged in a liquid of greater specific gravity than the oil. The oil by contact with the liquid becomes thin, so that in passing through the filtering material the substances held in suspension are more easily separated. The filtering material not being exposed to the atmosphere does not gum or clog up.—K. E. M.

*An Improved Filter for Oil and other Liquids.* C. A. Koelher, Neumühlen, Germany. Eng. Pat. 7229, May 9, 1890. 6d.

THE filter is so constructed that the liquid to be filtered passes through the filtering medium contained between perforated vertical plates which are movable so that the porosity of the filtering material may be increased or decreased by loosening or compressing it.—K. E. M.

### XIII.—PAINTS, PIGMENTS, VARNISHES, AND RESINS.

*The Origin of Gutta-Percha.* Sérullas. Compt. Rend. 111, 423—426.

THE discovery of gutta-percha was announced by W. Montgomerie in 1842, and the first samples brought to London in 1843 by José d'Almeida. Hancock and Wheatstone recognised its potential usefulness, but Walker was the first to apply it to submarine telegraphy in 1849. Substitutes have been sought for it without success, and attempts to utilize the exudations of other trees than those of the genus *Isonandra* have also failed, though adulteration by such means is not unknown. Reckless destruction of the original forests and failure to replace the trees when exhausted have decreased the available supply and make the prospects of future cables by no means satisfactory. The tree first recognised as a source of gutta-percha is the *Isonandra gutta* of Hooker; it has been regarded as extinct in Singapore, but is as a matter of fact only extremely rare. As recently as 1887, it flourished in the forest of Boukett Timah (Tin Hill) in the interior of Singapore, where it was originally discovered in 1847 by Lobb, who brought specimens to the collection at Kew. It is still found in other portions of the Malay peninsula. A detailed botanical description is given in the *London Journal of Botany*, 6, 463, under the head of *Isonandra gutta*. The author describes it when at maturity (30 years): the height of the cylindrical stem is 13—14 m. and its circumference about 0.9 m.; its leaves are 11—13 cm. long by 4—6 cm. broad in the case of the fully grown tree, but larger when it is young. The length of the petiole is 1.75—3.75 cm., the flowers 13—14 mm., and their peduncle 6.7 cm.; the fruit is about 3—4 cm. by 2.5—3.5 cm.; the seed about 1.8 × 1.2 cm.

There are at most five other trees which may be mistaken for the true *Isonandra gutta*, and the quality of their product at once differentiates them from it.

It is noteworthy that the word *gutta* (*guetate* or *guetutta*) simply means gum or bird-lime, so that the French term *gomme gutte* is a pleonasm. *Percha* (*pertcha* or *perfia*) is not, as has been supposed identical, with *perca* (i.e., the native name for Sumatra, meaning the world or habitable globe); but signifies *rag* or *fragment of stuff*, the term having reference to the appearance of the product after treatment with warm water.—B. B.

*Report on Compounds of Lead.* Report of the Central Laboratory of the Italian Customs Department, 1886—1889, 341—342.

AMONG substances sent to the laboratory were samples of lead acetate, specially mentioned in the tariff report, of a mixture of zinc oxide and sulphide with barium sulphate, declared as zinc oxide, but the laboratory decided that it should be classed as an adulterated white lead; and, lastly, of mixtures of barium sulphate and lead carbonate, which the tariff distinctly states are to be taxed as white lead.

—V. H. V.

*Report on Varnishes.* Report of the Central Laboratory of the Italian Customs Department, 1886—1889, 379—381.

VARNISHES are classified in the report under two different headings, viz., varnishes containing spirit which pay 30 lire per quintal, and in addition the surcharge on alcohol, and varnishes of every other kind which pay 20 lire per quintal. These varnishes are distinguished from oil of flax and siccativ resins, which are assessed as fixed oils not specified.

In cases of dispute the Department determines the alcohol and other volatile liquids, such as turpentine, benzene, carbon bisulphide, by processes of extraction and distillation, as also the nature of the residue. Among samples sent was one declared as a chemical product not specified, but recognised as a varnish containing turpentine, oil of flax, and lead oxide, while another declared as aluminium sulphate was a varnish of similar nature.—V. H. V.

*Cinnabar and Vermilion in China.* Board of Trade J. November 1890, 577.

THERE are cinnabar mines in the western and northern provinces of China. The pure metal is obtained in Ho-Nan and Kwei-Chow, and is brought to market in stone jars or enclosed in the joints of bamboo. It is also, says the United States Consul-General at Shanghai, manufactured from quicksilver by the reaction of sulphur and saltpetre on the metal in small copper furnaces, in which it is collected, after sublimation, in acicular crystals. Cinnabar is used in colouring red lacquered ware. It is also employed in painting, and forms an ingredient in many medicinal preparations. In former times it was highly prized for its virtues, and regarded as the true philosopher's stone and the immortal elixir.

Vermilion is made of fine cinnabar, which has been purified by sublimation, and is collected in acicular crystals of a bright violet red. It is powdered between two stones turned by hand, mixing a little water at the time. The sticky mass is then put into pure water, decanted, and finally dried on heated tiles or in the sun, when it is sifted for packing. The workmen are particular about the purity of the water used. It is used for making Chinese red ink, for painting on porcelain and wood, and colouring candles and paper. Its consumption is enormous. The most of the numerous temples throughout China are painted red, and everything lucky and pleasant among the Chinese is of vermilion colour. It is made by mixing together two parts of red sulphur and one part of mercury and sublimating the mixture. Fukein vermilion is said to be the best. Han-Kow supplies an inferior quality.

#### PATENTS.

*Improvements in the Treatment of India-Rubber for the Purpose of Dissolving it or rendering it Soluble.* S. Kenyon, Warrington. Eng. Pat. 14,150, September 7, 1889. 4d.

Two parts of old rubber, finely divided, are heated with one part of a "distillate or foots produced in the manufacture of varnish from gums or gum resins" in a still to a temperature of 120°–150° C. for about 12 hours. During the operation the sulphur volatilises, and the product becomes soluble in carbon bisulphide.—F. H. L.

*Improvements in the Treatment of Textiles by Means of Ammoniac Copper for the Purpose of making them Waterproof and Uninflamable.* C. Baswitz, Berlin, Germany. Eng. Pat. 16,708, October 22, 1889. 6d.

As is well known, cuprammonium affects and ultimately dissolves the cellulose of vegetable textile fibres. It is commercially prepared and applied by the patentee in the following way:—To a solution of copper sulphate, soda lye is added, but not to alkaline reaction, and the precipitate thus obtained collected on a filter and washed. It is then dissolved in ammonia solution of 0.91 specific gravity in the proportion of 1,000 cc. of ammonia solution to 150 grms. of copper sulphate. The textiles to be treated are by means of rollers passed through a vessel containing this solution, whereby their surface becomes parchment-like. Then the ammonia is removed from the textiles in a drying chamber, and the material afterwards passed through a hot solution of aluminium acetate, by which treatment basic aluminium acetate is formed, the waterproof property of which is well known. If a security against inflammability be desired, the aluminium acetate solution may be mixed with aluminium sulphate solution in the proportion of 10:1. At the same time basic ammonium aluminate is formed, which adheres fast to the fibres.—H. S.

*Improvements in Resistant Coverings for Battery-Room Floors or Surfaces, Laboratory Floors, and like uses.* R. E. B. Crompton and B. H. Jenkinson, London. Eng. Pat. 17,623, November 5, 1889. 4d.

See under IX., page 1038.

*Improved Means and Process for Treating Iron and other Metals, and the Production of a Fireproof Coating thereon preparatory to Ornamenting with Fusible Colours.* J. Meese, Leer, Prussia. Eng. Pat. 17,878, November 9, 1889. 4d.

FINELY powdered oxide of nickel is mixed with a small percentage of chromate of iron, lead glass flux is then added and the whole triturated with "stearic oil" and turpentine. The articles are coated with this mixture and "fired" in a muffle. A coating, valuable on account of its light shade and dull appearance, is thus obtained upon which fusible colours or metals in the form of powder can be applied and burnt in.—S. G. R.

*Improvements in the Manufacture of Floor Oil-Cloth and like Painted Fabrics, and Apparatus therefor.* F. E. Warburg, London. From C. van Nueffel, Antwerp, Belgium. Eng. Pat. 18,915, November 25, 1889. 8d.

This invention economises time, simplifies the machinery, and prevents loss in the manufacture of floor cloth, American cloth, table covers, and similar fabrics, by supplanting the different processes and manipulations of shearing, pumicing, coating, and drying by a combination of apparatus which allows a length of fabric sufficient to completely fill a drying chamber to be finished without handling. For detailed information the drawings accompanying the specification must be consulted.—K. E. M.

*An Improved Cement used for Covering Boilers, Steam Pipes, or Vessels containing Steam or Heated Water.* W. H. Lonsdale, Blackburn. Eng. Pat. 19,906, December 11, 1889. 4d.

See under I., page 1024.

*A Furniture Polish and Reviver of Leather and the like.* W. W. Box, Crayford. Eng. Pat. 20,438, December 19, 1889. 4d.

This polish consists of equal parts of linseed oil, vinegar, and methylated spirit, with a small quantity of butter of antimony. Colouring matter may be added if desired.

—F. H. L.

*Improvements in the Process of Manufacturing Thin Sheets of Nitro-Cellulose.* E. N. Todd, Newark, New Jersey, U.S.A. Eng. Pat. 9315, June 16, 1890. 8d.

See under XIX., page 1060.

*The Manufacture of New Material designed to serve as a Substitute for Bone or Celluloid.* W. M. C. Callender, London. Eng. Pat. 12,375, August 7, 1890. 4d.

THE new material termed "lactite," is produced by reducing casein to a gelatinous condition by means of borax, ammonia, &c., and then mixing it with a mineral acid and water. The following procedure gives good results:—10 kilos. of casein are placed in a vessel and 3 kilos. of borax dissolved in 6 kilos. of water are incorporated with it and heated. The water is then run off and to the gelatinous casein 1 kilo. of acetate of lead dissolved in 3 kilos. of acetic acid is added and the mass thoroughly worked together in a mechanical mixer. The acid water is then drawn off and the solid matter subjected to great pressure to drive out all possible moisture. The last traces of moisture should be driven out by the aid of heat, the resulting product being the new material. The new material may be supplied in the form of sheets, &c., and may be coloured by suitable pigments or dyes.—B. H.

## XIV.—TANNING, LEATHER, GLUE, AND SIZE.

### PATENTS.

*Processes for Preserving and Removing or Recovering the Colouring Matters from Tanning and other Vegetable Extracts.* B. Nicholson, South Norwood, and T. Palmer, London. Eng. Pat. 15,450, October 1, 1889. 6d.

For preserving the extract, the means described in Eng. Pat. 15,766 of 1887 (this Journal, 1888, 779) are used. The following method has been found effectual for the removal of the colouring matter from hemlock-bark extract. To the liquor, "at a density of about 40° Barmetre," is added about 1 per cent. by weight of powdered "Cayota" bark and 0.25 grms. mercuric iodide (dissolved in a suitable solvent) to each 1,000 grms. of the liquor. It is preferable to add also 2 per cent. of turpentine, either crude or specially oxidised. In place of "cayota" or in conjunction with it and turpentine, certain bodies, such as "sea-water, milk, egg albumen, blood serum, solution of borax, and the like, also herring brine, and some of the amides which possess an affinity for the camphoraceous and other substances produced during the oxidation of the turpentine" may be used. The liquor is run into oxidising vessels, and a current of air, warmed to a temperature of 100° F. is forced through it for a period of from 20 to 30 hours. Means may be employed to collect the volatilised products. The liquor is then diluted with water, warmed to 180° F., and filtered. The oil of turpentine may be recovered by mixing the liquor with "ether, light turpentine, light petroleum, benzoline, or the like," and separating the liquor from the ethereal solution.

The above process is not suited to the extracts from certain materials, such as chestnut. In the case of these barks the liquor is violently agitated in the cold with petroleum ether, run into a separator, and after standing, the liquor run off from the ethereal solution.—B. H.

*Process and Apparatus for Glazing Leather.* H. Bogenschield and F. Basch, Berlin, Germany. Eng. Pat. 16,595, October 21, 1889. 6d.

This invention is "to improve the glazing or glossing of leather by means of an apparatus in which the glazing cylinder not only moves to and fro, but also has a turning, pendulous or tilting motion given it, owing to which the cylinder presses the leather with greater energy, at the same time sliding, with the alteration in the direction of force, over the leather." Details are given in the specification.

—B. H.

*New or Improved Flexible Material applicable as a Substitute for Wood or Leather, and Process of making the same.* W. P. Thompson, Liverpool. From C. Heepe, Boekenheim, Germany. Eng. Pat. 18,730, November 22, 1889. 8d.

The material consists of wood plates or veneers and a tough and elastic foundation material or core.

The material is manufactured in the following manner:—The core or foundation (hemp, &c.) is laid on a veneer or sheet of wood in such a way that the single threads stand apart at least 6 to 8 mm. A thin layer of glue (containing glycerin or similar substance to prevent the glue becoming brittle) is then put on, and another veneer laid on. The whole is placed in a press, both pressure surfaces of which are coated with a sheet of india-rubber, or the like, and the pressing continued until the shape of the core or foundation is shown on the outer surfaces of the wood, whereby it is judged that the two surfaces of wood have come into complete contact. A characteristic of the invention is the employment of a wide meshed net between the veneers as a core, so that contact of the surfaces takes place between the meshes of the net. Instead of wood, pasteboard may be employed. A drawing is given.—B. H.

## XV.—AGRICULTURE, MANURES, Etc.

*Commercial Fertilisers.* Kentucky Agricultural Experiment Station Bulletin, 29, 1890.

This bulletin is explanatory of the constitution and uses of common commercial fertilisers, and is issued for the instruction of the Kentucky farmer; it therefore contains little that is not well known to agricultural chemists. The following may be noted as bearing upon points which are, perhaps, debatable or new.

Steamed or desiccated bones, if not very strongly steamed, are better for fertilisers than raw bones, and this, notwithstanding that the steaming removes some of the nitrogen. The fat of the raw bones adds weight, clogs the meal, and hinders the decomposition of the bone in the soil.

After standing, or when applied to the soil, the phosphoric acid of superphosphate has reverted to a form insoluble in water. This reverting of the phosphoric acid does not materially change its value as a fertiliser, for experiments (not here quoted) have shown that plants can take up the phosphoric acid in this state as readily as in the soluble form. The term "available phosphate" is sometimes employed to include both "soluble" and "reverted" phosphates.

An acre of wheat, yielding 20 bushels, requires about 28 lb. of potash ( $K_2O$ ?); an average crop of potatoes requires 100 lb. of potash per acre, and an acre of tobacco, yielding 3,800 lb. of leaves and stalks, assimilates over 200 lb. of potash.

Fertilisers are generally best sown broad-cast or drilled. When a small quantity of fertiliser is applied to each hill or row at planting time, it acts mainly as a stimulant to produce an early and vigorous start, which is considered necessary for the tobacco crop, but often renders the crop more sensitive to drought. In any case care should be taken to mix the fertiliser with the soil, so that it shall not come in contact with the seeds or plants. Most fertilisers, and especially those containing much nitrogen, soluble phosphoric acid, or potash, will injure or destroy young plants if brought in contact with them.

The following values per pound are ascribed at this station to the essential ingredients of fertilisers:—Phosphoric acid, soluble in water, 8½ cents; "reverted" phosphoric acid, 8½ cents; insoluble phosphoric acid, 3 cents; phosphoric acid in fine bone (capable of passing through a sieve with meshes one-twenty-fifth sq. in.), 4½ cents; phosphoric acid in medium bone (passing through meshes one-sixth sq. in., but not including fine bone) 4 cents; potash from potassium chloride, 5½ cents; potash from potassium sulphate, 7 cents; nitrogen, 20 cents.

Tables showing the analyses of various fertilisers now in the American market are appended to the bulletin.

—A. G. B.

*International Congress for Agriculture and Forestry, held in Vienna from September 2—6, 1890.* Chem. Zeit. 14, 1246—1247.

In the section of the Congress for agricultural investigations, the methods at present in use for the analysis and valuation of artificial manures received special attention. The following points were discussed:—

*Bone Meals.*—J. Stoklasa recommended the determination of the fat, the degree of fineness, and the character of the nitrogenous substance.

*Thomas Phosphate Powder.*—Stoklasa considers that the phosphoric acid rendered soluble by treatment with 20 per cent. citric acid should be considered the available phosphoric acid.

*Superphosphates.*—N. von Lorenz recommended the "citrate" method as used at Halle. The ammonium citrate solution is prepared by dissolving 1,500 grms. of citric acid in water, to which is added 5,000 cc. of 24 per cent. ammonia, the whole being then diluted to 15 litres. 50 cc. of a solution of the superphosphate (= 1 gm. of substance) are treated with 50 cc. of the ammonium



citrate; 25 cc. of magnesia mixture are immediately added, and the whole well shaken during half an hour. In the case of concentrated or "double" superphosphates, one-half the amount of the solution is taken (= 0.5 gm.) diluted with 50—75 cc. of water, heated on the sand-bath for one hour with 10 cc. of fuming nitric acid, and then neutralised with ammonia, again slightly acidified with nitric acid, and finally treated with 50 cc. of citrate of ammonia and 25 cc. of magnesia mixture as above described.

*Nitrogen in various manures.*—For the determination of nitric nitrogen, both Jodlbauer's modification of Kjeldahl's method and also Schloesing's were recommended by A. Devarda.

In the section for agricultural industries the question was discussed as to what extent saccharine has replaced sugar, more especially beetroot sugar, in commerce, with the result that the evidence indicated that saccharine was not displacing sugar in the markets, although it appears to be without any dangerous or even unpleasant consequences on the human being. During the last four years, 40,000 kilos. of saccharine have been manufactured.—J. W. L.

*The Composition of Vegetable Cell-Membranes.* E. Schulze. Ber. 23, 2579—2583.

See under XVI., page 1051.

## XVI.—SUGAR, STARCH, GUM, Etc.

*Invertase: A Contribution to the History of an Enzyme or Unorganised Ferment.* C. O'Sullivan and F. W. Tompson. J. Chem. Soc. 1890, 834—931.

BEFORE the publication of this paper, little was known concerning the nature and properties of unorganised ferments, and that little was of a very conflicting nature, the statements of various observers differing considerably. The objects which the authors had before them in the pursuit of their work were: to throw some light on the mode of action of these ferments, *i.e.*, to determine whether it was a life action, or, in general, whether it differed in any way from a simple chemical action; and to determine if they had a constant composition, and, if so, their chemical constitution. The authors chose invertase for the purpose of the investigation because its special property, the inversion of cane sugar in the cold and at moderate temperatures, is a very simple action and one which can be easily followed by polarimetric observations; also because they succeeded in discovering a method by which it may be prepared in quantity.

In order to devise a method for determining invertase, it is necessary to first obtain a knowledge of its properties.

Invertase is the only known body which has the property of inverting cane sugar solution in the cold and at moderate temperatures; and this property is the only known distinctive one of it.

It has been thought that this property of invertase was a life property, and that invertase might exist with different amounts of invertive power; if this is so, it is obvious that invertase could not be estimated by a determination of its invertive power. If, however, the invertive power of invertase is a fixed and definite property of invertase, just as the inverting power of dilute sulphuric acid is a fixed and definite property of sulphuric acid, it will be possible to estimate invertase by a determination of its invertive power. To determine this and to discover the laws affecting the action of invertase on cane sugar, the authors have made a large number of experiments under varying conditions. The experiments were in general conducted as follows:—A weighed quantity of cane sugar was dissolved in boiling water, and the solution made up to a definite bulk at 15.5° C., space being left for the invertase solution. The solution was placed in a covered vessel kept at a

constant temperature, and a weighed quantity of a preparation of invertase added and allowed to act on the cane sugar for a definite time. At the conclusion, the inversion was stopped by adding one drop of concentrated potash, and the optical activity observed after an interval of 15 minutes or so.

### THE ACTION OF INVERTASE ON CANE SUGAR.

*Rapidity of the Reaction.*—Three series of experiments were made, one at 15.5° C., one at 35.5° C., and one at 54° C. The results are plotted as a curve, the ordinates representing the percentage of sugar inverted and the abscissae the time units. One time unit is the time taken to invert 10 per cent. of the sugar. The three curves do not materially differ from the curve representing a simple chemical action in which no condition varies but the diminution of the changing substance; there is however a slight divergence.

*Influence of the Proportion of Invertase present.*—Unless the solution contain that amount of sulphuric acid necessary to produce the most rapid inversion (see influence of sulphuric acid), no intelligible results can be obtained; but if this condition is observed, it is found that the time necessary to reach any given percentage of inversion is inversely proportional to the amount of invertase solution employed, *i.e.*, to the amount of invertase. Experiments were made at 15.5° C. and 56.5° C. The results of these experiments were expressed by the number of minutes required to reach the point of no optical activity, that is 74.1 per cent. of cane sugar inverted. These numbers were calculated from the experimental results as follows. From the observed optical activity of the partially inverted solution, the percentage of inversion was calculated; by referring to the curve expressing the rapidity of the action, the number of time units are found which are equivalent to this percentage of inversion. The same curve shows that the point of no optical activity is reached in 12.7 time units. Knowing how many minutes it took to reach the observed optical activity and which have been found to be equivalent to so many time units, a simple proportion will give the number of minutes equivalent to 12.7 time units, that is the number of minutes required to reach the point of no optical activity. For the sake of convenience this is expressed by  $\pm 0 = n'$ .

*Influence of Concentration of the Solution.*—These experiments were made with yeast as the inverting agent at 54° C., and the results calculated in accordance with the previous experiments to the point of no optical activity and a constant amount of invertase. The most favourable concentration was found to be of solutions containing 20 grms. of cane sugar per 100 cc. of solution.

*Influence of Temperature.*—The results were calculated to the number of minutes required to reach the point of no optical activity with 0.1 gm. of invertase preparation, the calculations being made as above explained. The results were plotted as a curve, the temperature being represented on the ordinates and the number of minutes as above, on the abscissae. The curve agrees at low temperatures with that representing a simple chemical reaction taking place at varying temperatures. At high temperatures, the inverting power is much less than it should be if this simple relation held. The authors point out that these temperatures approach very near the point at which the invertase is destroyed, and that this is probably the explanation of the discrepancies.

*Influence of Alkalis.*—Small amounts of alkali completely destroy the inverting power. If a solution of cane sugar undergoing inversion, be "stopped" by a small quantity of alkali, the optical activity is found to decrease on standing, attaining a constant value in about 15 minutes. If a larger quantity of alkali is used, this constant value is reached instantaneously. It is, then, evident that this decrease when using a small quantity of alkali is not due to inversion continuing for a further short time. Additional proof of the truth of this has been derived from determinations of the molecular weight of solutions of cane sugar undergoing inversion, the molecular weight thus determined agrees



with that calculated from the constant value of the optical activity after "stopping" with potash at the moment at which the freezing point was determined, and does not agree with the value of the molecular weight as deduced from the optical activity possessed by the solution at the moment at which the freezing point was determined. This is perfectly analogous to the case of "bi-rotating" dextrose. The molecular weight of such bi-rotating solutions being the same as when the constant value of the optical activity has been reached, and it was found that small quantities of concentrated potash instantaneously destroyed the bi-rotation of dextrose solutions, there can therefore be no doubt that the dextrose is liberated in the bi-rotating state, when cane sugar is inverted in the cold by invertase.

**Influence of Sulphuric Acid on the Speed of Inversion.**—Small quantities of acid greatly increase the rapidity of inversion, larger quantities retard it. It is of great importance to determine the most favourable quantity of acid, and a great number of experiments were made for this purpose. It was found that this varies with the temperature and amount of invertase.

The following table expresses the most important results:—

Invertase Solution, Per cent. on the Sugar.	Most favourable amount of Sulphuric Acid, expressed as Parts of SO <sub>3</sub> per 100,000 of Solution.	
	Temp. 15.5° C.	Temp. 56° C.
0.4	..	1.25
0.7	..	1.25
1.5	7.5	1.5
4.5	15.0	..
15.0	25.0	..

**Influence of Alcohol on the Speed of Inversion.**—This varies in arithmetical progression with the amount present; it retards the reaction. This is in accordance with the influence of foreign bodies on simple chemical reactions.

**Amount of Inverting Power.**—This is practically unlimited. In one experiment, 1 part of invertase inverted 100,000 parts of cane sugar.

From the above experiments it is evident that the action of invertase on cane sugar is a definite and measurable quantity, and that in many ways it follows the course of a simple chemical reaction of which no condition varies but a diminution of the changing substance. The following method for estimating invertase is deduced from the above-described experiments.

Fifty grms. of cane sugar are dissolved in water to nearly 250 cc. and put in a water-bath kept at 15.5° C. When the temperature is constant, 0.5 gm. of the invertase containing preparation is added and the time noted. The solution is quickly made up to 250 cc. and divided fairly equally amongst five beakers which are placed in the water-bath. To the contents of each beaker different amounts of sulphuric acid are added. Under ordinary conditions the amounts may be 0.1, 0.3, 0.6, 1.0, and 1.4 cc. of decinormal sulphuric acid. After standing one hour at 15.5° C., a small quantity of the solution in the third beaker is taken and stopped by a drop of potash, the time noted, and the optical activity determined, and the time required for the solution to reach the point of no optical activity is calculated as explained above. When the calculated time approximately arrives, the inversion in each beaker is stopped by a few drops of potash, the time being accurately noted. After standing a quarter of an hour the optical activity of each solution is determined. If the lowest optical activity is possessed by the solution containing the least or greatest quantity of sulphuric acid, the experiments must be repeated with more suitable quantities of sulphuric acid; if one of the other three solutions has the lowest optical activity, this one is calculated as above explained to the number of minutes required to reach the point of no optical activity.

In some cases it is necessary to vary the conditions from those given above. In the case of yeast, good results can only be obtained at a higher temperature, 54° C. is most convenient; much smaller quantities of sulphuric acid are required; the amounts may be 0.0, 0.05, 0.1, 0.2, 0.3 cc. of decinormal acid for the five beakers. The results multiplied by 7.5 will bring them to the standard temperature of 15.5° C.

The results of a number of determinations made under varying conditions show that constant and reliable numbers are obtained.

#### THE PREPARATION OF INVERTASE.

If sound pressed brewers' yeast be kept in a lightly covered vessel in a warm place it liquefies, but even after several months does not putrefy. If after standing some weeks the liquid be filtered, almost the whole of the invertive power is found to be possessed by the filtrate; and from this alcohol precipitates a syrupy precipitate possessing high invertive power. This precipitate if dissolved in water and filtered yields a solution of invertase which, if a little alcohol be added, will often retain its invertive power for long periods. The residue on the filter is yeast albuminoid.

Full details for preparing the most active preparation are given in the original paper. The following analysis of liquefied yeast is given, which was obtained on allowing pressed yeast to stand for a month in a warm place:—

	Per Cent. on Dry Solid Matter of Yeast.	Grms. per 100 cc. of Solution.	Inverting Activity Calculated on the Yeast Liquor ± 0 =
Cellulose and other insoluble organic matter.....	22.80	..	..
Organic matter soluble in 17 per cent. alcohol .....	56.53	17.72	None.
Yeast albuminoid.....	5.93	1.86	None.
Organic matter insoluble in alcohol and soluble in water (invertase) .....	5.81	1.82	1380'
Ash.....	8.93	2.80	..
Total .....	100.00	24.2	1210'

The invertase above was the most active preparation obtained, and had an activity  $\pm 0 = 25.1'$  (calculated on the dry preparation). From the invertase solution the ash could be separated by dialysis in presence of dilute hydrochloric acid without impairing the activity of the invertase; it was difficult to remove the whole of the ash in this way as the solution began to decompose before the whole of the ash could be dialysed out. The invertase solution, as obtained above, could not be further purified, any attempt at fractional precipitation with alcohol destroying its invertive power. The most active preparations are very unstable; less active ones may be sometimes kept for years without material diminution of their inverting power.

**Yeast Albuminoid.**—As has been stated above, when the precipitate, obtained by adding alcohol to yeast liquor, is dissolved in water, invertase dissolves and a precipitate remains called yeast albuminoid; this resembles other albuminoids; it contains about 15 per cent. nitrogen, it is soluble in alkaline solutions, and is reprecipitated by acids.

#### THE INVERTAN SERIES.

Solutions of invertase having an inverting power expressed by the formula  $\pm 0 = 25' - 30'$  have an optical activity of  $[\alpha]_D^{20} = 70^\circ - 74^\circ$ , and contain 3.5—3.9 per cent. nitrogen. The substance in solution is called  $\beta$ -invertan. When any attempt is made to purify this by fractional precipitation with alcohol, the invertive power is destroyed

and the optical activity sinks to about  $[\alpha]_D = 54^\circ$ , and the fractional precipitates are partly soluble and partly insoluble in water; it is therefore evident that the invertase has been decomposed.

The insoluble body is called *α-invertan*; it is obtained as a crumbly precipitate; it contains 8.35 per cent. of nitrogen, and its optical activity is  $[\alpha]_D = -11^\circ$  to  $-20^\circ$ . If *α-invertan* be dissolved in potash and an alkaline solution of a copper salt and a large excess of potash added, a precipitate is thrown down, from which *ζ-invertan* may be prepared (q.v.); on acidifying the clear solution and adding alcohol a precipitate is obtained, which, when purified, is found to be identical with yeast albuminoid.

The soluble body is called *δ-invertan*, it is obtained as a syrupy precipitate; it contains 2.4 per cent. nitrogen, and has an optical activity of  $[\alpha]_D = 54^\circ$ . If it is fractioned several times by alcohol in the presence of acid it splits up into *α-invertan* and *ε-invertan*.

*ε-invertan* is also sometimes obtained directly in the fractionation of invertase, especially if much acid is present; it contains 2.07 per cent. nitrogen and has  $[\alpha]_D = 65.2^\circ$ . If it is often fractioned by alcohol and acid it breaks down into *ζ-invertan*, and an insoluble body resembling *α-invertan*, which yields *α-invertan* on purification.

*ζ-invertan* may also be obtained from *α-invertan* by forming the copper compound in the presence of a large excess of alkali, and the copper subsequently removed by hydrochloric acid. *ζ-invertan* is very stable, and can be fractioned by acid alcohol repeatedly without alteration. It contains 1.6 per cent. nitrogen, and has an optical activity  $[\alpha]_D = 75^\circ$ . It is broken down slowly when boiled

with dilute sulphuric acid. After boiling  $1\frac{1}{2}$  hours a body is obtained similar to the others of the invertan series, and called *η-invertan*; it contains 1.05 per cent. nitrogen and has an optical activity  $[\alpha]_D = 75.7^\circ$ .

All the invertan compounds yield with an alkaline solution of copper oxide a voluminous precipitate difficult to purify, and containing quantities of copper varying from 14–32 per cent. CuO. All the bodies thus obtained from invertase are evidently intimately related. The purest sample of *β-invertan* (invertase) had an inverting power expressed by the formula  $+0 = 25.1^\circ$ , and as this solution contained nothing but invertase, and the bodies obtained when invertase is decomposed as above, over 80 per cent. being invertase, the inverting activity of pure invertase would be expressed by the formula  $+0 = 22.5^\circ$ . Invertase is a white powder, which, when dry, will withstand the action of a temperature of  $100^\circ\text{C}$ . without much loss of inverting power. In solution its inverting power is diminished by very moderate temperatures and completely destroyed by exposure to a temperature of  $50^\circ\text{C}$ .; but in the presence of a large quantity of cane sugar it can withstand a temperature of  $60^\circ\text{C}$ . without loss of inverting power; this is, however, completely destroyed at  $75^\circ\text{C}$ . The composition of the invertan compounds agrees well with a theory that they are members of a series, each one being a compound of yeast albumen and a carbohydrate.

The following table shows their relation:—

A represents 1 part by weight of albuminoid and 8, 1 part by weight of carbohydrate, *α* represents *α-invertan*, that is  $\Lambda_1S_3$ , *η* represents three times *η-invertan*, that is  $3\Lambda_3S_3$ , so that *η* represents 57 parts by weight and *α*, 7 parts by weight:—

Name of Substance.	Inverting Power, $\pm 0 =$	Optical Activity of $[\alpha]_D =$	Constitution.	Composition Parts by Weight.	Composition According to Theory.			Composition Found.		
					C	H	N	C	H	N
<i>α-invertan</i> .....	$\infty$	$-15^\circ$ (Approx.)	<i>α</i>	$\Lambda_1S_3$	43.41	6.90	8.30	48.06	6.65	8.35
<i>β-invertan</i> .....	$22.5^\circ$ (Approx.)	$+80^\circ$ (Approx.)	$\eta\alpha_3$	$\Lambda_{23}S_{69}$	45.93	6.57	3.63	46.41	6.63	3.69
<i>γ-invertan</i> .....	$\infty$	$+45^\circ$ (Approx.)	$\eta\alpha_4$	$\Lambda_{19}S_{56}$	45.64	6.54	3.25	45.62	6.55	3.15
<i>δ-invertan</i> .....	$\infty$	$+54^\circ$	$\eta\alpha_3$	$\Lambda_{15}S_{45}$	45.30	6.51	2.79	46.50	6.82	2.43
<i>ε-invertan</i> .....	$\infty$	$+65^\circ$	$\eta\alpha_2$	$\Lambda_{11}S_{33}$	44.90	6.47	2.25	44.45	6.36	2.07
<i>ζ-invertan</i> .....	$\infty$	$+75^\circ$	$\eta\alpha$	$\Lambda_7S_{21}$	44.40	6.42	1.59	44.73	6.40	1.61
<i>η-invertan</i> .....	$\infty$	$+75.7^\circ$ (?)	$\eta$	$\Lambda_3S_{21}$	43.78	6.36	0.76	..	..	1.05

It has already been shown that in the inversion of cane sugar by invertase, the invertase is unaltered. The authors are inclined to consider that the invertase forms a loose compound with cane sugar and water and that this breaks down into invert sugar and invertase.—A. L. S.

actually give. If the other sugar was really galactose, treatment with nitric acid should produce mucic acid. As much as 57.3 per cent. of mucic acid was obtained.

Thus peach gum from either bark or fruit contains certain substances which on hydrolysis yield arabinose and galactose.

—T. L. B.

#### *The Carbohydrates of Peach Gum.* W. E. Stone. Ber. 23, 2574–2576.

WHEN the skin of unripe peaches or the bark of the tree is broken a peculiar gum oozes out, that from the bark being browner than that from the fruit. The presence of a carbohydrate giving galactose, as indicated previously by Bauer, was confirmed in both forms of gum by recognising the presence of furfural and of mucic acid. Evidence was obtained, however, of the presence of a second substance, which on hydrolysis yielded a pentaglucose. To isolate the carbohydrates the gum was heated for 9–10 hours on a water-bath with dilute sulphuric acid (sp. gr. 1.03), the yellow liquid was neutralised with barium carbonate, filtered, and evaporated to a syrup, which crystallised in the course of a few days. Fractional crystallisation was resorted to and the products were examined polarimetrically. In this way products were obtained which, from their specific rotatory power, pointed to the presence of arabinose and galactose. The supposed arabinose should, when distilled with dilute sulphuric acid, give furfuralamide, and this it did

#### *The Composition of Vegetable Cell-Membranes.* E. Schulze. Ber. 23, 2579–2583.

It is well known that many vegetable cell-membranes contain besides true cellulose other carbohydrates which are quickly converted by dilute mineral acids into mannose, galactose, or penta-glucoses. The question arises whether the portion of the cell wall unacted on by dilute acids, and which is generally looked on as true cellulose, furnishes only dextrose on hydrolysis. To decide this point the author has prepared cellulose from various seeds &c., including peas and lupin seeds, coffee beans, wheat bran, cocoa-nut kernels, and lupin shells, by treating these bodies successively with the following solvents: ether, dilute potash, and dilute acid. In some cases cold dilute nitric acid and potassium chlorate were also used. The remaining cellulose was in each instance converted into glucose by treatment with strong sulphuric acid. All the glucose syrups obtained furnished more or less saccharic acid on oxidation with dilute nitric acid, and the preparations from lupin seeds and shells, and wheat bran yielded crystals of dextrose, which was

identified by its optical activity and other properties. The cellulose from lupin shells, although apparently quite pure, was stained an intense violet-red colour when boiled with hydrochloric acid and phloroglucinol. The stained cellulose placed in a 50 per cent. solution of chloral imparted to the latter a similar violet colour. This behaviour could certainly not be due to the presence of lignin, since that body would be all extracted from the cellulose during the purifying process. The cellulose from lupin shells also yielded appreciable quantities of furfural on distillation with sulphuric acid. The glucose from coffee-bean cellulose was found to contain besides dextrose a considerable amount of mannose. Traces of mannose were also present in the preparation from cocoa-nut kernels. None of the glucose syrups contained either galactose or penta-glucoses.

—H. T. P.

*Fucose.* A. Günther and B. Tollens. Ber. 23, 2585—2587.

THE authors have continued the research—commenced by Bieler and Tollens—on fucose, which is prepared from the "fucus" (seaweed). They have obtained the sugar in the crystalline state, by first converting the raw sugar into the corresponding hydrazone, the latter, being, after purification, decomposed in the manner described by Fischer and Hirschberger. The sugar is very soluble, is sweet, and crystallises slowly, forming microscopic needles and plates, having the composition of rhamnose,  $C_6H_{12}O_5$ . Fucose is strongly levo-rotatory and exhibits the phenomenon of bi-rotation. The rotatory power of a freshly-made solution is  $-112^\circ$ , which gradually falls to  $-77^\circ$ . On distillation with hydrochloric acid, fucose yields methyl-furfural. Fucose strongly reduces Fehling's solution; 1 cc. of Fehling's solution = 6—7 mgrms. of fucose. With phloroglucinol, resorcinol, and orcinol, fucose gives merely a yellow solution. The hydrazone and osazone of fucose melt at  $170^\circ$  C. and  $159^\circ$  C. respectively.—H. T. P.

*On the Optical Isomers of Dextrose, Gluconic Acid and Saccharic Acid.* E. Fischer. Ber. 23, 2611—2624.

JUST as *d*-mannonic acid may be converted into *d*-gluconic acid, which may be reduced to *d*-glucose (dextrose) (Ber. 23, 800; this Journal, 1890, 528) so in the same way by heating *l*-mannonic acid (arabinose-carboxylic acid) with quinoline it is converted into *l*-gluconic acid which by reduction yields *l*-glucose. It is, however, difficult to prepare any quantity of *l*-gluconic acid in this way.

In the preparation of *l*-mannonic acid from arabinose, *l*-gluconic acid is also produced, and this is the most convenient source from which to prepare it.

The compound of arabinose and hydrocyanic acid is prepared and saponified by baryta; on removing the baryta by sulphuric acid a mixture of *l*-mannonic and *l*-gluconic acids is obtained; the lactone of *l*-mannonic acid may be almost entirely removed by concentration and crystallisation; *l*-gluconic acid does not crystallise and is soluble in alcohol. The syrup remaining after the separation of the *l*-mannonic acid is diluted and the hydrazide of *l*-gluconic acid precipitated by phenylhydrazine acetate; it is filtered out and purified by treatment with charcoal and crystallisation. The hydrazide is decomposed by boiling with baryta, the phenylhydrazine removed by ether, the baryta removed by sulphuric acid and the acid liquid neutralised with calcium carbonate, and calcium *l*-gluconate precipitated by alcohol and purified by recrystallisation. *l*-gluconic acid,  $C_6H_{12}O_7$ , may be obtained by decomposing the calcium salt by oxalic acid; by boiling with water it is partially converted into the lactone. Neither the acid or lactone crystallise. They are strongly levo-rotatory. The calcium salt is soluble in 3—4 parts of hot water and crystallises from the solution on cooling in cauliflower-like forms strongly resembling the *d*-gluconate. Its optical activity is  $[\alpha]_D^{20} = -6.6$ , being equal and opposite to that of the *d*-gluconate. The acid forms a phenylhydrazide  $C_6H_{11}O_6 \cdot N_2H_2 \cdot C_6H_5$ , melting point  $200^\circ$  C.

In the preparation of *l*-gluconic acid from arabinose as above, 50 grms. of arabinose yielded 20 grms. of pure

*l*-mannonic acid lactone and 8—9 grms. of pure calcium *l*-gluconate; in preparing it from *l*-mannonic acid by heating with quinoline not more than 6 per cent. calcium *l*-gluconate could be obtained; the small yields being due chiefly to the difficulties of purification.

By heating with quinoline, *l*-gluconic acid is partially converted into *l*-mannonic acid; the reaction thus being reversible.

*i*-Gluconic Acid,  $C_6H_{12}O_7$ , is formed by mixing equivalent proportions of *d*- and *l*-gluconic acid. Its properties are similar to those of its components. The calcium salt is very characteristic, being less soluble than the corresponding salts of its component acids. The acids and its salts are optically inactive. It forms a hydrazide,  $C_6H_{11}O_6 \cdot N_2H_2 \cdot C_6H_5$ , melting point  $188^\circ$ — $190^\circ$  C. Analogously to its components it may be formed by heating *i*-mannonic acid with quinoline.

*l*-Glucose,  $C_6H_{12}O_6$  is formed by reducing the lactone of *l*-gluconic acid by sodium amalgam in acid solution. The reduced solution is made alkaline, filtered, neutralised and evaporated until the sodium sulphate begins to crystallise; the syrup is then extracted with alcohol. The alcoholic solution on evaporation yields the sugar as a syrup, which crystallises on standing. It may be purified by recrystallisation from methyl alcohol. It resembles dextrose very closely except that it is not fermentable. Its action on polarised light is equal and opposite to that of dextrose.

*i*-Glucose,  $C_6H_{12}O_6$ , is formed by mixing equal quantities of *d*- and *l*-glucose or by the reduction of *i*-gluconic acid. It resembles its components closely. Yeast ferments it partly and leaves *l*-glucose.

*l*-Saccharic Acid,  $C_6H_{10}O_8$ , is formed just as ordinary saccharic acid by oxidising *l*-gluconic acid by nitric acid, and is isolated as its calcium salt. It forms an acid potassium salt,  $C_6H_9O_8K$ , crystallising in bundles of needles. The silver salt,  $C_6H_7O_8Ag_2$ , is insoluble. It forms with phenylhydrazine a double hydrazide melting point  $214^\circ$ . The acid and all its compounds are levo-rotatory.

*i*-Saccharic Acid,  $C_6H_{10}O_8$ , is formed by mixing solutions of equal quantities of *i*- and *d*-saccharic acid; or by oxidation of *i*-gluconic acid. It forms a double hydrazide melting point  $209$ — $210^\circ$  C. The acid and its compounds are optically inactive.

The acid potassium salt is characteristic of the three saccharic acids; the crystalline form of the inactive salt may be easily distinguished from that of the other two. All three are distinguished one from the other by their optical activities, the rotation for the two active salts being greatly increased by acidifying.

When an organic compound containing an asymmetric carbon atom is formed synthetically an inactive substance is always obtained which is either a combination of two active substances or inactive by internal compensation as inactive tartaric acid. In the case of the acids produced from arabinose by the hydrocyanic reaction the last case is not possible, as arabinose is not symmetrical, but according to Le Bel and Van't Hoff's theory three acids are possible, two with the carbon atom, marked with a star oppositely asymmetrical,  $CH_2(OH) \cdot CH(OH) \cdot CH(OH)CH(OH) \cdot CH(OH) \cdot COOH$ , and a third, a combination of these two. Two of these are known, i.e., *l*-mannonic and *l*-gluconic acids, but they do not appear to combine to form the third. The author has attempted to form a combination of *d*-gluconic and *d*-mannonic acid, but also with no success. It appears that these inactive compounds are only formed between two compounds such as *d*- and *l*-gluconic acid, in which all the carbon atoms are asymmetrically opposite.—A. L. S.

*On some Acids of the Sugar Group.* E. Fischer. Ber. 23, 2625—2628.

*The Lactone of d-Gluconic Acid* may be obtained from calcium gluconate by removing the calcium by oxalic acid and evaporating the acid solution on the water-bath as far as possible; the syrup is placed over sulphuric acid, and in 8—14 days fine needle-shaped crystals separated. The crystals are the lactone of *d*-gluconic acid,  $C_6H_{10}O_6$ , and may be purified by recrystallisation from water. The

crystals melt at  $130^{\circ}$ – $135^{\circ}$ , and are easily soluble in alcohol. It is dextro-rotatory and more so than *d*-mannonic acid lactone;  $[\alpha]_D = 68.2$ , solutions of *d*-gluconic acid are levo-rotatory, but they soon become dextro-rotatory owing to the formation of the lactone.

*Calcium L-Mannonate* is prepared by boiling calcium carbonate with an aqueous solution of *L*-mannonic acid lactone; the filtered solution is evaporated to a small volume and as much alcohol added as possible without precipitating the hot solution. On cooling a syrup falls out, which on standing crystallises.

*Arabonic Acid Phenylhydrazide*,  $C_5H_9O_5 \cdot N_2H_2 \cdot C_6H_5$ , is prepared by heating a solution of arabonic acid or its lactone with an equal weight of phenylhydrazine and 50 per cent. acetic acid for  $1\frac{1}{2}$  hour on a water-bath. On cooling the hydrazide crystallises out, the yield being nearly quantitative. Recrystallised it forms colourless, shining leaves, melting point,  $215^{\circ}$ .

*Xylose Carbonylic Acid* is prepared by the action of hydrocyanic acid on xylose and saponification of the product by baryta. The barium salt,  $C_5H_{11}O_7 \cdot BaOH$ , is difficultly soluble in cold water, and crystallises on cooling from its solution in hot water; decomposed by sulphuric acid it yields an acid, which, when reduced by sodium amalgam yields a sugar not as yet identified with any of the known hexoses.—A. L. S.

#### *Purification of Waste Water from Sugar Refineries, &c.* H. Schreib. Chem. Zeit. 14, 1323–1324.

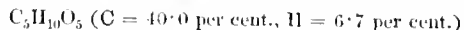
THE following method for the purification of the waste water of sugar refineries, &c. has been recommended by the Austrian Government inspector:—

The water, after being cleared by the addition of "chemicals," is filtered and pumped to an elevated reservoir, from whence it is allowed to flow over wire gauze for the purpose of thorough aeration, whereby the organic matters are presumed to become oxidised. That such oxidation takes place to only a very limited extent is proved by the results obtained by the application of several other methods having the same object in view. Storer's method, patented in 1880, appears to oxidise about 7 per cent. of the organic substance present; by Leeds' method, according to König, the water contained 17 per cent. of free oxygen and 53 per cent. of carbonic anhydride more than before treatment with air; the results of Knauer's method show that calcium carbonate becomes precipitated in the apparatus, but that the amount of organic substance is not materially reduced, and results obtained by the author with a method in which the water flowed over wire gauze showed that there was practically no change effected in the organic substance present.—J. W. L.

#### *Contributions to a Knowledge of the so-called Nitrogen Free Extract of Barley, particularly in Malt and Beer.* C. J. Lintner. Zeits. f. angew. Chem. 1890, 519–522.

ONE part of barley malt was digested with five parts of water for one day, filtered and washed with water. The filtrate was concentrated whereby some albuminoids were precipitated, filtered from these and alcohol added as long as a precipitate was formed. The precipitate was washed with ether, dried and dissolved in potash solution, the solution was filtered and a solution of copper sulphate added to the filtrate. A precipitate was thrown down, this was washed with water, until all the alkali was removed; it was then dissolved in concentrated hydrochloric acid and the gum precipitated by alcohol, redissolved and reprecipitated once or twice in the presence of hydrochloric acid until free from copper; it was then dehydrated with alcohol washed with ether and dried first in vacuo over sulphuric acid, and then in a current of dry air at  $110^{\circ}$  C. A similar product may be obtained from beer. It is a white powder, non-hygroscopic, and soluble in water to a cloudy solution, having a strongly acid reaction, not reducing Fehling's solution, and rotating the plane of

polarisation to the left  $[\alpha]_D = -27$  approx. After digestion with dilute acid the solution reduces Fehling's solution and becomes dextro-rotatory, but the nature of the sugar or sugars formed was not determined. A determination of the carbon and hydrogen of three different samples gave C = 42.5 to 39.8 per cent., H = 7.5–6.9 per cent. The author is inclined to consider it to have the formula—



It gives several of the reactions which are common to arabinose and xylose and bodies which yield these sugars on hydrolysis, i.e., it yields furfural on boiling with sulphuric acid, it gives with phloroglucinol and hydrochloric acid a cherry-red colouration.

A similar gum occurs in all grains, and a gum is also obtained by treating as above a solution of yeast in boiling water.—A. L. S.

#### *The Transformation of Glucose into Sorbite.* J. Meunier, Compt. Rend. 111, 49–51.

THE author has investigated the action of sodium amalgam on glucose, and describes a method for the preparation of sorbite from glucose by the action of sodium amalgam. A yield of 35–40 per cent. of sorbite has been obtained.

—H. T. P.

#### *Note on the Hydrogenisation of Sorbin and the Oxidation of Sorbite.* C. Vincent and Delachanal. Compt. Rend. 111, 51–53.

THE authors have converted sorbin by reduction into sorbite from which it differs by two atoms of hydrogen; but have not been able to reverse the process, viz., convert sorbite by oxidation into sorbin.—H. T. P.

#### *Automatic Apparatus for Sugar Refineries.* P. Horsin-Déon. Bull. Soc. Chim. [3], 4, 108–112.

*Automatic Measurer.*—This apparatus registers the amount of juice in the measuring back at any time, the time at which it was full, and the time at which it was emptied. The apparatus consists of a float in the measuring back which communicates by a light cord with the apparatus proper. The end of the cord is attached to a reel round which it winds. This reel is furnished with a pin which communicates a vertical motion to a copper rod, which carries a pen or pencil writing on a paper stretched round a revolving drum. The copper rod makes an electrical contact at its highest and lowest positions, which causes a bell to ring. The positions of these contacts are so arranged that they correspond to the empty and full state of the measuring back. There is also a counter which moves forward one unit every time the lowest contact is made, that is, every time the measuring back is emptied, thus registering the number of times the back is emptied.

*Registrating Densimeter.*—This consists of two chambers connected by a tube; the outside end of each chamber is closed by a sheet of caoutchouc. The whole is filled with water and placed vertically in a vessel in which the juice passes in at the bottom and out at the top, so that the height of the liquid is constant. It is evident that if the density of the liquid in the outer vessel increases there, the pressure on the water in the chamber and tube will increase, and the top sheet of caoutchouc will bulge out. This sheet is connected with a system of levers, which carry a pencil writing on a revolving drum, so that the position of the caoutchouc sheet is registered, that is, the density of the liquid passing through the outer vessel is registered. As the water in the chambers and tube is at the same temperature as the liquid passing through the outer vessel, no correction for temperature is necessary.

*Automatic Sampler.*—As the strength of the juice may not be uniform throughout the vessel from which the sample is taken, some arrangement is necessary that the sample taken may be of the average strength of the whole body of liquid. The sampler consists of a tube open at both ends and as long as the back is high. This is placed vertically in the back and fills as the back fills. The contents

of the tube will evidently be a true sample of the contents of the back. The lowest end of the tube is furnished with a three-way cock moved by a float. Whilst the back is filling the cock is placed so that the tube is in connexion with the liquid in the back. When the back is full the float alters the tap so that the contents of the tube flow out through a side tube into a vessel placed to receive them.

—A. L. S.

#### PATENTS.

*Improvements in or relating to Apparatus for Drying Sugar-Cane Trash.* A. J. Boulton, London. From L. Bon, Guantamano, Cuba. Eng. Pat. 4754, March 26, 1890. 6d.

This invention relates to apparatus whereby green sugar-cane trash may be rapidly dried and made available for use as fuel. The apparatus can only be intelligibly described by reference to the drawings accompanying the specification.

—A. J. K.

*Improvements in Sugar Refining.* H. Lake, London. From L. Stenberg, New York, U.S.A. Eng. Pat. 12,635, August 12, 1890. 6d.

A solution of cane sugar of  $10^{\circ}$  to  $15^{\circ}$  B. which contains glucose is boiled for about two hours with caustic lime, strontia, or baryta (preferably lime) in the proportion of 15 to 30 per cent. of lime on the weight of dry sugar plus the weight of glucose present. By this means the glucose is decomposed, and the products of decomposition form with the alkaline earths compounds which are insoluble so long as the solution remains alkaline. In addition to these insoluble compounds, which can be removed immediately by means of a filter-press, there are also formed soluble bodies which may be precipitated by treatment in a "blow-up" with carbonic acid, and finally certain liquid bodies which cannot be thus precipitated, but which are not injurious, because they do not interfere with the crystallisation of the cane sugar. This process is recommended as a useful preliminary to those processes in which low grade syrups are purified by means of lime or strontia and carbonic acid, such as the so-called lime process described in U.S. Pat. 270,879 of 1881.—A. J. K.

## XVII.—BREWING, WINES, SPIRITS, Etc.

*On the Influence of Various Fruit Ferments (Enzymes) on the Bouquet of Fermented Drinks; and on the Production of a Cider from Barley.* G. Jacquemin. Bull. Soc. Chim. [3] 4, 3—6.

The author refers to a previous paper (Compt. Rend. 106, 643; see also this Journal, 1888, 397), in which, starting from Pasteur's theory that the bouquet of an alcoholic liquor depends on the special enzyme used in its production, he described certain experiments on the improvement of barley wines, and was the first to put this theory to the test on a commercial scale. Later on, in 1888, he showed that barley wines made with the enzymes peculiar to the grapes of Beaune, Chablis, and Riquewyhr (Alsace) possessed the bouquet of the wines of these places. M. Quénot of Nancy has, with the author's help, produced in a similar way, on a commercial scale, raisin wines which cannot be distinguished by the taste from the white wines of Alsace and Chablis.

Since then, L. Marx (a pupil of Hansen's) has published an important memoir in the Monit. Scient. (November 1888), and Rommier has published a note (Compt. Rend. 108, 1322) on the same subject.

In the autumn of 1889 the author used the enzymes of grapes from Ay, in Champagne, from Beaune, Chablis, and Barsac in the making of barley wine. Each experiment was carried out on 60 hectolitres of must in one of the German *Gerstenwein* breweries, and the results showed how largely the special ferment influences the bouquet of the wine. In order to preserve the purified culture of the enzyme, with all its properties, Pasteur has shown that it

must be made dormant by cultivation in a 10 per cent. solution of sugar, which is renewed until fermentation ceases. The dormant ferment kept under the sugar solution becomes active as soon as it is placed in a must containing the necessary elements for its nutrition, such as a grape or barley must. The author has observed that in the period preceding the dormant state, so long as the enzyme acts at all on the sugar solution, the characteristic bouquet is still developed. The decanted sugar solution contains very little alcohol, but nevertheless has a delicious taste, the bouquet being more marked than in the wine itself.

Two practical applications of this discovery are possible:—(1.) Special liqueurs may be manufactured by adding alcohol to the saccharine solutions containing the bouquet of some first-class wine. (2.) These saccharine bouquets may be added by wine-growers to the must for new wines in conjunction with the special enzymes.

The author intends to attempt to isolate the bouquets this autumn by dissolving in ether and carbon disulphide.

*Barley Cider.*—In January 1890, the author isolated an apple enzyme from the lees of a Picardy cider. The cells were pyriform in shape, but on cultivation in a barley must containing tartrate, they become ellipsoidal, like most of the saccharomyces.

This apple enzyme, after purification, was finally employed to ferment 50 litres of barley-must, to which 3 per cent. of citric acid had been added, in order to prevent the competition of undesirable enzymes. The results of the experiment are favourable, and the fermented product possesses the peculiar taste of cider.

Barley-cider contains from 50 to 60 and even 70 grms. of solid extract per litre, and is, therefore, richer than ordinary cider. It contains from 5.5 to 6.5 per cent. of alcohol.

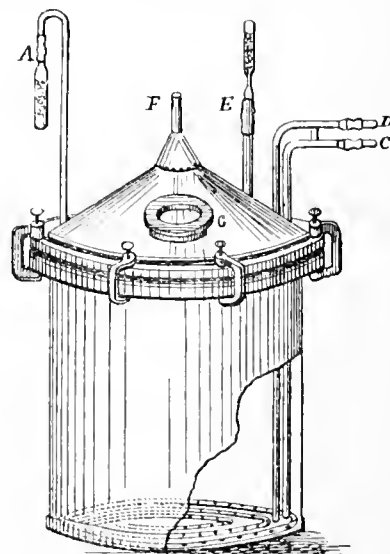
Barley-cider may easily be distinguished from ordinary cider, as a small quantity of a solution of tannin precipitates the tannates of the albuminoids derived from the barley, a reaction which does not take place with ordinary cider.

The new drink, which is more nourishing than cider, will probably replace it—at any rate, during seasons when there is a failure of the apple crop.

The author is continuing his investigations.—P. J. H.

*A New Apparatus for the Manufacture of Pure Yeast.* A. Fernbach. Bull. Soc. Chim. [3], 4, 113—116.

The apparatus consists of a tinned copper vessel furnished with a conical lid, which may be secured to it by clamps (see figure), a perfectly tight joint being produced by an



THE MANUFACTURE OF PURE YEAST.

india-rubber washer. There are two windows in the lid G, through which the contents of the vessel may be seen. Four tubes pass through the lid, one, A, passing to the bottom, and connected with a flattened coil lying on the bottom, and perforated with holes; a second, E, just passing through the lid; a third, D, passing to within 1 cm. of the bottom; and a fourth, C, passing down to a small recess in the bottom.

To use the apparatus, it is filled with a malt extract rich in maltose, and the contents boiled for some time so as to sterilise them and the vessel. The ends of the tubes A and E are connected by india-rubber tubing with short glass tubes loosely plugged with sterilised cotton wool, and the ends of the tubes C and D with short pieces of india-rubber tubing plugged with glass rods. When the apparatus is properly sterilised, the boiling is stopped and the vessel cooled by immersing it in cold water, and a stream of water allowed to trickle over it by the tube F.

When cold, the pure yeast is introduced by the tube E, after carefully removing the plugged glass tube, and replacing it as soon as the yeast is in. The liquid is kept well aerated by drawing a current of air through the apparatus by connecting E with an aspirator, the air entering at A. By this means a large growth of yeast is obtained.

When the fermentation is finished, the yeast is allowed to settle, and the fermented liquid drawn off by D, and then on agitating the liquid remaining, the yeast may be drawn off by the tube C.—A. L. S.

*A New Method for Determining the Amount of Sugar which it is necessary to add to Wines which are to become Sparkling with a Pressure of n-Atmospheres.*

M. E. J. Maumené. Bull. Soc. Chim. [3], 4, 119—122.

A COPPER vessel, silvered inside, is filled with a litre of the wine, and weighed; it is then connected with a manometer, and a reservoir of compressed carbonic acid gas. The absorption of gas is allowed to go on until the manometer indicates a constant pressure equal to the pressure required in the finished wine. The vessel, with the wine charged with gas, is reweighed, and the increase in weight shows the amount of carbonic acid required. The amount of sugar necessary to produce a given amount of carbonic acid is known, and so that the amount necessary in any particular case can be easily calculated. The only alteration in the wine after the fermentation of the added sugar, besides the production of carbonic acid, is the production of a small quantity of alcohol and other bodies, but the effect of these on the absorptive power of the wine is so small that they may be neglected.—A. L. S.

*The Preparation of certain Ethers by Fermentation.*

G. Jaquemin. Bull. Soc. Chim. [3], 4, 256—257.

DURING his studies on lactic fermentation the author has observed that under certain conditions mixed ethers are formed during fermentation. Sterilised barley-wort containing an excess of chalk was sown with a small quantity of a liquid in which a lactic fermentation had been previously started by means of cheese. This liquid contained lactic and butyric ferments and also yeast cells. The wort was contained in a flask so arranged as to permit the escape of gas but not the entrance of air. Some days afterwards the wort was found to possess an ethereal odour, and when distilled yielded a considerable amount of butyric ether and ethyl alcohol mixed with higher alcohols. The residue contained calcium butyrate and a little calcium lactate. A similar fermentation exposed to the air only led to the production of calcium lactate and very little calcium butyrate without any ethers. In another experiment, sterilised barley-wort was sown with a pure cultivation of the lactic ferment. After eight days' fermentation in presence of chalk and of pure air, a pure cultivation of *Saccharomyces ellipsoideus* was added, with the result that lactic ether was formed. The production of ether is probably owing to the facility afforded to the alcohol and acid of acting on each other whilst in the nascent state.

—H. T. P.

*New Uses for Fluorides and Hydrofluoric Acid.* Chemist and Druggist, 1890, 542.

J. EFFRONT, of Brussels, has described a number of experiments showing the beneficial influence of fluorides and hydrofluoric acid on diastase and yeast during the saccharifying and fermenting processes. In view of modern discoveries the effect seems to be one of selection, fluorides impeding or preventing the formation of lactic and butyric ferments, and thereby promoting pure alcoholic fermentation. Effront has succeeded in obtaining from maize 96 per cent. of the theoretical amount of maltose and 4 per cent. of dextrin, the solution yielding by simple evaporation crystallised maltose. The proportion of acid needed is very small, but not quite clearly stated. The temperature preferred is 30° C. instead of 50° C. as usual. To produce alcohol the author prefers potassium fluoride. He has found that with the addition of the fluoride maize may, without previous saccharification, be fermented at once after boiling to dissolve the starch and cooling. As a typical experiment, 9 kilos. of maize, 1 kilo. of malt, and water enough to make 54 kilos. of mash, on being, with 270 grms. of yeast and 27 centigrams. of potassium fluoride, set to ferment for three days at a temperature of 30° C., will yield in alcohol 63.26 per cent. of the weight of dry starch contained in the materials, while, under most favourable circumstances, the alcohol obtainable without fluoride averages 41 to 42 per cent. Another advantage claimed for the process is that, no butyric acid forming during the fermentation, the spirit obtained is free from unpleasant taste and smell. The process has been patented (this Journal, 1890, 960; Eng. Pat. 16,093 of 1889).

*Report on Wines.* Report of the Central Laboratory of the Italian Customs Department, 1836—1889, 3—59.

A SHORT time ago an extensive fraud on the Italian Customs was detected and exposed by the work of the laboratory. From Switzerland and Austria-Hungary so-called Cyprus, Samos, Smyrna, and Cephalonia wines were imported, but these liquids, generally of a musty odour, consisted of water, alcohol, and cane sugar; a red wine was made up by the addition of an aniline dye. These mixtures were worked up with suitable drugs and exported as vermouth. A double fraud was thus committed, *firstly*, on importation, when the liquids were assessed as wines and not as spirits; and, *secondly*, on exportation, when a claim was made for repayment of the duty on alcohol, a duty which had, however, never been paid. Analytical results are given of these mixtures, and the results compared with those of wines of undoubted genuineness.

In order to pass a judgment rapidly upon a sample the administration of the laboratory determined the proportion of alcohol by weight and by volume and the amount of sugar after inversion. The results are also given for the alcohol, by weight and volume, the grape sugar saccharose, and dry extract without sugar of 437 samples of typical genuine wine. It is also pointed out in the report that in the old Custom house tariff no definition was given of the word "wine," but in the recent (1888) regulations it is defined as follows:—

"Wine is the natural product of the fermentation of grape juice; under spirituous liquors shall be included so-called wines which contain saccharose, or other matters not contained in natural wines, as also those which are wanting in any of the characteristic components of wines, or which contain a quantity of these components in excess of the limits met with in natural wines or of the limits of the reciprocal relations found in these wines. Natural wines, of alcoholic strength of more than 15° shall pay, in addition to the proper duty on wine for every degree exceeding such a limit, the duty and surcharge on alcohol in the ratio of a litre of anhydrous spirit per degree and per hectolitre. Fractions of a degree less than five-tenths shall pass, in excess of five-tenths shall be counted as one degree."

The Department points out that in accordance with this definition the analysis of disputed samples of wine must be as complete as possible, so as to include not only artificial



beverages but also natural wines which have been sophisticated with water, alcohol, sugar, or glycerol. The method of analysis laid down by the German Imperial Commission of 1884 has been adopted, together with certain modifications proposed by the Austrian Wine Congress at Bozen (1887). Analyses made by these methods of 328 samples are given in a series of tables.

The beneficial effect of the work of the laboratory is shown in the relation of importation of wine and exportation during the last few years. In 1886 the importation of wine was about 300,000 hectolitres, and the exportation rose to 253,367 hectolitres. In 1887, when the fraud of the Cyprus wines was detected, the importation decreased to 132,520 hectolitres, though the duty remained the same. Again, from January 1st to June 30th, 1888, the importation was 24,670 hectolitres, but in the same period in 1889, under more rigorous regulations, the importation decreased to 5,604 hectolitres, a diminution traced, however, not only to the causes above mentioned, but also to others without the sphere of the laboratory.—V. H. V.

*Report on Syrups.* Report of the Central Laboratory of the Italian Customs Department, 1886—1889, 282—286.

The tariff report defines syrups generally as juices of fruit to which sugar has been added. If the juices of the fruit are natural they are classed in Category III. as orange juice, crude juice of citron and lemon, and concentrated juice of citron and lemon.

The report further lays down that "juice of orange, pine apple, berry, raspberry, pomegranate, ribes, prickly grape, and the like," pay as "orange juice"; "juice of orange, pine apple, &c. as above mixed with sugar, without alcohol pay as drinking syrups."

Juice of cherries, apples, and pears, fermented or not fermented, included as "elder," are classed with "wine." With syrups is also classed "carmine acid, mixed with sugar, which serves to colour saccharine matters," but syrups containing medicinal compounds are classed as medicines.

Analyses are given of samples sent, and two cases are specially mentioned of very intensely red liquids, of strong acid reaction, the one possessing the odour of pomegranate, the other of raspberry. It was stated on the labels of the bottles that syrup of pomegranate and raspberry could be prepared by adding one litre of the liquid to 100 litres of syrup of sugar. The Direction of the laboratory could not discover, however, the artificial colouring matter, and as no direct evidence could be obtained, these liquids were passed as fruit juice. Another sample was designated as a syrup of carabes and dried figs. The Government inquired if such a syrup might not be used as a substitute for starch syrup or even instead of glucose, in which case it should pay a manufacture tax. The laboratory replied that though it resembles an adulterated syrup of glucose, yet it should not be subjected to a manufacture tax. The product might be used as an inferior kind of food, but not for the manufacture of liquors or syrups; it might be used for certain industries, and even for the manufacture of alcohol.

—V. H. V.

*Report on Spirits.* Report of the Central Laboratory of the Italian Customs Department, 1886—1889, 59—64.

The tariff report distinguishes between "pure spirit and and sweetened or aromatic spirit," including rum, brandy, &c. In the former class are included those which have undergone no special preparation and are not extracts of substances which can impart an aroma to the distillate, while in the second are included liquids having alcohol as their bases and which are not classed under medicines, drugs, and essences.

Spirits, whether pure or not, are taxed according to the proportion of ethyl alcohol therein contained and determined by the official alcoholometer.

Disputes have arisen as to the difference between pure and sweetened spirit, especially if the samples are coloured yellow; an examination is then made to determine whether

the residue left on evaporation consists of tannin, derived from the wood of the containing cask, or of sugar. A liquid consisting of an alcoholic solution of cadmium, bromides, and iodides used in photography, was classed under sweetened and aromatic spirits. The laboratory has also been engaged in questions regarding a *manufacture tax* upon certain manufactured products obtained by extracting lees of wine with spirit; thus, as regards certain samples of various liquids of a red colour, the officials were in doubt as to whether wine had not been fraudulently added, while the importers maintained that the colour and vinous odour were derived from the wine casks in which the liquids had been kept. It was decided in favour of the latter side. Analyses are given of the various samples sent.

*Vermouth.*—This liquid is practically not imported into Italy. On exportation a *drawback* is made for the spirit and saccharose which it contains, it being supposed that the tax has been imposed in the course of the manufacture. The *drawback* for the sugar is settled, not as a result of analysis, but for a fixed proportion of 5 kilogrammes of saccharose per hectolitre. This drawback was conceded by a Ministerial decree, No. 5310 (Series 2), February 22, 1880, in which it is laid down that no drawback can be allowed unless the liquid contains sugar. The methods adopted for the determination of the sugars (dextrose, levulose, and invert) were the same as those used for wines. In all cases the amount of saccharose was in sufficient excess over the proportion fixed by the tariff for the *drawback*.—V. H. V.

## PATENTS.

*Improvements relating to the Pasteurisation of Beer, Wine, and other Liquids.* W. Kuhn, Clermont-Ferrand, France. Eng. Pat. 15,261A, October 23, 1888. Amended July 18, 1890. 6d

THIS patent (this Journal, 1889, 719), which was previously amended on May 31, 1890 (this Journal, 1890, 880) has been further amended by omitting the word "wine," and inserting the words "like fermentable" before the word "liquids" in the title. The body of the specification is amended in accordance, so that it only relates to beer and similar liquids.—A. L. S.

*Improved Processes of Maturing and Mellowing New Whiskey, Brandy, or like Alcoholic Liquors.* J. F. Gallogly, Dublin. Eng. Pat. 15,568, October 4, 1889. 4d.

THE "maturing" of the spirit is effected by inserting through the bung-hole or other opening of the containing vessel a prepared rod of charcoal or carbon. The carbon rod may be of any suitable size and shape, and is so arranged that its lower end dips into the alcoholic liquid. The rods are prepared by moulding and heating vegetable or animal charcoal, or a mixture of the two. Or a rod of oak, converted into charcoal and freed from noxious substances by some suitable treatment, may be employed. The rod of charcoal absorbs the deleterious substances from the spirit, and by bringing them into contact with the condensed oxygen in its pores, effects their conversion into wholesome flavouring bodies. For the further "mellowing" of the spirit the inventor immerses the containing vessel, either wholly or partially under water, the term "wet storage" being applied to this portion of the invention.

—H. T. P.

*An Improved Cooling Apparatus for Brewing and Domestic Purposes.* C. Huelser, London. From N. Reif, Hanover, Germany. Eng. Pat. 16,079, October 12, 1889. 8d.

THE object of this invention is to effect rapid cooling with comparatively little water. For this purpose a cylindrical mashing tub is surrounded by a circular porous cooling water reservoir, the outer mantle of which is corrugated in order to increase its surface. This mantle is perforated, and a second smaller cylinder, also perforated, is placed between the tub and the reservoir, the intervening spaces being packed with wood-flock or other fibrous or porous material.



The whole arrangement is surrounded at some distance by an outer casing in which a greater or less degree of rarefaction can be maintained by means of a ventilator. The rate of evaporation of the water passing through the porous reservoir is thus accelerated. The moist atmosphere removed by aspiration is forced into a box where a portion of the water condenses and is returned to the reservoir. The remainder of the moist air is diffused through a fan-shaped opening over the surface of the liquid in the tub, thus leading to further cooling. The cooler for domestic purposes is similar in principle to the above apparatus.

—H. T. P.

*An Improvement in the Manufacture of Beer.* H. T. Brown and G. H. Morris, Burton-on-Trent, and E. R. Moritz, London. Eng. Pat. 19,003, November 26, 1889. 6d.

MALT worts contain a greater or less proportion of substances called amyloins. Amyloins are unfermentable during the primary stage of wort fermentation, but are slowly decomposed during the secondary fermentation in cask. Amyloins are rapidly converted into maltose under the influence of diastase. When unfermented, the amyloins are mainly the source of the palate-fulness of beer; afterwards it is to the result of their fermentation that the "condition" or briskness of beer is due. The object of this invention is to ensure the presence of amyloins in beer in such proportion as may be most suitable to obtain the required palate-fulness and condition. If the wort be deficient in amyloins a sufficient quantity may be at once added. In presence of excess of amyloins diastase is added in order to destroy them; afterwards the correct proportion of amyloins is restored to the wort. The method for the estimation of amyloins, given in this Journal, 1890, 533—535, usually suffices, according to the patentee. A wort should contain about 12—20 per cent. of amyloins, reckoned on the wort solids before fermentation. Amyloins may be added to the beer after primary fermentation, alone, or along with other brewing sugars, and with or without cold-water malt extract, according as to whether a rapid or slow secondary fermentation is desired.

Amyloins may be prepared in the following manner: 100 parts of starch, 300 parts of water, and 3 parts of sulphuric acid are boiled together until the product has a specific rotatory power of about  $193^\circ$ , and a copper oxide redning power of about 21, the cold sample giving at the same time a full red colour with iodine solution. At this stage the boiling is discontinued, and the liquid neutralised. The liquid should be filtered and concentrated. By varying the proportions of the materials, and the conditions as to time or temperature under which the reaction takes place, various amyloins may be produced.—H. T. P.

*Improvements in Maturing Spirits and other Liquors and Apparatus connected therewith.* J. McKinless, Manchester. Eng. Pat. 4108, March 17, 1890. 8d.

This is for the purpose of maturing spirits by compressed air or oxygen, or a mixture of these. Two kinds of apparatus are described. The first consists of a closed cylinder, furnished with two or more perforated diaphragms, one near the top being of metal, with small holes about  $\frac{1}{4}$  inch diameter, and the others of wire gauze, with meshes about 30 to the inch. An air pipe communicates between the space between the diaphragms and the top of the vessel, so that as the liquid runs down the air may pass up.

The liquor to be treated is run into the vessels, the pressure of air within increased by about  $\frac{1}{2}$  atmosphere and the liquid drawn off at the bottom and passed in again at the top by a pump, whence it falls through the vessel, being divided into a fine spray by the diaphragms. This is repeated until the spirit is properly matured by the oxidation.

In the second form of apparatus, the circulation of the liquid, instead of being effected by a pump, is brought about by inverting the vessel at intervals.—A. L. S.

*An Improved Means of Applying certain Antiseptic Agents to Beer, Wine, or other Fermented Drinks.* A. Boake and P. G. A. Roberts, Stratford. Eng. Pat. 10,845, July 12, 1890. 4d.

The antiseptics are weighed out in convenient amounts, mixed with about 5 per cent. of inert gummy matter, and compressed in moulds, so that they become a coherent mass when the pressure is withdrawn. The tablets may be conveniently  $\frac{1}{8}$  oz. in weight.—A. L. S.

*Improvements in "Pasteurising" Apparatus.* A. Regel, Schöningen, Germany. Eng. Pat. 11,249, July 18, 1890. 8d.

The apparatus consists of two closed cylinders whose lower ends are connected by unions with a pipe, which serves to convey the beer to be pasteurised from the cask to the apparatus. The cylinders are jacketed outside, and each is provided with an air-tight piston, operated by compressed air. A thermometer passes through each piston so that the temperature of the space beneath it can be determined. The top of each cylinder is fitted with a glass dome, into which the thermometer fixed in the piston can enter, so that its scale can be observed from the outside. Further, each cylinder carries at top a pressure gauge and a tap for the entrance of compressed air. The beer to be pasteurised is forced in at the bottom of the first cylinder by means of air pressure. The entrance of the liquid causes the piston to rise, and the moment when the latter reaches its highest point is indicated by an electric alarm, operated by the piston itself. The beer is now heated to  $82^\circ$  C. by passing steam through the outer jacket of the cylinder. As soon as the beer has reached the required temperature, it is transferred to the second cylinder by forcing air into the top of the first cylinder, thus causing its piston to descend. The beer is rapidly cooled to  $5^\circ$  C. in the second cylinder by passing warm water and then ice-cold water through the outer jacket. The beer is subsequently forced by means of compressed air through a froth-preventing device, and from thence through a draw-off cock and pipe into bottles or casks.—H. T. P.

*Improvements in the Manufacture of Wine and in Apparatus therefor.* G. F. Redfern, London. From G. Perscheid, Jersey City, U.S.A. Eng. Pat. 13,257, August 22, 1890. 6d.

This is for the purpose of manufacturing effervescent wine in bulk, allowing the wine to stand until clear and then bottling the clear wine without loss of carbonic acid. The wine is fermented in a wooden vat, the bottom of which is furnished with a number of hollows to retain the lees. The vat is contained in an air-tight receiver capable of resisting considerable pressure. The wine in fermenting evolves carbonic acid, which raises the pressure within the receiver; if the pressure becomes too great, the excess of gas is allowed to escape into a separate reservoir. When the fermentation is complete, the wine is cooled by an ice jacket, and allowed to stand 12 hours in order that it may become clear; it is then led through a filter into the bottles to contain it, a second pipe from the receiver leading the carbonic acid to the bottles, and the bottling is conducted as usual with aerated water.—A. L. S.

*Improvements in Apparatus for Cooling and Aërating Wort in the Manufacture of Malt Liquors.* H. H. Lake, London. From the Hanford-Stanford Company, Portland, U.S.A. Eng. Pat. 13,428, August 26, 1890. 8d.

This describes improvements in the apparatus described in Eng. Pat. 4982 (this Journal, 1890, 960); these are:—

1. An arrangement whereby the spray is thrown over only half or a quarter the area of a circle, over the centre of which the apparatus is situated. This is done by continuing the walls of the vertical tube downwards along half or three-quarters of its circumference; so that along this part no annular slit is formed when the conical deflector is

pulled down. Thus it is possible to arrange the spray producers over the edges of the cooler and have the wort thrown so that it may all fall into the cooler.

2. The conical deflector is furnished with a shoulder, so that the film of wort, as it issues through the annular opening, is deflected upwards and outwards, thus producing a more perfect spray and a more rapid cooling.

3. The cords connected with the levers to which the conical deflectors are attached are all brought to a common axle which may be moved by a lever, thus causing the deflectors to move away from the ends of the tubes and produce a simultaneous flushing. The axle is furnished with an arrangement, whereby the cords controlling the deflectors may be separately adjusted. Detailed drawings of the apparatus accompany the specification.—A. L. S.

## XVIII.—CHEMISTRY OF FOODS, SANITARY CHEMISTRY, AND DISINFECTANTS.

### (A.)—CHEMISTRY OF FOOD.

*On the Phenomenon of Coagulation.* A. Béchamp. Bull. Soc. Chim. [3], 4, 181—186.

See under XXIII., page 1073.

*Report on Cocoa.* Report of the Central Laboratory of the Italian Customs Department, 288—289.

A DESCRIPTION is given of certain samples sent to the laboratory. It was decided to class a sample declared as cocoa powder, resembling the products found in commerce as soluble cocoa of Van Houten, &c., as "cocoa in powder or paste" and not as "chocolate." Certain samples were found to be sweetened, not with sugar, but with saccharine, though the importation of saccharine and products containing saccharine is strictly prohibited by Royal Decree No. 6407 (Series 3), September 29, 1889 (Journal, 9, 831). An exception is, however, made in the case of pharmaceutical products by Ministerial Decree No. 6416 (Series 3), October 5, 1889.—V. H. V.

### PATENT.

*A New or Improved Process and Apparatus for Preserving Organic Substances, and especially Alimentary Substances.* C. L. Bachelerie, Paris, France. Eng. Pat. 13,089, August 20, 1890. 8d.

THE articles—carcases of animals, poultry, fish, eggs, vegetables, &c.—are exposed, under a pressure varying with their size, to the carbonic anhydride and spray of hydrochloric acid evolved by the sudden addition of the desired quantity of hydrochloric acid to the requisite amount of sodium hydrogen carbonate.

The invention includes the design for a chamber capable of resisting the pressure, and fitted with movable supports for the material or articles, with arrangements:—for tipping the hydrochloric acid all at once on to the carbonate, for charging the chamber, for washing out the products, and for discharging them and also provided with the requisite safety-valves, indicators, &c.—D. A. L.

### (B.)—SANITARY CHEMISTRY.

*Purification of Waste Water from Sugar Refineries, &c.* H. Schreib. Chem. Zeit. 14, 1323—1324.

See under XVI., page 1053.

*On the alleged Sterilisation of River Water by Mine Water.* A. A. Breneman. J. Amer. Chem. Soc. 12, 12—15.

DURING an epidemic of typhoid fever which occurred in the city of Wilkesbarre in 1889, questions arose as to the purity of the different sources of water upon which the city depended, and the author was called upon to examine the waters. It was found that the supply from the Susquehanna river was preferred by the public and by local physicians in spite of the fact that it receives above Wilkesbarre the sewage of nearly 100,000 people in the course of 10 miles. It was believed by the people of Wilkesbarre that the river water was peculiarly free from possibility of transmitting infectious diseases because of the considerable quantities of mine water which it receives, the mine water being supposed to exert a sterilising effect through its well-known constituents, free sulphuric acid and sulphates of iron, but the results of the examination of the waters were sufficient to dispel this fancied security. Samples of water were taken:—1. From the Susquehanna river at the intake of the Wilkesbarre Water Co. 2. From the mouth of the shaft of the Hollenbach coal mine, the drainage from which runs into the river above Wilkesbarre. 3. (For comparison only) from the Hazleton coal mine at a depth of 300 feet from the surface.

### RESULTS OF CHEMICAL EXAMINATION.

	Grains per Gallon.					
	Total SO <sub>3</sub>	Free H <sub>2</sub> SO <sub>4</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	SiO <sub>2</sub>
River .....	1.00	.00	.71	1.88	.16	..
Hollenbach mine	51.16	15.59	12.23	8.21	4.00	2.50
Hazleton mine...	127.30	20.85	65.40	2.55	4.27	2.70

The river water was alkaline in reaction and contained no iron in solution, the portion present being suspended ferric oxide. These data alone dispel the idea that the river water can retain at Wilkesbarre any of the germicidal power that may be possible to salts of iron or free sulphuric acid, whilst the effect of any inflow from the mines is shown to be insignificant. Fresh samples of the waters 1 and 2 were taken in sterilised bottles for bacteriological examination. The river water was found to contain 400 to 500 germs per cc. of at least 14 different species, six of these being of species that liquefy gelatin (putrefactive bacteria), and after sterilisation by discontinuous heating, the water was found capable of supporting the growth of true typhoid bacillus introduced by inoculation. The growth of typhoid bacillus was also possible in No. 2 after sterilisation, but not in No. 3. It is evident from these results that no effective sterilisation of the water of any large stream can result from the small and varying contributions made to its volume by mine drainage, and even mine waters of considerable strength, and undiluted, cannot be relied upon for certain effect against the high resisting power that the typhoid bacillus is known to possess.

It is certain, moreover, that a water rich enough in sulphuric acid or iron salts to act as a disinfectant would be entirely unsuited for drinking.—G. H. B.

### PATENTS.

*Improvements in and relating to Apparatus for Handling and Treating Sewage.* W. S. West, New York, U.S.A. Eng. Pat. 8239, May 27, 1890. 8d.

THE invention consists of arranging a pumping station for distributing sewage from sewers on to filter beds or elsewhere in such a manner that paper, insoluble matters and grease are intercepted and good ventilation is provided. The sewage enters the underground portions of the building, the discharge being regulated by gate-valves, and passes on to screens constructed of boards and framework filled in with sawdust or other suitable combustible material, and

supported on beams. Paper, grease, &c., are retained by this screening material, which is from time to time cleared out and burnt; whilst the liquid portions run on to the inclined floor in a chamber below, and collect in a tank in which suction pumps work. For ventilation the chimneys from the boilers are enclosed together in one external wall, leaving an intervening air space all round them, extending from the bottom to the top of the chimney; this air space connects with the underground chambers and with the sewage inlet pipes, and at a suitable height up also with the main chimney by openings in the wall, so that noxious fumes may not only be drawn up but portion of them also consumed. Suitable ducts are provided for conducting fresh air down into the underground chambers both above and below the screening floor. The invention includes an arrangement for preventing clogging at the connexion of the house with the sewer pipe and a pan for reducing grease, obtained from the sewage, by means of waste heat.

—D. A. L.

*Improvements in and relating to Preserving Compounds for Embalming Purposes.* W. P. Thompson, Liverpool. From J. R. Bate, F. W. Owen, and J. W. Simcock, Detroit, U.S.A. Eng. Pat. 13,046, August 19, 1890. 4d.

THE body to be preserved is placed, without any preliminary preparation, not even removal of clothes if human, in a chamber and exposed to fumes produced by the combustion of a mixture containing:—three parts of sulphur, three parts of charcoal, two parts of borax, two parts of sodium chloride, and two parts of calcium chloride all powdered and dry.

According to the patentees, “a chemical union takes place between the fumes or gases thus produced and the liquids of the body, producing acids which arrest present and prevent future decomposition.”—D. A. L.

## (C)—DISINFECTANTS.

## PATENTS.

*An Improved Means of Applying Certain Antiseptic Agents to Beer, Wine, or other Fermented Drinks.* A. Boake and F. G. A. Roberts, Stratford. Eng. Pat. 10,845, July 12, 1890. 4d.

See under XVII., page 1057.

*The Manufacture and Production of a New Antiseptic.* J. Y. Johnson, London. From E. Salomon, Paris, France. Eng. Pat. 13,488, August 27, 1890. 4d.

To produce a non-poisonous but powerful antiseptic a solution containing from 1 gm. to 0.5 gm. of mercuric chloride per litre is mixed with 8 to 10 per cent. of sodium chloride, and with a variable proportion of a sulphate such as zinc, copper, or best of all cadmium sulphate, tartaric acid may also be added. The accessory constituents are intended to contribute emetic properties and prevent poisoning by absorption.—D. A. L.

## XIX.—PAPER, PASTEBOARD, Etc.

*On the Qualitative Determination of Mechanical Wood-Pulp in Paper.* W. Herzberg. Mitt. Königl. tech. Versuchs. 1890, 132—140.

See under XXIII., page 1068.

*On the Quality of Certain Hand-Made Papers.* W. Herzberg. Mitt. Königl. tech. Versuchs. 1890, 141—143.

DIFFERENCES which occur in the tearing length and elongation at fracture in testing the same paper, originate partly in inequalities of the paper itself, partly in the changes of temperature and moisture of the testing room, and partly in the apparatus used for the tests (see Mitt. Königl. tech. Versuchs. 1887, 36, and 1888, 78). A series of seven different hand-made papers tested at the beginning, in the middle, and at the end of the manufacturing process, gives some further information on this subject. The tests were made with Wendler's apparatus, strips of 180 × 5 mm. being torn in both directions (five across and five along the web) for each test. No regular changes of the tearing length and elongation at fracture have been found in comparing the three mentioned stages of manufacture, and the variations are comparatively small. The maximum differences were found in the two following tests:—

Stages in Manufacture.	Ash.	Weight.	Tearing Length.			Elongation at Fracture.			Difference from Total Mean.		Temperature of Testing Room.	Moisture of Testing Room.
			Along Web.	Across Web.	Mean.	Along Web.	Across Web.	Mean.	Tearing Length.	Elongation at Fracture.		

## I.—LINEN FIBRES, WOOD CELLULOSE, AND COTTON FIBRES.

	Per Cent.	Grm.Sq.M.	Km.	Km.	Km.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	° C.	Per Cent.
Beginning.	12.0	252	2.98	2.16	2.57	4.1	4.8	4.5	+4.47	+18.4	15.2	60
Middle....	12.0	332	2.63	2.24	2.44	3.2	3.5	3.4	-0.81	-10.5	16.1	56
End.....	11.5	315	2.47	2.26	2.37	3.1	4.0	3.6	-3.66	-5.3	16.2	58

## II.—WOOD CELLULOSE AND A SMALL QUANTITY OF MECHANICAL WOOD PULP.

	Per Cent.	Grm.Sq.M.	Km.	Km.	Km.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	° C.	Per Cent.
Beginning.	15.3	409	2.36	2.24	2.30	2.7	3.5	3.1	-6.50	-8.8	14.8	55
Middle....	15.0	365	2.38	1.97	2.18	3.1	3.6	3.4	-11.38	± 0	16.7	60
End.....	6.8	383	3.04	2.74	2.89	3.6	3.7	3.7	+17.48	+8.8	16.2	56

No satisfactory explanation is given for the remarkable change of percentage of ash in the second sample, no similar differences being found in any other cases of the whole series. The manufacturer tried to account for it by

suggesting the paper had been improperly treated with china clay, but admitted that a mistake in the manufacture is not impossible.—H. S.

## PATENTS.

*Improvements in the Process of Manufacturing Thin Sheets of Nitro-Cellulose.* E. N. Todd, Newark, New Jersey, U.S.A. Eng. Pat. 9315, June 16, 1890. 8d.

NITRO-CELLULOSE is dissolved in a mixture of acetate of methyl, acetate of amyl, fusel oil, and oil of camphor, with or without the addition of solid camphor. Glass plates are suspended from the top in a vessel fitted with a cover and provided at the bottom with a valve. The glass plates being in position the solution of nitro-cellulose is run in. After remaining some time to allow the escape of air bubbles the solution is slowly run off, leaving a thin layer on the glass plates which quickly dries as a thin film of transparent celluloid. When thoroughly dry the films are stripped from the sheets of glass and the latter are transferred again to the vessel.—E. J. B.

*Improvements in the Preparation or Treatment of Paper for Copying Letters, Documents, and the like, including such as are done with a Type-Writing Machine, and Improvements in Chemical Compounds or Preparations therefor.* W. Rogers, Liverpool. Eng. Pat. 12,139, August 2, 1890. 4d.

For press-copying type-writing the inventor used a mixture of 9 parts of methylated spirit and 1 part of glycerin instead of water. When type-writing and ordinary writing are to be copied at the same time he used a mixture of the above solution with what is known as Roger's Copying Preparation (Eng. Pat. 14,274 of 1886), together with the addition of one or other of the following chemicals: oxalate of potassium or sodium, sulphide of ammonium, vanadate of ammonium, chromate or bichromate of potassium, ferri- or ferrocyanide of potassium, galle, pyrogallie or tannic acid.—E. J. B.

## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

*Iso- and Allo-Cinnamic Acid.* C. Liebermann. Ber. 23, 2510—2516.

ISO-CINNAMIC acid has been found to be associated with another acid, to which the name of *allo-cinnamic acid* is given, in the acid liquors obtained as a by-product in the manufacture of cocaine, after the removal of the insoluble acids (this Journal, 1890, 541). The two acids resemble each other closely, but differ in their crystalline form, melting point, and solubility in petroleum spirit. This solvent was employed for separating them, *allo-cinnamic acid* being the less soluble. Analyses of the new acid agree with the formula  $C_9H_8O_2$ , i.e., with its being an isomer of cinnamic acid. It melts at  $68^\circ$  (*iso-cinnamic acid* melts at  $57^\circ$ ) and crystallises in the monoclinic system.

Several of the salts and properties of the new acid are described and compared with the corresponding ones of the *iso*-compound.—C. A. K.

### *Theoretical Considerations on the Truxillie Acids.*

C. Liebermann. Ber. 23, 2516—2518.

FOUR truxillie acids (di-cinnamic acids) have been described by the author, all of which he regards as derivatives of tetramethylene.

The formation of cinnamic acid by the distillation of the truxillie acids is also regarded as being a proof of their being derived from tetramethylene.—C. A. K.

*The Oxidation of Ecgonine.* C. Liebermann. Ber. 23, 2518—2522.

EINHORN has pointed out (this Journal 1890, 890) the near relationship existing between tropidine and ecgonine, since anhydro-ecgonine is decomposed by heating with hydrochloric acid to  $280^\circ$  with formation of tropidine.

Should this constitution for anhydro-ecgonine be correct, then the author regards it as probable that ecgonine itself is tropin-carboxylic acid, with the carboxyl group in the side chain, in which case both anhydro-ecgonine and ecgonine should yield when oxidised under like conditions the same oxidation products. The author has studied the oxidation products of ecgonine by means of chromic acid, following Merling's directions.—C. A. K.

*The Formation of Lepidine Derivatives from Quinine and Cinchene.* W. Koenigs. Ber. 23, 2669—2679.

COMSTOCK and Koenigs (this Journal, 1885, 64) have shown the relationship of quinine and conquinine  $C_{20}H_{22}N_2O_2$  to quinine  $C_{20}H_{22}N_2O_2$ , and of cinehonine and cinechonidine  $C_{19}H_{20}N_2O$  to cinchene  $C_{19}H_{20}N_2$ .

Quinine, cinehonine, and cinchene are now studied by the author, and further derivatives are prepared.

Since cinechonine is decomposed by concentrated alkali with the formation of both quinoline and lepidine it would be expected that quinine would yield para-methoxylepidine in conjunction with the para-methoxyquinoline which is known to be formed under the same conditions. In fact Krakau (this Journal, 1885, 688) has shown that when the cinchona alkaloids are heated with caustic potash to  $200^\circ$  in a current of superheated steam a base is formed whose hydrate melts at  $52^\circ$ . The author has repeated this experiment and finds the base to be para-methoxylepidine, identical in all its properties with the base obtained from quinine.

It is found not improbable that the carbon atoms attached to the quinoline residue in quinine and cinchene are doubly linked, thus:  $-C_9H_6N-CH=CH-$  and that in quinine and cinechonine these carbon atoms are attached both to a hydrogen atom and to a hydroxyl group, which would account for the greater difficulty with which they are decomposed when compared with their "anhydro bases," quinine and cinchene (see also this Journal, 1887, 146 and 736.)—C. A. K.

*Substitution Products of Lepidine.* A. Busch and W. Koenigs. Ber. 23, 2679—2688.

THE following derivatives of lepidine have been prepared:—*Para-lepidine sulphonic acid*, *Benzylidene-lepidine sulphonic acid*, *Para-hydroxylepidine*, and *Para-amido-lepidine*.

Lepidine is treated with a mixture of fuming nitric and sulphuric acids in the cold, and the nitro-compound formed, crystallised from hot alcohol. It is colourless and melts at  $126^\circ$ — $127^\circ$ . Other products appear to be formed in the reaction.—C. A. K.

*Crystalline Veratrine.* F. B. Ahrens. Ber. 23, 2700—2707.

THE material for the following experiments was obtained from Merck of Darmstadt, the identity of the veratrine itself being established by an analysis of its gold double chloride, which gave values corresponding with the formula  $C_{32}H_{49}NO_9 \cdot HCl \cdot AuCl_3$ . The double salt, when dried at  $100^\circ$ , melts, with decomposition, at  $182^\circ$ . The mercuric chloride double salt can also be obtained crystalline from alcoholic solution. It melts and decomposes at  $172^\circ$ . When an aqueous solution of picric acid is added to an alcoholic solution of veratrine hydrochloride, crystals of the picrate separate on standing. These are very stable, and only begin to decompose at  $228^\circ$ . All these salts are very difficultly soluble in water and fairly readily soluble in alcohol.

With platinum chloride, veratrine hydrochloride forms an amorphous precipitate, which dissolves in alcohol, but is decomposed by water.

Veratrine, when treated with bromine water, readily takes up 4 molecules of the halogen, to form a tetra-brom-addition product,  $C_{25}H_{49}NO_5Br_4$ , an amorphous yellow powder insoluble in water, soluble in alcohol, ether, &c. This body is easily decomposed by dilute alkali to form a dibromo-addition product,  $C_{25}H_{49}NO_5Br_2$ , also an amorphous body. Substitution products appear also to be formed when veratrine is treated with bromine, but were not isolated. Aqueous caustic potash, baryta water, ammonia, and even water in a closed tube at  $200^\circ$ , all decompose veratrine more or less completely into angelic acid,  $C_5H_8O_3$ , and a base which appears to have the composition  $C_{25}H_{45}NO_3$ . The latter is probably identical with the so-called "cevin," and differs very little from "ceirdine," the bases obtained by Wright and Luff and by Bosetti respectively, in decomposing veratrine by means of alkali.

By heating veratrine with concentrated hydrochloric acid, tiglic acid,  $CH_3 - CH = C.(CH_3).CO.OH$ , is obtained, together with a basic substance of unknown composition. Oxidising agents act very energetically upon the alkaloid; nitric acid decomposes it completely. With potassium permanganate, acetic and oxalic acids result, whilst chromic acid yields carbon dioxide and acetaldehyde. When subjected to dry distillation, tiglic acid and  $\beta$ -picoline are formed, whilst when heated with lime  $\beta$ -picolin, a secondary base,  $\beta$ -pipercoline,  $C_6H_{13}N$ , and iso-butane appear to result.—C. A. K.

#### On Strychnine. J. Tafel. Ber. 23, 2731—2739.

TWENTY years ago Stahl Schmidt found (Pogg. Ann. 108, 513) that methyl iodide forms with strychnine an addition product from which may be obtained the base methyl-strychnine. The author has undertaken an examination of this compound and of its further derivatives, with a view to ascertaining the constitution of strychnine. The strychnine-methyl iodide Stahl Schmidt looked upon as a hydriodide, viz.,  $C_{23}H_{29}N_2O_3.IH$ , although it is unacted on by potash. Strychnine-methyl iodide has the true character of an ammonium iodide, it possesses a neutral reaction, and gives, in aqueous solution with silver oxide, an alkaline base. This base, when neutralised with hydriodic acid, gives the methyl iodide again. It is, without doubt, the ammonium hydroxide corresponding to the strychnine-methyl iodide.

By molecular change from this ammonium base, methyl-strychnine is obtained.

Methyl-strychnine unites at ordinary temperatures with methyl iodide, forming a methyl iodide,  $C_{23}H_{29}N_2O_3.I$ , which with silver oxide gives an alkaline substance, but as secondary product a beautifully crystalline neutral body of the formula  $C_{23}H_{28}N_2O_3$ . This the author calls dimethyl-strychnine. It forms salts, which monomethyl-strychnine does not do.

Dimethyl-strychnine further unites with methyl iodide, giving a product of the composition  $C_{24}H_{31}N_2O_3.I$ . This product is a true methyl iodide, giving an alkaline basic substance on treatment with silver oxide; all attempts at isolation proved fruitless, however, strychnine itself being always re-formed—



The author has also undertaken an examination of the hydrate of strychnine.

Gal and Etard (Bull. Soc. Chim. 31, 98) obtained a compound,  $C_{21}H_{26}N_2O_4$ , by heating strychnine with baryta water in a closed tube at  $130^\circ$ — $140^\circ$ ; this compound they called dihydro-strychnine. Loebisch and Schoop (Monatsh. 7, 83), by the action of alcoholic sodium hydrate on strychnine, obtained a compound which they called "strychnol." This, from the present memoir, appears to differ from the preceding compound by the elements of water. Hence, the former substance is termed here strychnine dihydrate, the latter strychnine monohydrate.

The soda salt of strychnine dihydrate by the action of methyl iodide gives the methyl iodide of a tetramethyl-strychnine dihydrate.—T. L. B.

#### Tropidine. A. Einhorn. Ber. 23, 2889—2894.

TROPIDINE, when treated with an aqueous solution of hypochlorous acid, yields two products, which can be separated by virtue of their different solubilities in a mixture of alcohol (2 parts) and water (3 parts). The more soluble of these melts at  $108^\circ$ — $109^\circ$ , and appears to have the composition  $C_5H_{13}N(HClO)_2$ . The less soluble body was only obtained in small quantity, and no analysis is given; it melts at  $138^\circ$ . Further investigations on the decomposition products of this body are delayed by the author, owing to the publication of Ladenburg's work on allied subjects (Ber. 23, 1780 and 2225).—C. A. K.

#### Contribution to the Knowledge of Papaveroline. K. Krauss. Monatsh. 11, 350—362.

PAPAYEROLINE is prepared by heating 30 grms. of papaverine and 250 grms. of hydriodic acid (sp. gr. 1.96) for 10—11 hours. On cooling, the hydriodide of the base crystallises out, and is purified by recrystallisation from water. The free base is obtained by adding a solution of sodium bicarbonate saturated with carbon dioxide to a cold saturated solution of the hydriodide. The base is precipitated as a white crystalline powder. It has a composition expressed by the formula  $C_{16}H_{13}NO_4.2H_2O$ . It contains four hydroxyl groups and is consequently a very feeble base, being precipitated from solutions of its salts by sodium acetate.

The base is easily soluble in dilute mineral acids and oxalic acid and also in acetic acid and glycerol, even if somewhat dilute. It is fairly easily soluble in alcohol, especially in warming. It is but slightly soluble in ether and chloroform and insoluble in benzene, petroleum spirit, and water. A weak alcoholic solution gives a fine blue colouration with a little potash, and with an excess of the latter a dark reddish-violet colouration; addition of hydrochloric acid causes the colour to become brown and potash again converts this into blue.

The author has also investigated the products of the distillation of the base with zinc; he obtained  $\alpha$ -methyl-isquinoline and another base which he was not able to identify with certainty.—A. L. S.

#### A Double Salt of Cocaine. W. Müller. Pharm. Zeit. 35, 522.

On mixing fairly concentrated solutions of mercuric chloride and cocaine hydrochloride, the double salt  $C_{17}H_{19}NO_4.HgCl_2.2H_2O$  separates as a bulky precipitate, having a fine crystalline appearance after washing, and drying in the desiccator. It melts at  $124^\circ$  C. to a clear colourless liquid, which solidifies on cooling to an opaque mass. It has a distinct metallic taste characteristic of mercury compounds, perceptible in spite of the anæsthetic action of the cocaine on the tongue.—B. B.

#### The Preparation and Properties of Aricine. H. Moissan and E. Landrin. Bull. Soc. Chim. [3], 4, 257—260.

ARICINE was first prepared and described by Pelletier and Coriol, who obtained it from a bark bearing the name of "Quinquina de Cusco" or "Arica bark." Since then the alkaloid has been examined by various observers. The bark employed by the authors was of a grey colour externally and reddish inside. It contained neither quinine nor cinchonine, and was particularly rich in aricine, of which it contained 3—3.5 per cent. The method adopted for extracting the alkaloid was as follows.—One kilo. of the coarsely powdered bark is mixed with 100 grms. of lime and 100 grms. of soda lye at  $40^\circ$ , the mixture being evaporated almost to dryness on the water-bath. The residue is continuously shaken for half an hour with 4 litres of ether. The ether is decanted and shaken for a short time with 100 grms. of sulphuric acid (1 in 10) and 60 cc. of water. Insoluble aricine sulphate separates as a yellowish cheesy mass. The ether is decanted, being employed to extract a further quantity of alkaloid from the bark. The ethereal extract is treated with sulphuric acid as before, and so on, the operation being repeated about six times. The aricine

sulphate is separated from the united acid liquids by filtration. The clear filtrate gives with caustic soda a precipitate of an uncrystallisable alkaloid which will be referred to at some future time. To obtain the arietine in a state of purity, 150 grms. of the crude sulphate are dissolved in 5 litres of boiling water, and ammonia is added until the base is completely precipitated. 115 grms. of a viscous mass are thus obtained which solidifies on cooling to a friable substance resembling resin. The precipitate is powdered and dissolved in boiling alcohol at the rate of 100 grms. per litre. On cooling the arietine crystallises out. The operation is repeated until the crystals are colourless.

Arietine forms beautiful transparent crystals, 5—8 mm. in length, which on exposure to sunlight assume after some time a slight yellow tinge. If the crystals be not quite pure the colouration is rapidly produced. Arietine is insoluble in water; at 15° C., one litre of alcohol dissolves 10 grms. and at the boiling point, 90 grms. Ether takes up 30 grms. per litre at 15° C. Arietine melts at 188°—189° C. Its specific rotatory power in alcoholic solution (alcohol of 95 per cent.) was found to be  $(\alpha)_D = -58.18'$  at 16.6° C., the solution containing about 0.8 gm. per 100 cc. In alcoholic hydrochloric acid solution arietine has the specific rotatory power  $(\alpha)_D = -14.30'$ .

The melting point and optical activity serve to differentiate arietine from euscimine, with which it is isomeric.—H. T. P.

*The Manufacture of Commercial Hydrogen Dioxide and its Applications.* A. Bourgongnon. J. Amer. Chem. Soc. 12, 64—69.

THE author describes the following method for the production of hydrogen peroxide on a commercial scale. The first step, and a very important one, is the hydration of the barium peroxide, which is effected by pouring the powdered substance into a cylindrical stone pot half full of water, and stirring the mixture with a wooden spatula occasionally during three or four hours, when the barium peroxide forms a thick, perfectly white and smooth, pasty mass resembling white clay mixed with water. While the hydration is progressing, a mixture of water and hydrofluoric acid is made in a vessel lined with sheet lead and surrounded with ice water so as to keep the temperature of the acid mixture below 10° C. The hydrated barium peroxide is then added in portions of three to four pounds at a time to the acid mixture, stirring all the time to mix the contents of the vessel thoroughly. All the barium is transferred to the acidulated water in about two hours, and the agitation continued for four hours. If the operation has been well conducted all the barium peroxide is transformed into fluoride, which falls to the bottom of the vessel. When this precipitate is well settled the supernatant liquid is decanted into a vessel similar to the one used for its production, and also surrounded by ice-water. The clear liquid contains an excess of acid and impurities derived from the materials employed, consisting chiefly of ferric oxide, alumina, and manganese oxide, which must be removed to insure the keeping properties of the product. To this end small quantities of hydrated barium peroxide are added with stirring to the cold solution until the last traces of acid are saturated, when the appearance of the solution suddenly changes from a bright yellow to a greyish colour, and the impurities separate and collect at the surface of the liquid. Without losing time the liquid is filtered through a cheese cloth stretched on a frame, and received in a vessel which contains a small quantity of dilute sulphuric acid (one part acid to eight parts water). This filtration must be quickly done. As long as the solution is alkaline there is great danger of decomposition and loss of oxygen. If the liquid is thrown upon the filter the latter will be quickly clogged, the filtration stopped, and the hydrogen peroxide will rapidly decompose, emitting large bubbles of oxygen with a hissing noise; but as the precipitated impurities collect first on the top of the liquid the solution is syphoned from below these impurities, and it filters quickly and satisfactorily. The filtered liquid is tested for barium and sulphuric acid is added until all is removed. The liquid is left over night, the precipitate of barium sulphate settles,

and the perfectly clear and colourless solution is ready for the market. All the precipitates are separated by pressure from the liquid they contain, which is added to the water employed in the next operation. Nearly 40 gallons of hydrogen peroxide were produced at the following cost:—

	Dollars.
60 lb. barium peroxide at 25 cents ...	15.00
25 lb. hydrofluoric acid at 18 cents ...	4.50
Ice .....	0.50
	<hr/> 20.00

The method recommended by Thomsen consists in dissolving the barium dioxide in dilute nitric acid, whereby a large amount of the barium becomes precipitated as nitrate, from which barium peroxide can be recovered by ignition; the small quantity of barium remaining in solution is removed by addition of sulphuric acid. The barium peroxide may also be suspended in water and decomposed by a stream of carbonic acid, which produces a very pure solution of hydrogen peroxide, but too weak for industrial applications.—G. H. B.

*On the Preparation of Thiophen.* L. H. Friedburg. J. Amer. Chem. Soc. 12, 83—90.

THE author gives full details of the method of preparation of thiophen by Volhard and Erdmann's method (Ber. 18, 454) from sodium succinate and phosphorus trisulphide. The sodium succinate was prepared from succinic acid and sodium carbonate and dehydrated at 110°; the phosphorus trisulphide was got by heating the mixture of phosphorus and sulphur in a porcelain crucible, small portions being added at intervals after the first portion had been ignited.

To prepare the thiophen, 155 grms. of sodium succinate and 310 grms. of well crushed phosphorus trisulphide were distilled from a 3-litre tubulated retort, heated over the free flame. The distillate was redistilled from the water-bath, washed with sodium hydrate, dried over metallic sodium and redistilled. 150 grms. of pure thiophen were obtained from 610 grms. of sodium succinate and 1,200 grms. of phosphorus trisulphide. It was found advisable to prepare several lots of thiophen before subjecting them to the final purification, on account of the mechanical loss that takes place in the latter process.

The residue in the retort evolves sulphuretted hydrogen on the addition of water, and appears to contain either phosphorus acid or phosphites or both.—C. A. K.

*Is Saccharine Innocuous?* Chem. Zeit. Rep. 14, 189—207.

FAHLBERG, List and Co., publish from time to time the opinion of numerous authorities on this subject. One of the first medical men in Austria has stated that saccharine does not appear to be injurious either to healthy or to sick persons, even when administered in considerable quantities. Professors Javorsky and Rosenzweig have come to the conclusion that a daily dose of 100 grms. has no disturbing effect on the organs of sense, the nervous system, the circulation, or the respiratory or urinary organs. In the digestive organs any dose above 25 grms. per day causes irregularities. Larger doses produce diarrhoea, but less so than an equal weight of common salt would do. Acute diseases do not seem to be affected in any way by saccharine. It does not reduce the appetite. The saliva does not absorb it, but the urine contains almost the total quantity taken after about half an hour. The yearly consumption of the sodium salt of saccharine per head might be taken at 109—172 grms.; the action of this quantity on the functions of the body must be practically nil, and very small indeed on the digestive organs, much less so, at least, than the spices usually employed. In certain diseases, such as diabetes mellitus, obesity, cancer of the stomach, &c., the new sweetening agent is warmly recommended.—A. R.

*Report on Compounded Medicines, Gums, and Resins.*

Report of the Central Laboratory of the Italian Customs Department, 1886—1889, 352—356.

COMPOUNDED medicines are subject to a duty of 120 lire per quintal, and are defined in a note as follows: "Medicinal drugs mixed together or mixed with pulps, sugars, extracts, whether moist or dissolved with syrups, mucilages, spirits, &c., or when such mixtures are found in the form of powder or dried paste, or when they are soft or in the liquid state. Among compounded medicines shall likewise be reckoned artificial balsams and effervescent citrates of magnesia and lithia.

The following cases, among others, are specially cited: A liquid declared as "sweetened spirit" was found to contain besides alcohol and sugar, a considerable quantity of extract of quinine and quassia, coloured brown with caramel and flavoured with orange essence. It was classified under the title in the tariff report "of stomachic and pharmaceutical elixirs of every sort." A sample declared as chrome iron-stone was found to be nothing but reduced iron, which is specially mentioned in the report.

In the case of sulphonal a Ministerial decree decided (October 28, 1888) that this substance is to be classed with chloral hydrate under the category of "compounded medicines not specified" (with alcohol as a basis). Ichthyol and its preparations were classed by a decree (December 13, 1888) in the same category; similar decrees were issued regarding malt extract (Gesundheits-bier) and pepsin with hydrochloric acid (April 6, 1889).—V. H. V.

*Report on Perfumery.* Report of the Central Laboratory of the Italian Customs Department, 366—367.

IN the tariff report perfumery is classed as alcoholic and non-alcoholic, the former paying, besides the tax of 100 lire per quintal, the surcharge on the alcohol contained therein. Among substances specially mentioned are a sample, declared as common soap, but recognised as a perfumed fat containing sandstone, another as fatty perfumery, recognised as alcoholic perfumery, containing brilliantine.—V. H. V.

## PATENTS.

*Improvements in the Treatment of the Disulphones for Medicinal Purposes.* G. Lunan, Edinburgh. Eng. Pat. 18,434, November 19, 1889. 4d.

THE disulphones (sulphonal, trional, tetranal, &c.) are dissolved in chloroform, ether, &c., and the dissolved drug separated in an amorphous condition either by precipitation with water or by evaporating off the solvent. The drugs thus prepared, or whilst still in solution, are acted on by an oleaginous solvent such as olive oil, castor oil, hydrocarbon oil, or jellies, whereby they are obtained in a more soluble form and one in which they possess an improved physiological action. This may be done enclosed in a flexible gelatin capsule or other suitable material.—C. A. K.

*Improvements in the Manufacture of Tetrachloride of Carbon.* W. H. Lever, J. D. Lever, and E. J. Scott, Port Sunlight. Eng. Pat. 18,990, November 26, 1889. 6d.

DRY chlorine is passed slowly and in a fine stream into carbon bisulphide, in which is dissolved from 2—12 per cent. of its weight of iodine, the following reaction taking place,  $\text{CS}_2 + 3 \text{Cl}_2 = \text{CCl}_4 + \text{S}_2\text{Cl}_2$ , the iodine acting as a carrier of chlorine. When the original volume of the carbon bisulphide has increased threefold the product is slowly distilled, the tetrachloride coming over first and the sulphur chloride remaining behind. The former is refractionated, washed with alkali, and redistilled. The iodine is recovered from the alkaline washings. Bromine and pentachloride of antimony can be substituted for the iodine, but are less efficacious.—C. A. K.

## XXII.—EXPLOSIVES, MATCHES, Etc.

*Determinations of the Firing Points of Various Explosives.* C. E. Munroe. J. Amer. Chem. Soc. 12, 57—64.

For this purpose an apparatus, devised by Horsley, was used, which consisted of an iron stand with a ring support holding a hemispherical iron vessel, in which paraffin or tin was put. Above this was another movable support, from which a thermometer was suspended and so adjusted that its bulb was immersed in molten material in the iron vessel. A thin copper cartridge case,  $\frac{3}{4}$  in. in diameter and  $1\frac{1}{8}$  in. long, was suspended over the bath by means of a triangle so that the end of the case was 1 in. below the surface of the liquid. On beginning the experiment, the material in the bath was heated to just above the melting point, the thermometer was inserted in it, and a minute quantity of the explosive was placed in the bottom of the cartridge case. The temperature marked by the thermometer was noted as the *initial temperature*, the cartridge case containing the explosive was inserted in the bath, and the temperature quickly raised until the explosive flashed off or exploded, when the temperature marked by the thermometer was again noted as the *firing point*. The tables given show the results of about six experiments with each explosive. The initial temperatures range from 65 to 280 °C. in some cases, but as the firing points remain fairly constant, only the extremes of the latter are quoted in the following table:—

Description of Explosive.	Firing Point in ° C.
Compressed military gun-cotton.....	186—201
Air-dried military gun-cotton.....	179—186
" " " .....	186—189
" " " .....	197—199
" " " .....	154—161
Gun-cotton dried at 65° C.....	196—191
Air-dried collodion gun-cotton.....	186—191
" " " .....	197—199
" " " .....	193—195
Air-dried gun-cotton.....	192—197
" " " .....	194—199
Hydro-nitrocellulose.....	201—213
Nitroglycerin.....	203—205
Kieselghur dynamite, No. 1.....	197—200
Explosive gelatin.....	203—209
Explosive gelatin, camphorated.....	174—182
Mercury fulminate.....	175—181
Gunpowder.....	278—287
Hill's picric powder.....	273—283
" " " .....	273—290
Forcite, No. 1.....	184—200
Atlas powder, 75 per cent.....	175—185
Emmensite, No. 1.....	167—184
Emmensite, No. 2.....	165—177
Emmensite, No. 5.....	205—217

—G. H. B.



## PATENTS.

*Improvements in or connected with Wax Matches.* P. Molyneux, London. Eng. Pat. 16,028, October 11, 1889. 4d.

THE material for the stems of the matches is made by forcing a mass of some fibrous pulp, such as Swedish wood pulp and paraffin mixed with a small quantity of stearin, and rendered plastic by heat, through a jet or orifice, whence it issues in the form of a long thin rod, which becomes hard on cooling, and is afterwards cut into suitable lengths and dipped in the usual way. Or the wood pulp may be rendered plastic by mixing it with one of the lighter petroleum spirits, and formed into a rod as before, and then passed through a bath of melted paraffin and stearin.—W. M.

*Improvements in Plant for Burning Special Charcoal for the Manufacture of Gunpowder.* J. S. Taylor and S. W. Challen, Birmingham. Eng. Pat. 16,426, October 18, 1889. 8d.

THE improvements consist in constructing the revolving drum, which contains the wood to be carbonised, and the lower part of which is in a furnace, with hollow trunnions, or with a hollow shaft which passes right through the centre of the drum, and which is perforated. The gases which are given off during the carbonisation of the wood escape by the hollow trunnions or shaft, and are led into the furnace, where they are consumed, and thus effect a saving in the main supply of fuel. The shaft is attached to the cylinder or drum in such a way that when the latter burns out it can easily be removed and a new one substituted for it.—W. M.

*Improvements in Plant for Preparing Gunpowder Carbon, consisting in the Application of Gaseous or Liquid Fuel to Heat the Carbonising Vessel.* J. S. Taylor and S. W. Challen, Birmingham. Eng. Pat. 16,629, October 22, 1889. 8d.

IN this patent "any ordinary well-known system of furnace suitable for burning liquid or gaseous fuel" is employed in conjunction with the patentee's improved method of supplementing the fuel by conducting the gases evolved from the material during its carbonisation (see Eng. Pat. 16,426, preceding abstract), into the fire chamber. Drawings accompany the specification.—W. M.

*An Apparatus for Nitrifying Cotton, Cellulose, Straw, and similar Material.* E. Müller, Cologne, Germany. Eng. Pat. 8843, June 7, 1890. 6d.

THE material is treated with acid in a vessel provided with a perforated false bottom. This vessel is kept cool by a water-jacket. A pipe leads from below the perforated bottom to an acid reservoir, which is in connexion with a vacuum pump. When the process of nitration is ended, communication is established between the vacuum pump, reservoir, and nitrating vessel; the acid is thus all removed from the cellulose, &c. by suction, and drawn into the reservoir, from whence it can be allowed to flow back into the nitrating vessel, after the material has been removed from it, for another operation.—W. M.

*Improvements in or appertaining to Explosive Compounds.* W. P. Thompson, Liverpool. From S. D. Smolianinoff, Washington, U.S.A. Eng. Pat. 11,018, July 15, 1890. 4d.

THE object of this invention is to render nitroglycerin insensitive to concussion.

AN alcohol, preferably methyl alcohol, is added to nitroglycerin in the proportion of one volume of alcohol to three volumes of nitroglycerin. The union is facilitated by slightly heating the nitroglycerin. Or one volume of "acetic ether of glycerin" is added to three volumes of nitroglycerin. The resulting compound may be mixed with the usual absorbents, and can be exploded by a fulminate.—W. M.

*Improved Vacuum Drying Apparatus for Explosive Substances.* H. J. Haddan, London. From J. H. Voelkel, Breslau, Germany. Eng. Pat. 11,745, July 26, 1890. 11d.

ACCORDING to this invention the explosive material is dried in vacuo. The apparatus consists essentially of a drying chamber and an expansion chamber which are in communication and from which the air is more or less exhausted. The drying chamber is provided with air-tight doors opening outwards, so that in the event of an explosion generating more gas than will fill the exhausted space, the pressure is at once relieved by these doors.

The apparatus is fully illustrated by drawings.—W. M.

## XXIII.—ANALYTICAL CHEMISTRY.

## APPARATUS.

*On the Determination of the Viscosity of Lubricating Oils.* A. Martens. Mitt. Königl. techn. Versuchs. 1890, 143—151.

See under XII., page 1042.

INORGANIC CHEMISTRY.—  
QUANTITATIVE.

*Determination of Alumina for Technical Purposes.* M. Kretzschmar. Chem. Zeit. 14, 1223.

THE determination of alumina by the usual gravimetric methods is wasteful of time on account of the tedious washing requisite. Attempts to use the volumetric process consisting in precipitating the alumina in acetic solution with a known quantity of sodium phosphate and titrating the excess of phosphoric acid by the ordinary uranium method, have not met with much success owing to the uncertainty of the point when excess of uranium is indicated by its reaction with potassium ferrocyanide, the perceptible solubility of alumina in ammonium salts, and the delay occasioned by the need for repeated titrations, which renders the rapidity of the process somewhat illusory.

By avoiding the use of ammonium salts altogether, adding sodium phosphate at once in excess and in the cold, and standardising the phosphate solution by means of an aluminium salt of known composition, the process becomes trustworthy.

175 grms. of uranium acetate (not nitrate) are dissolved and made up to five litres, and the exact strength of the solution determined by titration against sodium ammonium phosphate (14.718 grms. per litre), the purity of which has been ascertained by analysis; it is then diluted so that 1 cc. corresponds to about 0.004 grm. of  $P_2O_5$ . 10 grms. of pure potash alum, free from iron, are dissolved in water, sodium acetate and acetic acid added in excess, and the whole made up to 500 cc.; 50 cc. are taken, 10 cc. sodium acetate solution added, a known quantity of standard sodium phosphate solution added in excess, the mixture brought to the boil, and titrated with uranium solution. The titration is performed several times until the quantity requisite is so nearly known that the end point can be determined by the addition of only a few tenths of a cubic centimetre of the solution.

THE standard solution of alum cannot be taken as correct, as even the best and smallest crystals contain excess of water, but its exact content of alumina must be determined as follows: 50 cc. are precipitated by sodium phosphate in excess, followed by sodium acetate, the precipitate being then redissolved by hydrochloric acid. The solution is heated and reprecipitated by sodium thiosulphate in great excess, and boiled until it will settle, leaving the liquid clear; prolonged boiling is to be avoided. It is filtered,

washed with hot water, dried, ignited with free access of air, and weighed as aluminium phosphate. Its weight multiplied by 0.4204 gives that of the alumina present.

In order to avoid the tiresome repetitions of the titration with uranium acetate which are usually necessary, the quantity of alumina present in any given sample to be analysed may be approximately determined by adding to its solution a very little methyl-orange and titrating with caustic potash until the red colour disappears, showing that the free acid has been neutralised. Phenolphthalein is then added and the titration continued until a red colouration is produced; the difference between the first and second titrations gives the quantity of potash corresponding with the alumina present. The determination by means of the phosphate process described above can then be carried out in the ordinary way, one titration sufficing. As in analysis of most aluminium salts the free acid must be determined, no additional consumption of time results from the use of this plan. The application of the method to practical requirements is generally easy. In the case of an ammonia alum, caustic soda should be added and the bulk of the ammonia boiled off before titration. The analysis of an impure ferruginous sample of aluminium sulphate may be taken as an example of the working of the process: 5 grms. are dissolved and made up to 500 cc., the free acid determined by titrating 50 cc. with caustic potash, using methyl-orange as an indicator, and the approximate quantity of alumina by continuing the titration after the addition of phenolphthalein. Another portion of 50 cc. is heated with a few drops of hydrochloric acid containing bromine, sodium acetate added in excess, then a known quantity of sodium phosphate in excess, and the approximate quantity of alumina being known, the exact quantity of unprecipitated phosphate determined by a single titration with uranium acetate.

The total quantity of iron may be determined in another 50 cc. by reduction with zinc and titration with potassium permanganate, and the alumina corresponding to it calculated and deducted from the result of the uranium titration, giving the real percentage of alumina present.

The proportion of ferrous iron may be determined by direct titration with permanganate.

In the case of aluminium sulphate it is oxidised to sulphate by means of bromine and dealt with in the same way.—B. B.

#### *Estimation of Available Phosphoric Acid in Fertilisers containing Cotton-Seed Meal.* F. B. Daney. Pamphlet, 1890.

By dissolving fertilisers containing cotton-seed meal in concentrated nitric acid with a little hydrochloric acid, as recommended by the Association of Official Agricultural Chemists (United States) in one of their alternative methods for analysing phosphates, or in hydrochloric acid and potassium chlorate, a solution is obtained from which the phosphoric acid is not wholly precipitated by molybdic solution, therefore methods entailing these modes of treatment yield low results, which are more marked in new than in old samples of cotton-seed meal. The author has investigated this subject and finds that fusion with a mixture of equal parts of sodium carbonate and potassium nitrate, and probably also incineration and solution in hydrochloric acid are reliable modes of dealing with the fertilisers in question. In course of this investigation it was noticed that nitric acid is not an adequate solvent for incinerated phosphate.—D. A. L.

#### *Bénger and Stingl's Method of Softening Water.* W. Kalmann. Mitt. des k.k. tech. Gew.-Museums, 1890, 10—15.

To explain Bénger and Stingl's method, the author finds it convenient to divide the hardening substances into three classes, of which the first class comprises all lime salts except the bicarbonate, the second the bicarbonates of lime and magnesia, and the third all magnesium salts other than the bicarbonate. The members of the first class are

precipitated with sodium carbonate, 1 mol. of calcium oxide requiring 1 mol. of sodium carbonate, those of the second class by calcium or sodium hydrate, separately or together, 1 mol. of calcium carbonate requiring 1 mol. of calcium hydrate or 2 mols. of sodium hydrate, whilst 1 mol. of magnesium carbonate requires 2 mols. of calcium hydrate or 4 mols. of sodium hydrate since magnesium carbonate is soluble in saline water, and it is therefore necessary to precipitate the magnesium in the form of hydrate, which is much less soluble. The members of the third class are removed with sodium hydrate.

When a water contains compounds of both the first and second classes and sodium hydrate is used as the precipitant, an equivalent amount of sodium carbonate is produced, which in turn reacts with the salts of the first group, so that in this case a correspondingly less amount of sodium carbonate requires to be added. In fact, the relative amounts of sodium hydrate and carbonate required depend on the relative amounts of the compounds of these two classes which are present, and the precise mode of treatment will vary according as—

(Case 1.) The quantity of sodium carbonate required for precipitation is less than that formed in the decomposition of the salts of the second class. In this case the excess of sodium hydrate may with advantage be replaced by lime, a mixture of sodium and calcium hydrates being used.

(Case 2.) The quantity of sodium carbonate formed in the decomposition of the salts of the second class exactly suffices to precipitate the salts of the first class. In this case sodium hydrate alone is employed.

(Case 3.) More sodium carbonate is required than is formed from the sodium hydrate in the course of the precipitation; a mixture of sodium hydrate and carbonate must therefore be used.

In order to ascertain the quantities of softening reagents which are to be added to the water, the following points must be ascertained: 1. The quantity of combined carbon dioxide contained in the water, which may be readily estimated with standard acid and methyl-orange. 2. The quantity of calcium oxide contained in the water. 3. The total hardness.

Then, suppose  $a$  represents the amount of sodium carbonate equivalent to the combined carbon dioxide present, and that  $x$  represents the amount of sodium carbonate equivalent to the calcium carbonate, and  $y$  that equivalent to the magnesium carbonate, we have—

$$x + y = a \dots \dots \dots (1).$$

And if  $b$  represents the amount of sodium carbonate equivalent to the hardness due to lime salts,  $x$  parts of  $b$ , being equivalent to the calcium carbonate, and  $y$  parts of the same to other calcium salts, we have—

$$x + z = b \dots \dots \dots (2).$$

Taking  $c$  to denote the total hardness of the water, we have  $x$ ,  $y$ , and  $z$  equivalents of sodium carbonate required for the removal of the lime salts and magnesium carbonate and representing by  $r$  the soda equivalent to the magnesium salts other than the carbonate, we have—

$$x + y + z + r = c \dots \dots \dots (3).$$

The soda represented by  $z$  and  $r$  must be causticised, completely or partially, according to circumstances.

From equations (1) and (3) it follows that—

$$z + r = c - a \dots \dots \dots (4).$$

In precipitating the carbonates with sodium hydrate  $x + 2y$  parts by weight of sodium carbonate are formed, and of these,  $z$  parts are required for the precipitation of the lime salts of the above-mentioned first class. The difference  $x + 2y - z$  represents the amount or equivalent of sodium carbonate required in any of the above-cited three cases. In the first case  $x + 2y$  is less than, in the second equal to, and in the third greater than  $z$ . Now, from equations (1) and (2)—

$$x + 2y - z = 2a - b \dots \dots (5).$$

With  $2a - b = +m$  (first case),  $c - a$  parts of soda (equation 4) must be causticised for use and  $m$  parts of soda may be replaced by their equivalent of calcium hydrate.

With  $2a - b = 0$  (second case),  $c - a$  parts by weight of soda must be used in the form of hydrate, and with  $2a - b = -n$  (third case),  $c - a - n$  parts by weight of soda must be causticised and  $n$  parts used uncausticised.

The author, further, gives a volumetric method of estimating the quantity of lime contained in water. The principle of the method consists in precipitating the lime with an excess of standard solution of sodium oxalate, filtering, and determining the excess with standard potassium permanganate. 100 cc. of the water to be tested are placed in a 250 cc. flask, a few cc. of ammonium chloride solution added, then a slight excess of standard solution of sodium oxalate (0.3 gm. of crystallised salt per litre) and the flask filled to the mark. After allowing the contents to stand 2–3 hours, 100 cc. are withdrawn, filtered, and titrated with potassium permanganate, the amount of lime being estimated by the difference between the oxalate added and that found. Allowance must be made for the permanganate decolourised by the organic matters originally present in the water. Should other oxides precipitable by oxalic acid be present the ultimate result will not be affected, as such substances will require removal in the process of softening the water. The author adds that there are some waters which must be completely analysed before any method of purification can be adopted for them, but that chemists will quickly discover when they have such exceptional cases to deal with.—E. B.

#### *Determination of Lithium in Mineral Waters.* E. Waller. J. Amer. Chem. Soc. **12**, 214–223.

THERE are three methods in general use for the determination of lithium, viz., (a) Mayer's modification of the phosphate method (Ann. **98**, 193); (b) the amyl alcohol method (Gooch, Amer. Chem. Jour. **9**, 33); (c) the fluoride method (Carnot, Bull. Soc. Chim. [3], **1**, 280).

For all these processes it is necessary to obtain from a known quantity of the water the alkalis as chlorides free from admixture with other bases, and in most cases a large proportion of the sodium and potassium salts, which usually predominate largely over those of lithium, must be removed. The usual method is to acidify with hydrochloric acid, to evaporate, treat with barium hydrate, remove excess of baryta by ammonium carbonate, drive off the ammonium salts, and then to extract with alcohol to remove lithium chloride, which is inevitably accompanied by some sodium and potassium chlorides.

The spectroscope is used to determine when the washing is complete. Barium precipitates are very apt to mechanically take down lithium compounds with them, and it seems better to work with rather dilute solutions.

Then, in carrying out the phosphate method, an excess of hydrogen disodium phosphate and a moderate excess of sodium hydrate are added, the whole is evaporated to dryness and the residue is taken up with warm water; strong ammonia is next added and the mixture is warmed and allowed to stand for 12 hours. The precipitate is ignited and weighed as  $\text{Li}_3\text{PO}_4$ . Further quantities may be obtained from the filtrate. The whole of the lithium can scarcely be obtained by this method.

Gooch takes the concentrated solution of the alkaline chlorides (which should contain only about 0.2 gm. of salts in all), and adds 30–50 cc. of pure anhydrous amyl alcohol. The whole is then heated for some time carefully on a sand-bath, a few drops of hydrochloric acid are added, the heating repeated, and the alcohol then filtered into a measuring cylinder and its volume noted (usually 10–15 cc.). The undissolved salts are washed with cold amyl alcohol until no trace of lithium is perceptible in them by the spectroscope; the filtrate and washings are evaporated in a weighed platinum dish, and the chlorides converted into sulphates, ignited and weighed. From this weight, for every 10 cc. of

amyl alcohol which remained in contact with the chlorides after heating, the following deduction is made:—

	Gm.
If only sodium and lithium chlorides present .....	0.0005
" potassium " " " .....	0.00059
If sodium, potassium, and lithium chlorides present .....	0.00109

In Carnot's method the mixed alkaline chlorides after being taken nearly to dryness are extracted with a mixture of about equal volumes of 90 per cent. alcohol and ether so as to obtain the lithium chloride comparatively free from the others. After evaporating off the alcohol and ether, the salts are dissolved in the least possible quantity of water and filtered into a weighed platinum dish. Pure ammonium fluoride and ammonia are added to the concentrated filtrate, and the whole is allowed to stand 12 hours. The fluoride is collected and converted into the sulphate and weighed as such. 0.004 gm. is added for every 7 cc. of filtrate and washings (ammonium fluoride and ammonia must be used for washing the fluoride). The process is good, but tends to yield results slightly high.

An examination of various American natural lithia waters was made by the fluoride method with the result that some waters were found to contain mere traces of lithium and others only very small quantities.—T. L. B.

#### *A New Method for Determining the Value of Bleaching Powder.* L. Vanins. Zeits. f. angew. Chem. 1890, 509–510.

A WEIGHED quantity of bleaching powder is triturated with water and placed in a flask; sufficient of a solution of hydrogen dioxide is added to decompose the bleaching powder, and the oxygen which is evolved collected in a large glass jar. The volume of the water displaced is measured, and this gives the volume of the oxygen liberated, the weight of which may be found in the usual way. The results are accurate to 0.5 per cent.—A. L. S.

#### *Electrolytic Determination of Nitric Acid.* G. Vortmann. Ber. **23**, 2798–2801.

THE author has ascertained the conditions under which the nitric acid of nitrates may conveniently be reduced electrolytically to ammonia.

On passing a galvanic current through dilute nitric acid, or through a solution of the nitrate of an alkali, acidified with sulphuric acid, no ammonia is formed; but, if there be present a metallic salt whose acidified solution is decomposed by the current with deposition of the metal, then reduction of the nitric acid to ammonia takes place. A copper salt is found to work best, but complete reduction takes place also in presence of platinum or mercury salts. When zinc salts are used, complete reduction is not certain. The determination of the nitric acid is carried out as follows:—To the solution of the nitrate in a platinum dish is added a sufficiency of pure copper sulphate; the whole is acidified with sulphuric acid and electrolysed. After all the copper has been deposited, the solution is evaporated to small bulk and the ammonia is distilled as usual. It is convenient to use a current liberating 1–2 cc. of electrolytic gas per minute. The amount of copper salt to be used depends on the quantity of nitric acid present; in determining the nitric acid in potassium nitrate it is necessary to take half as much crystallised copper sulphate as potassium nitrate. A smaller amount may be used, but in such a case the copper must be more slowly deposited.

The following experiment illustrates a modification of the method which may be used in cases where a neutral nitrate is under consideration, or where other salts are present, which undergo no permanent decomposition: potassium nitrate is dissolved in water and to the solution is added a measured quantity of sulphuric acid of known strength. The positive electrode is coppered, and the current is now passed until all the copper has been deposited on the negative electrode. The excess of sulphuric acid is then

determined by titration. It is to be noted in this particular instance that part of the titrated acid is taken up by the potassium, thus—



—T. L. B.

*Volumetric Determination of Manganese.* G. Vortmann.  
Ber. 23, 2801—2803.

It is found that in presence of a salt of a sesquioxide (of aluminium or iron) manganous salts may be completely oxidised by iodine in presence of an alkali. Hence the following method serves for estimation of manganese:—The weighed salt of manganese is dissolved in water together with two to three times its weight of potash alum; a measured quantity of decinormal iodine solution is added, and some pure caustic soda solution. The whole is warmed for 5–10 minutes on a water-bath, allowed to cool, and then made up to known volume. An aliquot part is filtered through a dry fluted filter and acidified, and the excess of iodine is determined with sodium thiosulphate solution. Two atomic weights of iodine correspond to one atomic weight of manganese.—T. L. B.

*Electrolytic Separations.* E. F. Smith and L. K. Frankel.  
Amer. Chem. J. 12, 428—435.

It has already been stated by the authors (Amer. Chem. J. 12, 212) that the deposition of palladium from the solution of its double cyanide was not possible so long as any undecomposed potassium cyanide remained in the solution, and acting upon this they have now attempted the separation of palladium from the metals which are deposited from their double cyanide solutions. (See also this Journal, 1889, 639; and 1890, 108, 656, and 808—809.)

*Mercury from Palladium.*—Solutions of mercuric chloride and of palladium chloride were used in these experiments, potassium cyanide being added so that the precipitate first formed completely dissolved. The potassium cyanide was as a rule in large excess to prevent any danger of precipitating the cyanides from the solution. The current used varied from 0.08 to 0.22 cc. of OH gas per minute, and the results from -0.66 to +0.85 per cent. of the mercury actually present in the original solution.

*Silver and Cadmium* being metals like mercury and separating quite readily from cyanide solutions when acted upon by a feeble current, it was expected that they could be separated from palladium without any particular difficulty, but out of a series of 10 experiments made with varying quantities of alkaline cyanide and with currents varying from 0.5 cc. to 0.1 cc. of oxyhydrogen gas per minute, neither silver nor cadmium could be obtained free from palladium. In fact, the presence of these two metals appeared to favour the deposition of the palladium from the solution.

*Mercury from Arsenic.*—It was found that arsenic could not be deposited from solution of alkaline arsenites or arsenates, or from solutions containing alkaline cyanides. In the separation of mercury from arsenic, the precipitate formed on adding a solution of mercuric chloride to a solution either of potassium arsenite or arsenate was dissolved in potassium cyanide and subjected to the action of the current.

The separation proceeded without any difficulty, the amount of mercury found varied from -0.66 to +0.48 per cent. from the theoretical.

*Cadmium from Arsenic.*—The separation was not complete unless the arsenic existed in the solution as the higher oxide. In experiments with arsenious compounds, deposits of cadmium were obtained, which were invariably dotted over its surface with black spots of metallic arsenic. With the arsenic present as arsenate, the cadmium obtained varied from -0.93 to +0.19.

*Silver from Arsenic.*—The conditions essential for complete deposition are similar to the preceding. Attempts to separate the two metals from solutions in which the arsenic

existed as lower oxide resulted in the deposition of the arsenic with the silver. Using arseniate solutions, the results obtained varied from -0.31 to +0.63 per cent. of silver.

*Copper from Arsenic.*—The conditions are the same as in the preceding case, except that increased strength of current is necessary for the deposition of the copper. The amount of copper varied from -0.56 to +0.33 per cent. The potassium cyanide should be carefully added until the precipitate formed dissolves and the solution assumes a pink tinge. An excess of potassium cyanide is not harmful, but the greater the excess the longer will be the time required for the deposition, since the current will not deposit the copper till the excess is decomposed. Copper may be separated from arsenic in a solution containing an excess of ammonia, but the method requires skill and close attention to details for its successful performance, the copper from such a solution being deposited in a spongy coating, having a tendency to carry down and retain the salts of the alkalis which may be in the solution. In three experiments in which 0.1772 grm. of copper was present, the amount of deposit obtained varied from 0.1754 to 0.1797.

The authors having found that the oxides of tungsten and molybdenum dissolve readily in potassium cyanide, and that such solutions or solutions of ammonium tungstate or molybdate in the presence of potassium cyanide give no deposit of metal, even when electrolysed by strong currents, have based the following separations upon them.

*Mercury from Tungsten.*—For this separation, weighed quantities of mercuric chloride and ammonium tungstate were dissolved in water, the precipitate of mercury tungstate dissolved in potassium cyanide, and the resulting solution electrolysed. The amount of mercury varied from +0.09 to +0.70 per cent. The current should not exceed 0.8 cc. of OH gas.

*Silver from Tungsten.*—The conditions are the same as in the preceding case. The results varied from -0.38 to +0.95 per cent. of silver.

*Cadmium from Tungsten.*—In this separation the strength of the current is extremely important. If the current exceed 0.6 cc. of gas per minute, the deposits are apt to become spongy, and the results correspondingly high. The results usually vary from -0.38 to +0.95 per cent. of cadmium.

*Mercury from Molybdenum.*—The method was carried out as in the separation of mercury from tungsten. The results varied from +0.22 to +0.79 per cent.

*Silver from Molybdenum.*—The silver was added as nitrate, the conditions being otherwise similar to those in the preceding separation. The results varied from +0.06 to +0.70 per cent. of silver.

*Cadmium from Molybdenum.*—In these experiments the tendency of the cadmium to form spongy deposits was not so noticeable. In most cases the coating was dense and compact, and hence permitted thorough washing. The results varied from -0.66 to +0.91 per cent. of cadmium.

*Copper from Bismuth.*—Up to the present no successful method has been proposed for the electrolytic separation of these two metals. The authors have found that by adding a solution of copper sulphate to a solution of bismuth citrate containing an excess of alkali, afterwards adding a slight excess of potassium cyanide, bismuth can be deposited without carrying down any of the copper. It is necessary for complete deposition that sufficient citrate and alkali be present to keep the solution clear on the addition of the potassium cyanide. The results vary from -0.63 to +0.63. The above conditions must be carefully complied with.

In all the above separations, the deposited metal was washed with boiling water, except in the case of bismuth, where washing with cold water and absolute alcohol proved decidedly advantageous. The drying was done upon a moderately warm iron plate. The current in all cases was derived from Daniell cells of the crowfoot pattern, and the current was passed for 16 hours.—O. H.

## ORGANIC CHEMISTRY.—QUALITATIVE.

*Examination of Acetanilide.* E. Ritsert. Pharm. Zeit. 1890, **35**, 306.

MANY statements have been made on this subject, but the author is of opinion that all these are insufficient to decide whether the sample of acetanilide tested is pure or not. He has ascertained that absolutely pure acetanilide shows the following properties and reactions:—(1.) The melting point of pure acetanilide, dried at 105° C., is exactly 114° C., not 112° or 113° C., as hitherto assumed. (2.) 0.1 gm. shaken with 1 cc. of concentrated hydrochloric acid dissolves to a clear solution, which after a few moments precipitates acetanilide hydrochloride (exalgin, if present, is not precipitated). No colouration appears when a drop of nitric acid is added, whilst the presence of oxyethyl- and oxymethylacetanilide would after some time produce a yellow or brown colouration. (3.) 0.1 gm. boiled several times with 2 cc. of concentrated hydrochloric acid, and mixed, when cold, with a little chlorine water, gives a beautifully blue colouration, which, however, soon disappears. (4.) An aqueous solution of pure acetanilide shows no acid reaction (no acetic acid present), and if boiled and some drops of ferric chloride are added, a dark red-brown colouration appears. The addition of a mineral acid, however, causes this colouration to disappear. (5.) If 1 gm. of pure acetanilide be boiled with 30 grms. of water, and a drop of an aqueous solution of potassium permanganate (0.1 per cent.) be added, a pink colouration appears, which lasts for about five minutes, and does not change to yellow or yield precipitates, if the solution be boiled again (the latter takes place, if acetoluidines, free aniline, resinous products, or other impurities are present). (6.) No residue is left when heated on platinum foil (compare this Journal, 1890, 329 and 555).—H. S.

*On the Qualitative Determination of Mechanical Wood Pulp in Paper.* W. Herzberg. Mitt. König. tech. Versuchs. 1890, 132—140.

THE author shows that chemical tests are not in all cases sufficient to distinguish mechanical wood pulp from other substances which may be used in the manufacture of paper, and recommends a microscopical examination in addition to the chemical one. For this purpose portions of the paper are boiled in a dilute solution of caustic soda, then washed with water in a fine sieve, and then disintegrated by shaking with garnets; the liquid for the microscopical objects is an aqueous solution of iodine which is well known to give a yellow colour to all cells containing lignified substances. (Compare this Journal, 1890, 112.) This alone is not sufficient, but the anatomical distinctions of the different fibrous substances are so characteristic that in all cases mistakes are entirely excluded. The colouration of mechanical wood pulp under the microscope is light or dark yellow, according to the thickness of the substance. Isolated cells are not found entire, the result of the grinding process being irregular bundles or particles of cells; the ends of the cells are very seldom found. Very characteristic are the pitted vessels, the concentric circles of which are generally plainly discernible. Also the cells of medullary rays are very characteristic of mechanical wood-pulp. Contrary to these properties, imperfectly boiled wood cellulose is of a lighter colour, and on account of the chemical treatment, only isolated cells or parts of them are found microscopically. Unseparated bundles or pieces of cells are never found, whilst these, as we have seen, are the characteristic features of mechanical wood-pulp. The ends of the cells are always found in great numbers. On account of the chemical treatment, the structure of the cells is much less discernible, especially the concentric circles of pitted vessels which appear only as light spots of circular or elliptic shape. The cells of medullary rays which are, as we have seen, highly characteristic of mechanical wood pulp, are entirely absent in imperfectly boiled wood cellulose. From all these statements it is evident that the differences between these two materials are so great and characteristic that errors in testing are entirely out of the question.—H. S.

## ORGANIC CHEMISTRY.—QUANTITATIVE.

*Lubricating Oils.* A. C. J. Charlier. The Engineer, 1890, 305—306.

See under XII., page 1043.

*Determination of Morphine in Opium.* G. Looff. Apoth. Zeit. 1890, **5**, 271.

1. *Opium Powder.*—5 grms. of fine opium powder were well mixed with water, diluted to 75 grms., and after having been shaken several times, allowed to stand for about two hours and then filtered. To 60.8 grms. of the filtrate (containing 4 grms. of opium) 0.2 gm. of oxalic acid were added, this mixture was well shaken and allowed to stand for half an hour. Then 5.2 grms. of potassium carbonate solution (1:2) were mixed with it without shaking, and the solution quickly filtered through a dry folded filter before any morphine was precipitated. 16.5 grms. of the filtrate (containing 1 gm. of opium) were mixed in a small Erlenmeyer flask with 5 grms. of pure ether, the flask stoppered and vigorously shaken for 10 minutes. Then the ether was evaporated at ordinary temperature, the residue filtered, the precipitate thoroughly washed with water, saturated with ether, and dried at 40—50° C. The morphine thus obtained was removed from the filter by means of a small brush, and dried until the weight remained constant.

2. *Opium Solution.*—To 50 grms. 0.2 gm. of oxalic acid were added, and the solution evaporated to the consistency of treacle, diluted by slowly adding water to 70 grms., 5 grms. of potassium carbonate solution (1:2) added, filtered and 15 grms. of the filtrate further treated as above.

3. *Opium Extract.*—2.5 grms. were dissolved, 0.2 gm. of oxalic acid added, diluted to 70 grms., and further treated as No. 2.

E. Schmidt states that this method gives very exact results, the maximum differences of various tests having been not more than 0.3 per cent. It gives about 1 per cent. of morphine less than Dieterich's method, as more liquid is used in the described test, and as the morphine obtained is purer.—H. S.

*Determination of Tartar, Tartaric Acid, and Malic Acid in Wines, and some Remarks on the Inorganic Salts contained in Wines.* M. Schneider. Mitt. a. d. Pharm. Inst. d. Univers. Erlangen, 1890, **3**, 57; Chem. Zeit. Rep. **14**, 174.

THE method proposed by the "Reichsgesundheitsamt" (Chem. Zeit. **8**, 965) for the determination of tartar and tartaric acid is a good one, but it gives somewhat too high results, as the greater part of the acid salts contained in wines is by this method precipitated and titrated together with the tartar. It is therefore necessary for the exact determination of tartar and tartaric acid, to dissolve the precipitated tartar in boiling water, to neutralise with decinormal alkali, evaporate, incinerate, and to determine tartar and tartaric acid from the ash (carbon dioxide determination). It has not been possible to determine malic acid in wines by the methods employed hitherto, as part of this acid was always decomposed. The author recommends that the wine be neutralised by decinormal alkali, evaporated, incinerated, and the tarter determined as above. From this calculation, which is of course too high, the actual amount of tartar and tartaric acid (calculated as tartar) is subtracted, and the difference corresponds to the quantity of malic acid, and is calculated accordingly.

The following substances have been found in wines by the author:—Free tartaric and malic acids, tartar, acid potassium phosphate, sodium chloride, calcium sulphate and acid calcium phosphate, magnesium phosphate and ferric phosphate.

For making the above tests the author recommends the following process:—100 cc. of wine are evaporated to a syrup, treated with alcohol and the mixture allowed to stand

in a cool place for about 12 hours, before dissolving the tartar in boiling water. The incineration has to be effected very carefully.—H. S.

*Determination of Caffeine in Tea.* F. Vité. Mitt. a. d. Pharm. Inst. d. Univers. Erlangen, 1890, **3**, 113; Chem. Zeit. Rep. **14**, 174—175.

THE author considers Hilger's method to be the best, and applies it as follows:—5 grms. of tea are boiled for three consecutive times for one hour, each time in 300 cc. of water. The extract obtained is evaporated to one-fourth of its volume, and, while hot, mixed with freshly precipitated lead hydroxide and quartz sand; then evaporated on the water-bath, and extracted with chloroform for three hours in Soxhlet's or Flückiger's apparatus. Then the chloroform is removed by distillation, the residue dissolved in hot water, filtered, the filtrate evaporated on the water-bath, and the dry residue weighed.—H. S.

*A New Method for the Determination of Theine in Tea.* G. L. Spencer. Jour. Anal. Chem. 1890, **4**, 158.

To two or three grms. of finely powdered tea, 3 or 4 cc. water are added, the mixture boiled, allowed to stand, and then decanted. This operation is repeated seven times, the residue washed, and about 8 cc. of basic lead acetate added to the solution, which is then filtered, washed with hot water, and freed from lead with sulphuretted hydrogen; 5 grms. of hydrate of magnesia or of lime are added, the filtrate concentrated to about 50 cc., filtered, the residue washed with hot water, and the filtrate, when cold, seven times extracted with chloroform. Then the chloroform is removed

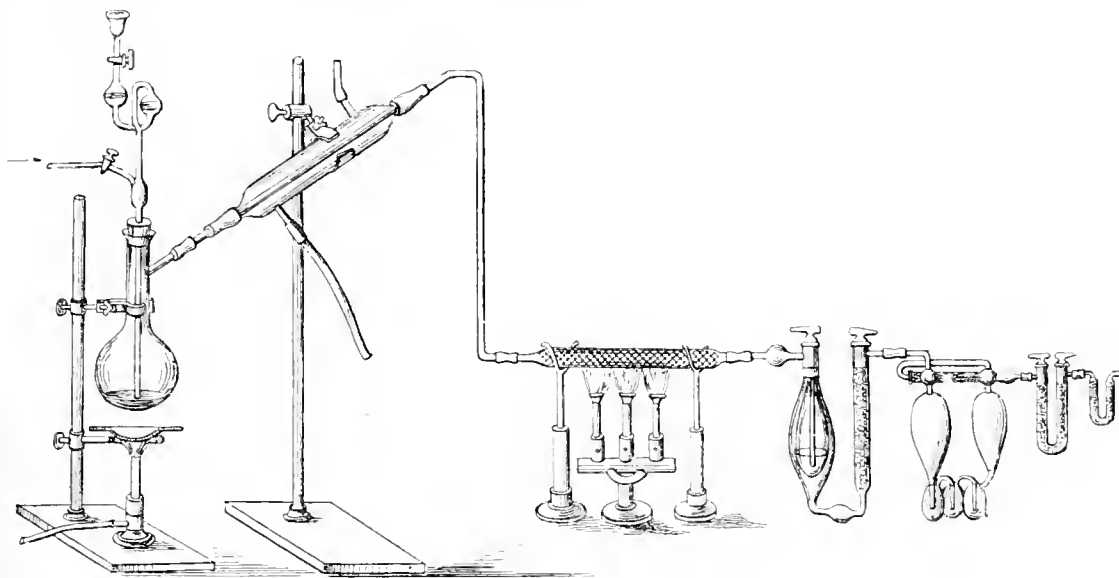
by distillation, the theine thus obtained dried at 75 °C., and weighed. The theine prepared in this way is very pure. (Compare this Journal, 1886, 178.)—H. S.

*The Oxidation of the Sulphur contained in Organic Bodies.* Berthelot, André, and Matignon. Compt. Rend. **111**, 6—9.

THE various methods in use for the oxidation of organic sulphur into sulphuric acid are tedious and difficult. The process described by the authors is not only a very accurate, but also a very rapid one, and is applicable to all organic sulphur compounds. It consists in burning the substance with oxygen under a pressure of 25 atmospheres in a calorimetric bomb, in presence of 10 cc. of water. The combustion takes place instantaneously, dilute sulphuric acid only being produced, provided the substance taken contained sufficient hydrogen. Bodies poor in hydrogen lead to the production of a little sulphurous acid. In such cases the substance must be mixed with from one-half to its own weight of camphor prior to combustion. Finally, the contents of the bomb are washed into a beaker, and the sulphuric acid is precipitated by barium chloride in the usual way. The test analyses, which include the estimation of sulphur in such bodies as albumin, taurine, carbon disulphide, &c., are exceedingly satisfactory.—H. T. P.

*Wet Method of Estimating Carbon in Organic Substances.* J. Messinger. Ber. **23**, 2756—2760.

THIS is an improvement on the author's method of two years ago (Ber. **21**, 2910). The method is now applicable to compounds with which previously low results were obtained.



THE ESTIMATION OF CARBON IN ORGANIC SUBSTANCES.

The substance is weighed into a small tube 24 mm. long and 11 mm. wide, and is then introduced into the decomposition flask, which contains 6—8 grms. of chromic acid, care being taken that the chromic acid does not come into contact with the substance under analysis. The decomposition flask is fitted with a thistle funnel, and is connected to the reversed condenser and apparatus shown in the figure. 50 cc. of concentrated sulphuric acid are run into the flask. During the whole of the operation a gentle current of air (free from carbon dioxide) is passed through the apparatus. The asbestos plate underneath the flask is then warmed and thus the flask and contents are warmed by radiant heat from the plate alone until the sulphuric acid darkens. At this point, where decomposition of

the organic substance begins, the flame is entirely removed. The carbon dioxide (with some carbon monoxide) passes through the condenser and then over a heated mixture of copper oxide and lead chromate contained in a tube 15 cm. long. The gas ( $\text{CO}_2$ ) then passes through a U-tube, in one limb of which is sulphuric acid, in the other glacial phosphoric acid.

Thus dried it passes through weighed potash bulbs, after which is placed for safety a small tube containing soda lime and phosphoric acid. After the lapse of about 20 minutes warming may be once more proceeded with in the same manner as before, and after about 2½ hours the asbestos plate may be placed directly below the flask, and more strongly heated.

The whole operation is very easily carried out, and needs no watching.

With substances containing halogens, it is advisable to place, after the copper oxide tube, a small washing flask containing potassium iodide solution.—T. L. B.

*Apparatus for the Determination of Ammonias in Filter Sand and Sewage.* A. Hazen. Amer. Chem. J. **12**, 427—428.

In examining sand from filters it has been thought desirable to determine free and albuminoid ammonia, as this indicates the amount of organic matter stored, in the same terms as given for the filtered water or the sewage. The author carries out the determination by blowing steam, generated in a flask containing ammonia-free water, through another flask containing a quantity of the sand to be tested, and connected with a condenser. The steam very rapidly carries off the free ammonia which is invariably contained in the first 50 cc. of the distillate. Alkaline permanganate is then added to the sand, and the steaming continued. The first portion which distils over usually contains at least eight- or nine-tenths of the whole and the second portion almost all the rest.

The amount of free ammonia obtained in the case of sewage is the same as that found by the usual process of dilution with pure water and direct distillation. The albuminoid ammonia is commonly a little greater, owing to the concentration of the permanganate, but the results are sharper, and different determinations agree with each other more closely.—O. H.

*Volumetric Determination of Phenols.* J. Messinger and G. Vortmann. Ber. **23**, 2753—2756.

THE authors have described (Ber. **22**, 2312) a class of iodised phenols obtained by the action of iodine on alkaline solutions of the phenols. The formation of these compounds is made use of for the estimation of phenols, and a quantitative method has been worked out in the case of phenol, of thymol, of naphthol, and of salicylic acid.

**Phenol.**—In the reaction between iodine and an alkaline solution of phenol, 6 atoms of iodine are required for each molecule of phenol. The quantitative determination of phenol is carried out in the following manner:—2—3 grms. of the phenol are dissolved in caustic soda (at least 3 mols. of caustic soda to one of phenol). The solution is diluted to 250 or 500 cc., and of this, 5 or 10 cc., accurately measured, are warmed to about 60° in a small flask; decinormal iodine solution is now run in until the liquid has attained a deep yellow colour, due to excess of iodine. The whole is cooled, acidified with dilute sulphuric acid, and made up to 250 or 500 cc., and an aliquot part (about 100 cc.) is filtered and titrated with decinormal sodium thiosulphate solution. The amount of iodine used, multiplied by the factor 0.123518, gives the quantity of pure phenol. Thus:—

$$\begin{array}{rcl} 1 \text{ mol. phenol} & = & 93.78 \\ 6 \text{ atoms iodine} & = & 759.24 \\ & = & 0.123518. \end{array}$$

**Thymol.**—From an alkaline thymol solution the whole of the thymol is precipitated in the cold by iodine. For each molecule of thymol 4 atoms of iodine are necessary. The factor here is therefore—

$$\begin{array}{rcl} 1 \text{ mol. thymol} & = & 149.66 \\ 4 \text{ atoms iodine} & = & 598.64 \\ & = & 0.2956772. \end{array}$$

For the determination 0.1—0.3 grm. of thymol is dissolved in caustic soda (4 mols. of caustic soda to 1 of thymol), the solution has excess of decinormal iodine solution added to it, and is then acidified and further treated as in the case of phenol.

**$\beta$ -Naphthol.**—The alkaline solution (4 mols. of caustic soda to 1 of naphthol) must be heated to 50—60° before the addition of the iodine. The determination is carried out exactly as in the case of phenol. The factor is—

$$\begin{array}{rcl} 1 \text{ mol. naphthol} & = & 143.66 \\ 3 \text{ atoms iodine} & = & 379.62 \\ & = & 0.37843106. \end{array}$$

**Salicylic Acid.**—For solution of the salicylic acid it seems best to take 4 mols. of caustic soda to 1 of salicylic acid.

If too little alkali has been used a yellowish precipitate forms before an excess of iodine has been added; in such a case it is necessary to dissolve this precipitate by further addition of caustic soda. Precipitation should first occur when excess of iodine has been added. The alkaline solution requires warming to 50—60° before addition of the iodine. The factor here is—

$$\begin{array}{rcl} 1 \text{ mol. salicylic acid} & = & 137.67 \\ 6 \text{ atoms iodine} & = & 759.24 \\ & = & 0.18132606. \end{array}$$

Of course alkali free from nitrite must be used, or else the amount of iodine set free by a certain number of cc. of alkali on acidifying must be ascertained, and then the alkali used must always be accurately measured.—T. L. B.

*A Simplification of the Volumetric Determination of Free Acid in Oils.* Holde. Mitt. Königl. tech. Versuchs. 1890 151—153.

The method described in Mitt. Königl. tech. Versuchs. 1889, 116—119 (this Journal, 1890, 112), may be conveniently arranged for practical purposes as follows:—

1. **Light-coloured Oils.**—Ethereal solutions of which show the colour reactions of phenolphthalein.

The funnel B (see Fig. 1) is fitted into the cork A, which has two holes; and the cork then inserted into the mouth of the Erlenmeyer flask C, whose capacity is about 300 cc.; B is marked at *a* for 10 cc. and this quantity of the oil to

Fig. 1.

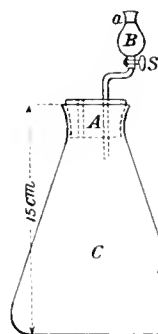


Fig. 2.

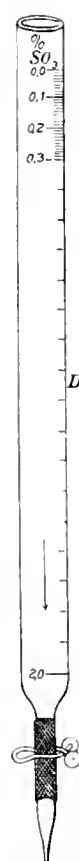
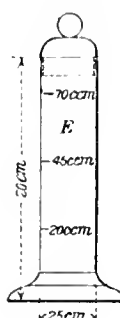


Fig. 3.





be tested, is poured into it, then the stop-cock S is opened and after the oil has flowed into C, B is washed with a neutralised mixture of ether (8 parts) and alcohol (2 parts) containing phenolphthalein, until the flask is filled to about c. Then A and B are removed and the liquid titrated. The burette D (see Fig. 2) contains an alcoholic solution of caustic soda 1 cc. of which corresponds to 0.005 gm. of sulphuric anhydride ( $\text{SO}_3$ ). The titration is continued until the red colouration becomes permanent. The graduation of D is such as to indicate directly the percentage of free acid contained in the oil, calculated as sulphuric anhydride, the latter being calculated as follows:—

If  $x$  cc. caustic soda be necessary to neutralise 10 cc. of oil, containing 1 per cent. free acid, then we have the equations—

$$\frac{10 \cdot x \times 0.005}{0.914} = 1, \text{ and } x = 18.3 \text{ cc.}$$

Where 0.914 is taken as an average specific gravity of the oils which are usually used for lubricating purposes, and 0.005 gm. as the quantity of sulphuric anhydride corresponding to 1 cc. of caustic soda, hence 18.3 cc. of the burette correspond with 1 per cent. of free acid in the oils, and this space is divided again so as to enable the operator to read  $\frac{1}{10}$ ,  $\frac{1}{100}$ ,  $\frac{1}{1000}$ , &c. per cents. Two per cent. is the maximum which the burette indicates.

2. *Dark-coloured Oils.*—The solutions of which in ether and alcohol do not show the reactions of phenolphthalein.

20 cc. of the oil to be tested are poured into the graduated and stoppered measure E (see Fig. 3), and 50 cc. of absolute alcohol added. Then both liquids are well shaken and allowed to stand. After the separation of the liquids 25 cc. of the alcoholic portion are poured into the Erlenmeyer flask, 20 cc. of the above described mixture of ether, alcohol, and phenolphthalein added, and then the liquid titrated as above. When the acid found is more than 0.03 per cent. then the remaining 25 cc. alcohol in the graduated measure has to be poured off and again shaken with 50 cc. of absolute alcohol. A second titration is then made, and the amount of acid found this time is the correct one. The calculation is the same as above, as for each titration 10 cc. oil (of the 20 cc. taken) were used.

The advantage of working according to this method lies in the convenience with which the necessary quantities of the substances are easily and exactly obtained, and in entirely excluding all possible losses of substance, as well as all tedious calculations.

The following table gives proofs of the correctness of this method, by comparing it with the method of Geissler (see this Journal, 1890, 112):—

No.	Kind of Oil.	Percentage of Acid calculated as $\text{SO}_3$ .	
		Geissler.	Holde.
1	Refined rape oil.....	0.64	0.64
2	" " .....	0.60	0.59
3	" " .....	0.95	0.95
4	Olive oil .....	0.44	0.44
5	" .....	2.82	2.82
6	Refined rape oil.....	1.00	1.01
7	" " .....	0.63	0.61
8	" " .....	0.28	0.28

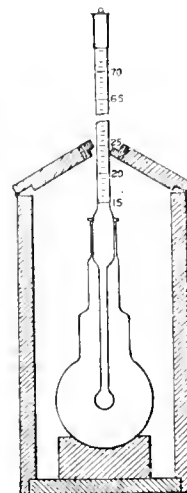
—H. S.

#### *A Method for Comparing the Solidifying Points of different Sorts of Tallow.* Finkener. Mitt. König. tech. Versuchs. 1890, 153—158.

In an article upon the determination of the fatty acids of tallow, &c. (see Mitt. König. tech. Versuchs. 1889, 27—41; this Journal, 1889, 423—424), it is explained that different

quantities of tallow used in the tests, and different ways of cooling the melted tallow, change the solidifying points considerably. For comparisons it is therefore essential that the working of the methods should always be the same.

The author used for the following experiments small globular flasks (see figure) of about 50 mm. diameter, and instead of covering them with wadding (compare this



THE SOLIDIFYING POINTS OF TALLOW.

Journal, 1889, 423), to effect a slow cooling of the tallow, he found it a more practical arrangement to place the flasks containing the tallow in a wooden box of 144 mm. in height and 70 mm. in length and width (see figure). In the middle of the bottom is fixed a piece of cork 22 mm. high, with a small hollow in which the flask rests. A thermometer is so arranged as to bring its bulb into the centre of the flask. The flask is marked for a determined quantity of tallow, and the thermometer has a rim, so that the tallow is always under the ordinary atmospheric pressure. The top of the box has two flaps, which can be closed as the figure shows, by which arrangement the thermometer as well as the flask are kept in the middle of the box. The middle part of the flask is 25 mm. wide, the upper 12 mm. The bulb of the thermometer has 9 mm. and its tube 5 mm. diameter.

The graduation of the thermometer which indicates  $\frac{1}{2}^{\circ}\text{C}$ . should go to about  $120^{\circ}\text{C}$ . This apparatus is used as follows:—150 grms. of tallow are melted on a water-bath (the water boiling), and left there not less than half an hour. Then the melted tallow is poured into the flask till the mark is reached, about 80 grms. of tallow being used for each test. Then the thermometer is put into the tallow and the flask placed in the box, as described above. Every two minutes the temperature is noted, the solidifying point being arrived at either when the temperature remains constant for some minutes, or when it ceases to fall and rises again a little; in the latter case the highest point of the rising is the solidifying point. The time necessary for the entire test is two hours; for the cooling alone about one hour; in working with three apparatus at the same time, three tests can conveniently be made in three hours. After the test the tallow is melted again in boiling water, the flask emptied, and cleaned with a little ether. The author has made experiments with different sorts of tallow with this apparatus, and has ascertained what effect alterations in the described details of making the experiments have upon the temperature of the solidifying points. If different maximum temperatures (from  $50^{\circ}$  to  $100^{\circ}\text{C}$ .) are applied in melting the tallow on the water-bath, the experiments show that the solidifying points become lower with the rising of the maximum temperature, but that they always become alike for the same sort of tallow after it has been heated to  $100^{\circ}\text{C}$ . for half an hour; further heating does not affect the test. At ordinary temperatures this result is not changed, but if the temperature of the testing room is

low (about 10° C.) a further reduction of 0.2° C. takes place. It is of no importance at what temperature of the tallow the flask is placed in the box, provided that such temperature is at least 10° C. above the solidifying point. (Compare also this Journal, 1890, 112, and 743—744).

—H. S.

*Determination of Fats in Soap.* M. Saupe. Pharm. C. H. N. F. 1890, 11, 314.

The author recommends shaking with ether as follows:—Two grms. of fine soap shavings taken from the interior of the sample to be tested, are dissolved in 50 cc. of water in a stoppered measure, and 5 cc. of dilute sulphuric acid (1:4) added. Then 54 cc. of dilute ether are added, the mixture shaken three or four times, and allowed to stand. 20 cc. of the clear ether solution of the fats thus obtained are withdrawn with a pipette, put into a small beaker, the ether evaporated, and the residue weighed. The percentage is obtained by multiplying by 125.—H. S.

*The Application of the Oleo-refractometer to the Detection of Adulterations.* F. Jean. Bull. Soc. Chim. [3], 4, 105—108.

The apparatus consists of a collimator, telescope, and a small metal vessel fitted with parallel plate glass sides, and into which is placed a small silver tube with plate glass ends, arranged at a definite angle. The apparatus is so arranged that the light passes through the collimator, the plate glass sides of the outer metal vessel and interior silver cylinder, and thence to the telescope. The collimator, instead of having a slit, has a semicircular stop, which leaves half the field in darkness; the telescope is furnished with a glass scale placed in the focus of the eye-piece. If both the outer metal vessel and the interior silver tube be filled with the same oil, there will be no refraction, and the field will be divided into a light half and a dark half. If the inner tube be filled with a different oil, there will be refraction, the amount depending on the nature of the oil, and being measured by the displacement of the line dividing the field into dark and light, relative to the scale of the telescope. If then one of these oils is known to be a pure sample, and the other a commercial sample, if the latter is pure there will be no refraction, and if it is not, the amount of refraction will indicate the nature of the adulteration and the amount. This method is stated to be particularly adapted to the examination of lard and butter. (See this Journal, 1890, 218.)—A. L. S.

*Japan Wax.* O. Kleinstück. Chem. Zeit. 14, 1303—1304.

In continuation of work previously done and recorded in the Arch. Pharm. 26, 166, the author obtained three samples of Japan wax from different sources for the present investigation. The closeness with which the specific gravity of Japan wax approximates to that of water, coupled with its high coefficient of expansion, gives rise to the curious phenomenon of its floating in water at temperatures above 18° C. and sinking below 15° C. This behaviour is modified by the property, which it shares with other similar substances, of being lighter after fusion and cooling than is proper to the temperature at which it is, its specific gravity becoming normal only some time after it has solidified.

The high coefficient of expansion of Japan wax allows of direct demonstration, a bar 625 mm. long expanding 4 mm. when its temperature is raised from -6° C. to 22° C. A similar experiment with a rod of beeswax gave 3 mm. as a result. The following interesting table, given in the next column, lays bare the whole reason for what is at first sight very puzzling behaviour.

The specific gravity of the Japan wax examined was so constant for fixed temperatures both in the case when it had been recently fused and in the case when it was in its normal and denser condition, that it was found possible to calculate to a nicety the temperature to which water in which it was placed had to be brought in order that the wax might be of the same specific gravity and sink or float indifferently. The change of specific gravity was sufficiently sharply defined to

Temperature °C.	Specific Gravity compared with that of Water at 4° C.		
	Japan Wax.		Water.
	In the Normal Condition.	After recent Fusion.	
6.5	..	1.00237	0.99995
7.2	1.00737	..	0.99991
17.0	..	0.99123	0.99884
17.5	0.99846	..	0.99875
23.0	..	0.98747	0.99762
26.5	0.98615	0.98683	0.99674

permit of a difference of 0.5° C. at the critical temperature determining whether it would float or sink. The mean of the temperature at which it just floated and that at which it just sank was in all cases almost absolutely identical with the calculated temperature at which the specific gravities of the water and wax should be the same. The precision of the occurrence of the phenomenon almost raises it into a means of differentiating Japan wax from similar bodies.

—B. B.

ANALYTICAL AND SCIENTIFIC NOTES.

*On the Action of Nitrous Anhydride (Nitrogen Trioxide), dissolved in Carbon Disulphide, upon different Organic Compounds.* L. H. Friedburg and J. A. Mandel. J. Amer. Chem. Soc. 12, 7—12 and 54—57.

The authors aim at modifying the intense action of free nitrous anhydride on organic bodies by the intervention of a diluent, and for this purpose they dissolve carefully-prepared nitrous anhydride in carbon disulphide. The substance to be acted upon is also dissolved preferably in the same solvent, and the heat of reaction is reduced as much as possible by keeping the solutions in a freezing mixture. By operating in this way on benzene, the products were exclusively mono-nitrobenzene and para-dinitrobenzene. Phenol yielded a mixture of ortho-nitrophenol, para-nitrophenol, and nitrosophenol. Weselsky had previously carried out this reaction in a similar manner, using ordinary ether as a diluent, but he obtained diazophenol nitrate in place of the nitrosophenol.

Diphenylamine was submitted to the reaction in three different ways: by solution in absolute alcohol, in carbon disulphide, and in carbon disulphide in presence of sulphur. By the first method para-mononitro-, para-dinitro-, and ortho-dinitro-diphenylamine were obtained, whilst the other two methods yielded principally para-mononitro-diphenylamine, the sulphur taking no part in the reaction.

The authors apply the method to diphenyl, prepared by passing the vapour of benzene in an atmosphere of carbonic acid several times through a red-hot tube, and purifying the product by distillation with steam and crystallisation from alcohol. On treatment with nitrous anhydride in the manner already described, a substance was obtained which crystallised from alcohol in long colourless brilliant needles, melting at 113° C., which proved to be para-mononitrodiphenyl. Benzoinitrile was prepared by Krüss's method by heating to 190° C. 2 mols. of benzoic acid with 1 mol. of lead sulphocyanide, but no notable reaction was obtained on treating it with nitrous anhydride. Benzaldehyde under the same treatment was simply oxidised to benzoic acid. Benzoic acid furnished a nitroso compound which, after purification, melted at 114° C., and gave numbers agreeing with the formula for nitrosobenzoic acid.—G. H. B.

*The Diffusion of Carbon Dioxide through Caoutchouc.* Kobbe. Thonind. Zeit. 1890, 14, 297.

The author has constructed an apparatus to show that carbon dioxide easily diffuses through caoutchouc. Lime water to about half an inch in depth is poured into two

Erlenmeyer flasks A and B, and A closed loosely, B airtight with corks. A and B are connected with each other by a U-tube, the ends of which reach the lime water in both flasks. The cork of B has two other holes, through which an india rubber tube of 40 cm. length is passed airtight, forming a sling or bend inside B, but not touching the lime water. One end of this tube passes into the open air, the other one is connected with a Woulfe's bottle. The lime water in B remains almost perfectly clear as long as atmospheric air goes through the india-rubber tube, but when the tube is filled with carbon dioxide and closed by screw clamps, and the apparatus allowed to stand, then the lime water in B loses its transparency, whilst that in A shows very little change. After 24 hours the precipitates of both bottles were filtered, washed, and heated to redness. It was thus found that B contained 0.0598 grm. more calcium carbonate than A. The author also ascertained that hydrogen does not show the same degree of diffusibility through caoutchouc as carbon dioxide.—H. S.

*On the Phenomenon of Coagulation.* A. Béchamp. Bull. Soc. Chim. [3], 4, 181—186.

The author points out the great confusion which exists in the use of the term "coagulation." Chemists now use the word "coagulate" as a synonym of "to become insoluble," and when albuminoids are referred to, coagulate = "to become insoluble in water." All colourless albuminoids which are precipitated from the aqueous solution on heating have been by some considered identical: casein has been identified with blood-fibrin, &c., and the precipitation of the albumen has been attributed to the presence of some acid or base as an impurity. Wurtz and the author have however both shown that some pure soluble albumens exist which are coagulable by heat, though other pure albumens are incoagulable.

The term coagulation has been applied to the spontaneous curdling of both fresh and boiled milk, to the curdling of fresh and boiled milk by addition of rennet and to their coagulation by means of a suitable acid. Yet these are essentially different phenomena, since fresh milk never curdles without previously becoming acid, whereas boiled milk may remain neutral and still curdle; rennet coagulates milk even when it is alkaline, &c. It is also evident that curdling in these cases is not again the same as that produced by the addition of an acid.

The author intends to investigate the subject completely; the present communication, however, deals experimentally only with the coagulation of milk by an addition of acid.

*Casein and the other Albuminoids of Milk.*—The author has previously shown that cow's milk does not contain casein, but alkaline caseinates, and concludes that when acetic acid precipitates casein, this is merely a phenomenon of displacement and not of "coagulation proper."

*Pure casein* is prepared as follows: Pure acetic acid is added drop by drop to cow's or goat's milk as soon as possible after it has been drawn from the udder, until the liquid turns litmus paper the colour of onion skin; at this point the diacaseinate first formed is completely decomposed. The milk must *not* be warmed previously to this operation, as the casein obtained would then contain lactalbumin. Shortly after the addition of the acid the milk curdles. It is then filtered and the whey comes through quite clear. The casein precipitated has brought down with it the milk globules and "microzymas"; it must be thoroughly washed to free it from all soluble milk constituents, drained and then treated with ether to dissolve out all the fatty matters. The mass is then again washed with water and stirred into a volume of water equal to that of the milk originally taken; and the liquid is made slightly but unmistakably alkaline with ammonium sesquicarbonate. The casein-precipitate dissolves, but the solution remains turbid owing to the presence of the debris of the globule envelopes and the microzymas. Filtration is hastened by heating up filter-paper with the solution. To the clear filtrate acetic acid is added as before, in just sufficient amount to exactly precipitate the casein.

The casein is then washed with water, and if the product is pure the wash-water will give no precipitate

either on heating or on addition of alcohol. If a precipitate be formed under these circumstances the whole mass must be redissolved and reprecipitated. Prolonged washing with water frees the casein from every trace of acetic acid.

By the above process a pure product is obtained with constant properties. The specific rotatory power in ammoniacal solution for the sensitive tint is  $[\alpha]_D = -130^\circ$ . The lower numbers obtained by previous observations are due to an admixture of lactalbumin. Casein is only slowly and slightly soluble in water at the temperature of the air; 1 litre of water left in contact with excess of the substance for 52 hours, with frequent agitation, dissolves 1.005 grms.

The specific rotatory power of the aqueous solution for sodium light is  $[\alpha]_D = -117^\circ$ . Gradually heated with water to  $100^\circ$  casein becomes softer and softer, and seems to melt. The water in contact with the soft mass contains 2.37 grms. of casein per litre. The solubility therefore increases with rise of temperature. When the quasi-fluid casein cools it becomes hard and may be powdered by grinding under water, redissolved in ammonium sesquicarbonate, and reprecipitated in its original state by the addition of acetic acid. Neither heat nor the addition of an acid coagulates casein solutions.

Casein solutions act on litmus like carbonic acid, and acid caseinate of potassium, sodium, ammonium, and calcium may be obtained which redden litmus and are not precipitated by carbonic acid.

The caseinates of these bases form solutions which are neither precipitated by alcohol nor coagulated on heating. At the boiling point, however, the solution of calcium caseinate becomes cloudy, but again becomes clear on cooling; casein is therefore a substance quite distinct from albumin.

*Whey Albuminoids.*—Whey contains other albuminoids, differing from casein. Whey is coagulable by heat, and the coagulum formed is insoluble both in water and ammonium sesquicarbonate. [It is evident, therefore, that in the preparation of pure casein, as above, the milk must not be warmed before precipitation.]

To the clear whey, "alcohol at  $95^\circ \text{C}.$ " is added until no further precipitate is formed, a volume of alcohol at least equal to twice the original volume being necessary. A voluminous precipitate is formed, which is collected on a filter and washed with "alcohol at  $80^\circ \text{C}.$ " until it is freed from lactose; it is then drained, but before it dries it is stirred into water and after some time filtered. A certain portion of the mass dissolves in water, from which it may be reprecipitated by alcohol; it is therefore washed with water till the wash water gives no precipitate with alcohol. The undissolved portion is taken up with a dilute solution of ammonium sesquicarbonate; a solution of an albuminoid and an inorganic residue are thus obtained. The albuminoid may be precipitated from its solution by means of acetic acid and this product is *lactalbumin*. The portion soluble in water is *galactozymase*; it must be freed from lactalbumin by dissolving in water and reprecipitating several times. The final product is entirely soluble in water, and it may be found impossible to reprecipitate it with alcohol except by adding a trace of sodium or ammonium acetate to the solution. The galactozymase of cow's milk renders starch paste fluid, but does not convert it into sugar.

It is evident that lactalbumin exists in the soluble state in milk, but the treatment with alcohol, which precipitates it, renders it at the same time insoluble in water, though it remains soluble in a solution of ammonium sesquicarbonate. If, however, it be stirred into water and heated to  $100^\circ$ , it contracts without softening, and then becomes insoluble not only in ammonium sesquicarbonate, but also in dilute ammonia.

Galactozymase, on the other hand, is in no wise coagulable by alcohol, but it also yields solutions which are coagulated by heat, and they thereby lose their "zymasic" function.

The study of the specific rotatory power of lactalbumin and galactozymase proves them to be definite chemical compounds.

The author intends to publish further results shortly.

—P. J. H.

## New Books.

**THE THRESHOLD OF SCIENCE.** A variety of simple and amusing experiments illustrating some of the chief Physical and Chemical Properties of Surrounding Objects and the effects upon them of Light and Heat. By C. R. ALDER WRIGHT, D.Sc., F.R.S., Lecturer on Chemistry and Physics in St. Mary's Hospital Medical School, London. London: Charles Griffin and Co., Exeter Street, Strand. 1891.

OCTAVO volume elegantly bound in cloth and gilt, price 7s. 6d., a volume more plainly bound in cloth being offered at 6s. It contains Frontispiece, Table of Contents, Introduction, and Text covering 384 pages, concluded by a copious Alphabetical Index. There are upwards of 203 well-executed wood engravings. The intent of the work may be readily gathered, especially after another glance at the title, from the following paragraph in Dr. C. R. Alder Wright's introduction:—"Whilst the juvenile philosopher finds pastime and entertainment in constructing simple apparatus and preparing elementary experiments, he may at the same time be led to observe correctly what happens, to draw inferences and make deductions therefrom, and by comparing together the results obtained in different cases may gradually acquire some degree of elementary training in the proper exercise of those mental qualities which systematic scientific education chiefly calls forth; and in addition may pick up a variety of pieces of information as to the 'why and wherefore' of things connected with the phenomena of nature generally, and more particularly with matters of every-day occurrence, especially the various arts and manufactures and the innumerable scientific applications nowadays met with on all sides of every-day life." It is claimed, as a special advantage, that the book is written not with a view to aid in preparing for examinations, but rather to foster the habit of scientific thought in an elementary way and of deliberate experimental inquiry.

The work is classified as follows:—§ 1. States of Matter. § 2. Physical Changes of State, due to Heat and Pressure and not accompanied by Chemical Action. § 3. Changes of State, due to Solution, not accompanied by Chemical Action. § 4. Solution and Separation from Solution by Actions involving Chemical Changes. § 5. Chemical Actions producing Change of State without the Employment of Solvents. § 6. Physical Adhesion and allied phenomena of Surface Action. § 7. Effects of Heat upon Bodies, other than Change of State, and Production of Chemical Action. § 8. Radiant Action; Visible Light. § 9. Radiant Action; Invisible Light.

**A TREATISE ON ELECTRO-METALLURGY.** Embracing the Application of Electrolysis to the Plating, Depositing, Smelting and Refining of various Metals, and to the Reproduction of Printing Surfaces and Art-work, &c. By WALTER G. McMILLAN, F.I.C., F.C.S., Chemist and Metallurgist to the Cossipore Foundry and Shell Factory; late Demonstrator of Metallurgy in King's College, London. London: Charles Griffin and Co., Exeter Street, Strand. 1890.

OCTAVO volume, bound in cloth, price 10s. 6d. It contains Preface, Table of Contents, List of Tables used in the treatise, 316 pages of Subject-matter, 29 pages devoted to a glossary of substances commonly employed in Electro-metallurgy, 12½ pages devoted to Addenda, chiefly composed of tables or tabulated matter, terminated by a List of Antidotes to Poisons contained amongst the substances used in Electro-metallurgy, and a copious Alphabetical Index. The text is illustrated by 101 effective and well-executed wood engravings. The matter is brought well up to date, including descriptions such, for example, as that of Elmore's recent process for the manufacture of seamless copper tubes of extraordinary strength and tenacity by electro-deposition of the pure metal, meanwhile continually burnishing it during the whole course of the depositing operation.

The work is sub-divided as follows:—CHAP. I. Introductory and Historical. II. Theoretical and General. III. Sources of Current. IV. General Conditions to be observed

in Electro-Plating. V. Plating Adjuncts and Disposition of Plant. VI. The Cleansing and Preparation of Work for the Depositing Vat, and subsequent Polishing of Plated Goods. VII. The Electro-deposition of Copper. VIII. Electrotyping. IX. The Electro-deposition of Silver. X. The Electro-deposition of Gold. XI. The Electro-deposition of Nickel and Cobalt. XII. The Electro-deposition of Iron. XIII. The Electro-deposition of Platinum, Zinc, Cadmium, Tin, Antimony, and Bismuth; Electro-chromy. XIV. The Electro-deposition of Alloys. XV. Electro-metallurgical Extraction and Refining Processes. XVI. The Recovery of certain Metals from their Solutions or Waste Substances. XVII. The Determination of the proportion of Metal in certain Depositing Solutions. XVIII. A Glossary of Substances commonly employed in Electro-metallurgy. Addenda.

**UNTERSUCHUNGEN AUS DER PRAXIS DER GÄRUNGS-INDUSTRIE.** VON DR. EMIL CHR. HANSEN. I. Heft. Zweite, vermehrte und neubearbeitete Auflage, mit vierzehn Abbildungen. München und Leipzig: Druck und Verlag von R. Oldenbourg. 1890. London: H. Grevel and Co., 33, King Street, Covent Garden.

LARGE 8vo. brochure, in paper cover, being part No. 1 of a work now in the press. It commences with an INTRODUCTION to the first edition, and this is followed by one to the second edition. The text covers 85 pages, and these are illustrated by 14 wood engravings.

The work is sub-divided as follows:—1. The Cultivation of Yeast in the Service of Industry: 1. The introduction of pure and selected Yeasts in the Making of Beer, and the results arrived at. 2. The Manufacture of Pure Cultivated Yeasts. II. Observations on Varieties of Brewery Yeasts: 1. Means of recognising the Varieties of *Saccharomyces*. 2. Higher and Lower Yeasts. 3. Investigations of Varieties of Yeasts, which have been tried in practice. 4. The Variations. 5. Chief Results. III. The Practical Investigation of Beer in the Store Casks, with respect to its permanency.

**REPORT ON ANALYSES AND RESEARCH** conducted in 1886-1889 in the Central Chemical Laboratory of Italian Customs, under the Direction of Senator CANNIZZARO. Compiled by R. NASINI and V. VILAVECCHIA.

This report is dedicated, through Senator Cannizzaro, to the Queen of Italy. Its object is twofold; 1st, to give an account of the industrial and commercial methods, and for the tests and analyses of various goods, together with suitable trade notices to serve as a guide to chemists engaged in analytical work connected with the tariff; and 2nd, to give a description of simple analytical methods and their interpretation, for the instruction of Custom house officials. Some of the analytical methods have been specially devised by the compilers for the exigencies of their work, while others are modifications and simplifications of those already in practice. In the latter case full references are given to scientific memoirs, text-books on commercial analysis, and reports of various congresses. Special attention is given to the criteria adopted by the Direction for the classification of samples under dispute. Reference is also made to certain investigations which are being carried on in the laboratory, especially upon the sophistication of salt, glucose, and spirits, the tariffs on acetic acids, as also on tobaccos in relation to the soil in which they are cultivated in Italy. These investigations will form the subject of special publications from the laboratory.

The report is divided into chapters and sections arranged according to the various categories of the tariff. A description, with plans of the laboratory, is given in the preface.

(See various sections of the Journal and Patent Literature and the Trade Report of this issue.)

**ERRATUM.**—This Journal, 1890, 980, "New Books" column 1, for "*Endiometry*" read "*Endimetry*."

## Trade Report.

## TARIFF CHANGES AND CUSTOMS REGULATIONS.

## THE CUSTOMS TARIFFS OF AUSTRALASIA.

(Showing the Duties on Chemicals in force throughout Australasia on January 1, 1890.)

Articles.	New South Wales.	Victoria.	South Australia.	Queensland	West Australia.	Tasmania.	New Zealand.	Fiji.
Alum.....	Free	Free	10 °	15	12½ °	Free	Free	15 °
Arsenic.....	Free	Free	10	15	12½ °	Free	Free	15 °
Artists' colours.....	Free	20 °	Free	15	12½ °	12½ °	Free	10
Benzol.....	Free	6d. gall.	6d. gall.	6d. gall.	12½ °	12½ °	6d. gall.	1d. gall.
Chemicals.....	Free	..	6d. lb.	15	12½ °	12½ °	15 °	15 °
Cream of tartar.....	Free	25 °	10 °	15	12½ °	12½ °	1d. lb.	15
Drugs (unenumerated).....	Free	25 °	10 °	15	12½ °	12½ °	15 °	15
Dyes.....	Free	Free	Free	Free	12½ °	Free	Free (aniline)	15
Essences (not being alcoholic compounds).....	Free	Free	10 °	15 °	12½ °	12½ °	15 °	15
India rubber tubing.....	Free	Free	10 °	15	12½ °	12½ °	Free	10
Instruments (optical, scientific, and surgical).....	Free	Free	Free	15 °	Free	Free	Free	5 °
Linseed oil.....	6d. gall.	6d. gall.	6d. gall.	1s. gall.	12½ °	12½ °	6d. gall.	9d. gall.
Methylated spirit.....	4s. gall.	1s. gall.	3s. gall.	5s. gall.	1s. gall.	3s. gall.	1s. gall.	2s. gall.
Naphtha.....	6d. gall.	6d. gall.	6d. gall.	6d. gall.	6d. gall.	1s. 3d. gall.	6d. gall.	6d. gall.
Oil, cocoa-nut.....	Free	Free	Free	1s. gall.	6d. gall.	Free	6d. gall.	Free
Oil, kerosene.....	Free	Free	3d. gall.	6d. gall.	6d. gall.	1s. gall.	6d. gall.	6d. to 1s. 3d. gall.
Oil, rape.....	6d. gall.	6d. gall.	6d. gall.	1s. gall.	6d. gall.	1s. 3d. gall.	6d. gall.	6d. gall.
Oil (unenumerated).....	6d. gall.	6d. gall.	6d. gall.	6d. gall.	6d. gall.	1s. 3d. gall.	Free	6d. gall.
Opium.....	20s. lb.	30s. lb.	20s. lb.	20s. lb.	20s. lb.	20s. lb.	40s. lb.	15s. lb.
Patent medicines.....	Free	25 °	20 °	15 °	12½ °	12½ °	25 °	15 °
Perfumery (including fancy and scented soaps).....	Free	25 °	25 °	15 °	20 °	12½ °	25 °	15 °
Phosphorus.....	Free	Free	Free	15 °	12½ °	Free	Free	15 °
Quicksilver.....	Free	Free	Free	Free	12½ °	Free	Free	5 °
Sheep-dip (Cooper's and Little's).....	Free	Free	Free	15 °	12½ °	10 °	Free	5 °
Shellac.....	Free	Free	Free	Free	12½ °	Free	Free	15 °
Soap.....	Free	2d. lb.	1d. lb.	10s. cwt.	3s. cwt.	1d. lb.	5s. cwt.	1d. lb.
Soap, scented and fancy.....	Free	4d. lb.	1d. lb.	10s. cwt.	20	3d. lb.	25 °	15 °
Soap powder.....	Free	20 °	2d. lb.	15	12½ °	12½ °	20 °	10 °
Soda ash.....	Free	Free	Free	Free	Free	Free	1s. cwt.	15 °
Soda, bicarbonate.....	Free	Free	2s. cwt.	1s. cwt.	12½ °	1d. lb.	1s. cwt.	15 °
Soda, caustic.....	Free	Free	Free	1s. 6d. cwt.	12½ °	Free	Free	15 °
Soda, crystals.....	Free	2s. cwt.	2s. cwt.	2s. cwt.	40s. ton	½d. lb.	2s. cwt.	20s. ton
Soda nitrate.....	Free	Free	5 °	5 °	12½ °	Free	Free	Free
Soda, for manure.....	Free	Free	Free	Free	Free	Free	Free	Free
Soda, silicate.....	Free	Free	5 °	15 °	12½ °	Free	Free	15 °
Spirits of wine.....	14s. gall. pf.	12s. gall. pf.	11s. gall. pf.	12s. gall. pf.	20s. gall. pf.	15s. gall. pf.	16s. gall. pf.	14s. gall.
Spirits, perfumed.....	15s. gall. pf.	21s. gall. pf.	21s. gall. pf.	20s. gall. pf.	20s. gall. pf.	21s. gall. pf.	21s. gall. pf.	14s. gall.
Sugar of milk.....	Free	Free	10 °	15 °	12½ °	12½ °	15 °	15 °
Salphuric acid.....	Free	5s. cwt.	5s. cwt.	2s. 6d. cwt.	12½ °	Free	Free	15 °
Thermometers.....	Free	Free	Free	15 °	Free	Free	Free	5 °
Vermilion.....	Free	Free	2s. cwt.	5s. cwt.	12½ °	½d. lb.	..	10 °
Vinegar.....	Free	6d. gall.	9d. gall.	9d. to 1s. gall.	6d. gall.	9d. gall.	6d. gall.	6d. gall.

—Chemist and Druggist.

## CANADA.

*New Customs Tariff—concluded.*

Articles.	Duties levied.
	Dols. Cts.
India-rubber clothing, or clothing made water-proof with india-rubber, not elsewhere specified.	35 % <i>ad val.</i>
Rubber belting, hose, packing, mats and matting, and cotton or linen hose lined with rubber.	Lb. 0'05 & 15 % <i>ad val.</i>
India-rubber vulcanised handles for knives and forks	10 % <i>ad val.</i>
Gutta-percha manufactures.	25 % <i>ad val.</i>
Japanned, patent, or enamelled leather.	25 % <i>ad val.</i>
Leather, sole.	Lb. 0'00 & 15 % <i>ad val.</i>
Leather, sole, tanned, but rough or undressed.	10 % <i>ad val.</i>
Skins for Morocco leather, tanned, but not further manufactured.	15 % <i>ad val.</i>
All other leather and skins, tanned, not otherwise specified	20 % <i>ad val.</i>
China and porcelain ware	30 % <i>ad val.</i>
Earthenware and stoneware, viz., demijohns or jugs, churns and crocks, 3 cents per gallon of holding capacity	Gall. 0'03
Earthenware or stoneware, brown or coloured, and Rockingham ware, white granite or ironstone ware, "C. C." or cream-coloured ware, decorated, printed, or spouged, and all earthenware not elsewhere specified	35 % <i>ad val.</i>
Drain tiles not glazed.	20 % <i>ad val.</i>
Drain-pipes, sewer-pipes, chimney linings, or vents and inverted blocks, glazed or unglazed, and earthenware tiles	35 % <i>ad val.</i>
Glass carboys and demijohns, empty or filled, bottles and decanters, flasks and phials, of less capacity than 8 oz.	30 % <i>ad val.</i>
Flasks and phials of 8 oz. capacity and over, telegraph and lightning rod insulators, jars, and glass balls, and cut, pressed, or moulded tableware	Doz. pieces 0'05 & 30 % <i>ad val.</i>
Common and colourless window glass, and plain, coloured, stained, or tinted, or muffled glass in sheets.	20 % <i>ad val.</i>
Plate glass, not coloured, in panes of not over 30 square feet	Sq. ft. 0'05
Plate glass, in panes of over 30 and not over 70 square feet each.	" 0'08
Photographic dry plates	" 00'9
Albuminised paper, chemically prepared for photographers use	25 % <i>ad val.</i>
Paper of all kinds, not elsewhere specified	25 % <i>ad val.</i>
Manufactures of paper, including ruled and border papers, paperies, boxed papers, envelopes, and blank books	35 % <i>ad val.</i>
Ink for writing	25 % <i>ad val.</i>
Type metal	10 % <i>ad val.</i>
Brass, manufactures of, not elsewhere specified.	30 % <i>ad val.</i>
Gold and silver leaf and Dutch metal.	30 % <i>ad val.</i>
Copper, manufactures of, not elsewhere specified.	30 % <i>ad val.</i>
German and nickel silver, manufactures of, not plated	25 % <i>ad val.</i>
Phosphor bronze in blocks, &c.	10 % <i>ad val.</i>
Plated ware, electroplated or gilt, of all kinds, whether plated wholly or in part	30 % <i>ad val.</i>
Ferro manganese, ferro silicon, spiegel, steel bloom crds, for the manufacture of iron and steel.	Ton 2'00
Lead bars, blocks and sheets.	100 lb. 0'00

CUSTOMS TARIFF OF CANADA—*cont.*

Articles.	Duties Levied.
	Dols. Cts.
Lead, old scrap and pig	100 lb. 0'40
Lead pipe and lead shot	Lb. 0'01½
Lead and all manufactures of lead not otherwise specified	30 % <i>ad val.</i>
Nickel anodes	10 % <i>ad val.</i>
Tinware and manufactures of tin not elsewhere specified.	25 % <i>ad val.</i>
Tin crystals	20 % <i>ad val.</i>
Zinc chloride, salts and sulphate of	5 % <i>ad val.</i>
Zinc, manufactures of, not elsewhere specified	25 % <i>ad val.</i>
Cement, burnt and unground.	100 lb. 0'07½
Cement, hydraulic, or water lime, ground, including barrels	Barrel 0'40
Cement in bulk or in bags	Bushel 0'09
Cement, Portland or Roman, shall be classed with all other cement at specific rates as above provided.	
Water limestone or cement stone	Ton of 13 cub. ft. 1'00
Coal tar and coal pitch.	10 % <i>ad val.</i>
Plaster of Paris or gypsum, ground, not calcined	100 lb. 0'10
Plaster of Paris, calcined or manufactured, 15 cents per 100 lb. or 45 cents per barrel of not over 300 lb.	" 0'15
Pimblago.	15 % <i>ad val.</i>
Do., all manufactures of, not otherwise specified.	30 % <i>ad val.</i>
Saltpetre	20 % <i>ad val.</i>
Asbestos, in any form other than crude, and all manufactures thereof	25 % <i>ad val.</i>
Celluloid, moulded into sizes for handles of knives and forks, not bored or otherwise manufactured; also moulded celluloid balls and cylinders coated with tin foil or not, but not finished or further manufactured	10 % <i>ad val.</i>
Fireworks	25 % <i>ad val.</i>
Blasting and mining powder	Lb. 0'03
Canister powder in pound and a half tins	" 0'15
Cannon and musket powder in kegs and barrels.	" 0'04
Giant powder, duxin, dynamite, and other explosives in which nitro-glycerin is a constituent part	" 0'05 and 20 % <i>ad val.</i>
Gun, rifle, and sporting powder in kegs, half kegs, or quarter kegs, and other similar packages.	Lb. 0'05
Nitro-glycerin	" 0'10 and 20 % <i>ad val.</i>
Electric arc light carbons or carbon points not exceeding 12 in. in length 2 dols. 50 cents per 1,000, and in proportion for greater or less lengths.	1,000 2'50
Oil, spermaceti, whale and other fish oils, and all other articles the produce of the fisheries, not specially provided for.	20 % <i>ad val.</i>

*Exemptions.*—The following articles are exempt from Customs duty:—

Ambergris.	Pelts, raw.
Bones, crude, not manufactured, burned, calcined, ground, or steamed.	Skins, undressed, dried, salted, or pickled.
Bone-dust and bone-ash for manufacture of phosphate and fertilisers.	Celluloid xylonite or xyolite in sheets, and in lumps, blocks, or balls, in the rough.
Guano and other animal and vegetable manures.	Ammonia, sulphate of.
Hides, raw, whether dry, salted, or pickled.	Sal-ammoniac.
	Aloes, ground or unground.
	Alum, in bulk only, ground or unground.

CUSTOMS TARIFF OF CANADA—*cont.*

Alumina or aluminium and alumina and chloride of aluminium or chloralum, sulphate of alumina and alum cake.

Antimony, not ground, pulverised, or otherwise manufactured.

Arsenic.

Argals or arzols, not refined.

Cream of tartar, in crystals.

Borax, ground or unground, in bulk of not less than 25 lb. only.

Boric acid.

Brimstone, crude, or in roll or flower.

Sulphur in roll or flower.

Bromine.

Cherry heat welding compound.

Cinnabar.

Cobalt, ore of.

Ashes, pot and pearl, in packages of not less than 25 lb. weight.

Kainite or German potash salts for fertilisers.

Potash, German mineral.

Potash, muriate and bichromate of, crude.

Soda, sulphate of, crude, known as salt cake.

Soda ash, caustic soda in drums, silicate of soda in crystals or in solution, bichromate of soda, nitrate of soda, or cubic nitre, salsoda, sulphide of sodium, arseniate, binarsenate, chloride and stannate of soda.

Iodine, crude.

Litbarze.

Manganese, oxide of.

Phosphorus.

Sulphate of iron (copperas), and sulphate of copper (blue vitriol).

Ultramarine blue, dry or in pulp.

Verdigris, or sub-acetate of copper, dry.

Whiting or whitening, gilders' whiting and Paris white.

Dragon's blood.

Musk, in pods or in grains.

Oxalic acid.

Quinine, sulphate of, in powder.

Aniline dyes and coal-tar dyes, in bulk or in packages of not less than 1 lb. in weight, including alizarin and artificial alizarin.

Aniline oil, crude.

Aniline salts and arseniate of aniline.

Annato, liquid or solid.

Annato seed.

Barilla.

Berries for dyeing or used for composing dyes.

Cochineal.

Dyeing or tanning articles, in a crude state, used in dyeing or tanning, not elsewhere specified.

Iron liquor, solution of acetate of iron for dyeing and calico printing.

Red liquor, a crude acetate of aluminium, prepared from pyroigneous acid, for dyeing and calico printing.

Extracts of logwood, fustic, oak, and of oak bark.

Indigo.

Indigo auxiliary or zine dust.

Do, paste, and extract of.

Lac dye, crude, seed, button, stick and shell.

Varnish, black and bright, for ships' use.

Cacoutheou, unmanufactured.

India-rubber, unmanufactured.

Rubber, crude, and hard rubber in sheets, but not further manufactured.

Recovered rubber and rubber substitute.

Gutta-percha, crude.

Madder and munjeet, or Indian madder, ground or prepared, and all extracts of.

Persis, or extract of arehail and cudbear.

Terra Japonica, gambier or cutch.

Turmeric.

Chloride of lime.

Colours, metallic, viz.: Oxides of cobalt, zinc, and tin, not elsewhere specified.

Ores of metals of all kinds.

Gypsum, crude (sulphate of lime).

Asphalt and bone pitch, crude only.

Clays.

Fire-clay.

Pipe-clay, unmanufactured.

Fullers' earth.

Cryolite.

Bismuth, metallic, in its natural state.

Copper, in sheets or plates of not less than 4 in. in width.

Precipitate of copper, crude.

Nickel.

Quicksilver.

Silver, German silver, and nickel silver, rolled or in sheets.

Spelter, in blocks and bars.

Tin in blocks, pigs, bars, and sheets, and plates and tinfoil.

Yellow metal, in bolts, bars, and for sheathing.

Zinc, in blocks, pigs, and sheets.

Cinchona bark.

Hemlock bark.

Oak bark.

Tanners' bark.

Cannwood and sumac and extract for dyeing or tanning purposes, when not further manufactured than crushed or ground.

Nut galls.

Litmus and all lichens, prepared and not prepared.

Alkanet root, crude, crushed, or ground.

Root, medicinal, viz.:—aconite, columba, ipæacuanha, sarsaparilla, squills, taraxacum, rhubarb, and valerian.

Gums, viz.:—amber, arabic, Australian, copal, dammar, kaurie, mastic, sandarac, Senegal, shellac; and white shellac in gum or flake, for manufacturing purposes; and gum tragacanth, gum goida, and gum tarberry.

Resin or rosin, in packages of not less than 100 lb.

Turpentine, raw or crude.

Foot grease, being the refuse of cotton seed after the oil has been pressed out, but not when treated with alkalis.

Oil cake and oil-cake meal, cotton-seed cake and cotton-seed meal, and palm-nut cake and meal.

Oils, viz.:—Cocoanut and palm in their natural state; ottar or attar of roses and oil of roses.

Blood albumen, tannic acid, antimony salts, tartar emetic and grey tartar, when imported by manufacturers for use in their factories only.

Chlorate of potash in crystals, when imported for manufacturing purposes only.

Esparto or Spanish grass, and other grasses, and pulp of, for the manufacture of paper.

Platinum wire, and retorts, pans, condensers, tubing, and pipe made of platinum, when imported by manufacturers of sulphuric acid for use in their works in the manufacture or concentration of sulphuric acid.

White shellac, for manufacturing purposes.

—Board of Trade J.

## RUSSIA.

*Chemicals in Russia.*

It is said that the new Russian Customs tariff, which is to come into force in July 1891, and is now being drafted by a commission sitting in St. Petersburg, will be particularly severe on chemical products. The duties on these goods will be advanced in an unprecedented manner, and no loopholes will be left by which it will be possible to import them at lower rates under other denominations.—*Chemist and Druggist.*

## MISCELLANEOUS TRADE NOTICES.

## THE OUTPUT OF PLATINUM.

In view of the recent enormous advance in the price of platinum, the following figures, showing the output of the crude ore in Russia, are of interest:—In 1881 2,986 kilos.; were produced; in 1882, 4,081 kilos.; in 1883, 3,537 kilos.; in 1884, 2,237 kilos.; in 1885, 2,591 kilos.; and in 1886, 4,317 kilos., or an average of hardly 660 cwt. a year. All this platinum is found in two districts of the Government of Perm. The mines of Goroblagodsk yield about two-thirds, those of Nigi-Tagnitsk one-third of the supply. The syndicate which is said to be cornering the platinum supply have, no doubt, in view the enormously growing demand for platinum for electric industries, but the cut-throat policy they are reported to pursue is likely to have the effect of stimulating the working of platinum deposits in other parts of the world, as well as putting electricians on their mettle in finding a substitute for platinum. In the Oural mines, until some years ago, platinum was obtained as a kind of by-product in the working of the auriferous deposits the existence of which led to the opening of these mines. But during the last few years the gold ore has become much scarcer, and platinum has superseded gold as the principal product of the mines. Besides the operations of the syndicate, the recent advance in the metal is attributed partly to the increasing scarcity of the ore and to the fact that labour, always an uncertain factor in the Oural mines, has become excessively scarce since the commencement of the Trans-Siberian railway. According to later advices, the whole of the platinum which will be produced in the Oural mines for the next 10 years has been sold in advance to certain foreign companies. Owing to these contracts the price of platinum has advanced from 2,500 to 12,500 roubles per pood. News comes opportunely from Mexico of the discovery of platinum in the ores extracted from certain mines in Sinaloa, a State famous for its mineral wealth.—*Chemist and Druggist.*

## THE MINERAL WEALTH OF CANADA.

It is fully established that Canada is rich beyond compare in all the more useful minerals, and that she possesses deposits of nickel, silver, and other ores of great extent. The Dominion has coal and iron in close proximity to each other on both her seaboard, and in each instance the fuel and the ores are of the highest qualities. In Nova Scotia there are, within six miles of the Atlantic, huge deposits of fine ironstone alongside pure limestone, and with coking coal in seams 30 feet thick. It is in this part—most probably in Pictou county—that the cheapest and best pig iron in the world will be made at no distant date, and it may be that from the Nova Scotian blast-furnaces, built and run by British capital, we shall at some future date derive a large proportion of our supplies of imported iron. Situated close to the sea, and with proper shipping facilities, these great mineral deposits have a future which is indubitable, and they are sure to attract capital and skill before many years have elapsed. In the Sudbury district the deposits of nickel ore are only just being opened up, but they have a bright future, not only because nickel is likely to be largely used as an alloy with steel, but because nickel itself possesses intrinsic merits which will ensure its free use so soon as it can be sold at reasonable prices and



in quantity. In the Algoma district of Lake Superior there are great deposits of copper ore—stated by Mr. Erastus Wiman to be at least twenty-five times as extensive as those of the Calumet and Hecla district on the American side of the lake. There is also silver in rich lodes and platinum in unusual quantities. Further west, in British Columbia, the mineral deposits are vast and varied. They include gold, silver, lead, iron, and coal, and exist in quantities not yet fully ascertained or appreciated, but known to be exceedingly great. On the Pacific coast, in Puget Sound and elsewhere, the deposits of iron ores and coal are on a tremendous scale, both being very high in quality, and so close together as to render it most easy and economical to carry on blast-furnace work.—*Ironmonger*.

#### THE UNITED ALKALI CO. (LIMITED).

This is the title under which the Chemical Union has been registered by a Liverpool firm of solicitors. The capital of the concern is 6,000,000*l.*, half in preference and half in ordinary shares of 10*l.* each. The first subscribers comprise the following, nearly all well known in the chemical trade:—J. Brock, Widnes; H. Gaskell, Widnes; J. K. Huntley, Flint; C. Wigg, Liverpool; J. H. Dennis, Liverpool; A. W. Allhusen, Gateshead; J. E. Davidson, Newcastle-on-Tyne; G. E. Barlow, Widnes; J. C. Stevenson, M.P., South Shields; J. Tennant, Saltwell, Gateshead; G. L. Wigg, Runcorn; P. J. Worsley, Bristol; J. A. E. Rayner, St. Helen's; E. K. Muspratt, Dale Street, Liverpool; W. J. Menzies, St. Helen's; H. Gaskell, jun., Widnes; R. Shaw, Widnes. The company also possesses in addition to its ordinary directors, one honorary president (Sir Charles Tennant) and four honorary vice-presidents, Sir E. Sullivan, and Messrs. H. Gaskell, J. K. Huntley, and J. H. Dennis. The qualification of each of these officers is holding 5,000*l.* shares in the company. Besides the honorary officers, who are directors *ex officio*, the board is to consist of from 9 to 20 directors, the first being: Messrs. John Brock, Chairman; Chas. Wigg and J. C. Stevenson, M.P., vice-chairmen; Thos. Alexander, Glasgow; A. Allhusen, C. E. Barlow, E. Baxter, J. E. Davidson, H. Gaskell, jun., Jas. Gaskell, W. J. Menzies, E. K. Muspratt, G. Pilkington, J. A. E. Rayner, R. Shaw, James Tennant, G. L. Wigg, and P. J. Worsley. Their qualification is 1,000*l.* The remuneration of the chairman is 2,500*l.*, with a further 500*l.* after 8 per cent. dividend on the ordinary shares; that of each of the other directors is 400*l.*, one half of which shall be paid irrespective of the number of attendances at board meetings, and the remainder to be decided at the end of the year according to the number of their respective attendances. The objects of the company are described as follows:—To carry on business as manufacturers of chemical products and drugs of all kinds and in all branches of such business; as colliery owners and rock-salt proprietors, miners, brine owners, and white salt manufacturers; as dyers, drysalts, and manufacturers of dyes, stains, colours, varnishes, paints, and pigments; as manufacturers of manure, soap, paper-pulp, paper grass, bricks, pottery, terra-cotta, and sanitary and disinfecting preparations, coke, cement, and artificial stone; as waterproofer, and india-rubber and leather manufacturers; as millwrights, makers of locomotive engines, waggons, and rolling stock, stone and limestone quarry proprietors, lime-burners, owners of mines of all descriptions, and winners and workers of minerals and mineral oils, and the business of preparing mineral substances for sale or for treatment in manufacturing processes; as metallurgists in all branches; to manufacture and supply gas to the property of the company or the neighbourhood, and, in connexion therewith, to carry on the business of a gasworks company; to carry on any business directly or indirectly connected with the generation, accumulation, distribution, supply, or application of electricity.

#### EXPORTATION OF CUBAN MANGANESE.

It is announced that during the month of August, 5,200 tons of manganese were exported from St. Jago de Cuba. Owing to the ready sale the manganese meets with in the

United States its production is fast increasing, and the exports for September will be larger than ever before. The owners of the mines have formed a syndicate to work themselves, some American companies which first undertook to work the mines having failed to fulfil their contracts. The exports of manganese ore from 1st July 1889 to 30th June 1890, amounted to 2,240 tons, and from 1st July to 20th August 1890, to 4,190 tons. In other words, the Cuban syndicate exported more in 51 days than American contractors did in one year.—*Chemical Trade Journal*.

#### THE TRADE IN SULPHURIC ACID IN TURKEY.

According to the Belgian *Bulletin du Musée Commercial*, for October 4th, there is at present but a relatively unimportant import of sulphuric acid into Constantinople. In Turkey, as elsewhere, the trade in this article is in direct proportion to the development of the industries which make use of it. The manufacture of mineral waters, the dye-works, the cloth factories belonging to the State, the electrical offices, such as telegraphs, fortifications, marine, and the jewellers' workshops are the principal clients. The annual consumption amounts to about 40,000 kilos, net, the value being about 2,000*l.*

The Custom house administration makes no distinction between nitric acid and sulphuric acid, and returns general figures which make it impossible to give more than a roughly approximate division. The following table gives the amounts of sulphuric and nitric acid imported into Constantinople and into the other parts of the Ottoman Empire in the course of the year 1888—89:—

From	To Constantinople	Import Duties.	Other Parts of the Empire.	Import Duties.
	Kilos.	Piastres.	Kilos.	Piastres.
England .....	..	..	2,186	210
Austro-Hungary .....	3,963	521	13,832	2,116
Italy .....	..	..	58	21
Bulgaria .....	..	..	80	37
Tunis .....	..	..	510	59
Servia .....	..	..	64	14
France .....	88,220	7,193	42,751	4,299
Greece .....	..	..	30	9
	92,222	7,714	59,511	6,765

About four-fifths of the total importation concern sulphuric acid, of which 10,000 kilos are used in the Imperial factories at Stamboul, Makrikeni, and Ismidt. Most of the sulphuric acid brought into Turkey comes from Marseilles, notably from the Rio Tinto Company and from the Saint Gobain factory. A certain important proportion of the consumption is indirectly supplied by England and Germany, through agents in London and Hamburg. For some little time past a house at Antwerp has been sending small quantities of sulphuric acid to Constantinople. The importation by way of Trieste and Galatz, from the Black Sea, is much less than it was.

Steamers are apt to refuse to take large cargoes of this article, as being dangerous. There is therefore serious difficulty in importing it from London and Hamburg, as most of the steam navigation companies now decline to carry inflammable articles. If, however, they do consent to take sulphuric acid on board, it ought to be highly insured, as such articles are thrown overboard at the least threat of danger. The Austro-Hungarian Lloyd has a limit of 15 cases, beyond which it will accept no consignment of this acid. Sailing vessels are much more accommodating and their freight is lower.

## REPORT ON ALKALOIDS.

*Report of the Central Laboratory of the Italian Customs Department, 339—341.*

According to the tariff, alkaloids are distinguished as "quinine salts, and alkaloids not specified and their salts;" both classes pay 12 lire per kilo. These are distinguished from medicinal extracts, which pay 120 lire per quintal, and are defined as follows:—"Medicinal extracts, liquid as well as solid, such as the extracts of aconite, monkshood, juniper, &c. &c., and such other herb, root, flower, wood, and fruit appertaining to pharmacy."

Among substances sent to the laboratory was phenacetine, used medicinally as an antipyretic and antineuralgic; though strictly speaking this substance is not an alkaloid, yet as the tariff report classes antifebrine (acetanilide) among the alkaloids, phenacetine must also be included in the same category. A sample of quinidine was classed as a quinine alkaloid, and one of pyridine as an unspecified alkaloid.

—V. H. V.

## REPORT ON MEDICINAL SUBSTANCES.

*Report of the Central Laboratory of the Italian Customs Department, 349—352.*

Among the substances declared under the category of medicines (duty, 10 lire per quintal, tariff No. 61) were samples of thymol, sawdust soaked with corrosive sublimate, and quinol, which the laboratory decided were rightly classed as medicines "specified and not specified." Their decision on quinol was based upon a verdict given in the case of resorcinol. Another importation declared as earbolic acid was found to consist of the substance known commercially as creoline; this was the subject of a Ministerial decree (February 25, 1889), as follows:—"The Minister, considering that creoline is used as an antiseptic, and as regards its therapeutic properties is to be classified among medicinal substances such as iodol, salol, and other similar products of analogous therapeutic property, decides, in accordance with the Consulting College of Experts, that creoline is to be classed with medicines not specified, and is subject to a duty of 10 lire per quintal. A decree of a similar kind was given (September 4, 1889) concerning the oil of the *Laurus cerasus*, "used exclusively in veterinary practice."—V. H. V.

## REPORT ON MINERALS, METALS, &amp;c.

*Report of the Central Laboratory of the Italian Customs Department, 405—411.*

The tariff classifies, under the generic title of metallic minerals, substances obtained from the surface or interior of the earth which serves as the first materials for the preparation of industrial metals such as iron, zinc, copper, lead, antimony, tin, &c. Such minerals are free of import duty, but on exportation pay 22 lire per ton of lead minerals, 2.20 lire for copper, and 5.50 lire for zinc. Scoriae of metallic minerals are free both of export and import duty.

The report also distinguishes between "natural oxide of manganese" classed with "minerals of all sorts" and "prepared oxide of manganese," which is subject to the same duty as the oxides of iron and lead.

In the former case the oxide is free of duty, in the latter it pays 4 lire per quintal. The Direction point out that though it is not difficult for a practised chemist to distinguish between a natural and a prepared oxide of manganese, yet this problem is beyond a Custom house official. Mention is made of certain disputed samples.

As regards antimony, the report distinguishes between the metal which pays 8 lire per quintal, "antimony sulphide," also called "crude antimony," which pays 4 lire per quintal, and metallic alloys of lead and antimony, and of lead, antimony, and tin classed under the sections 214a and 215a. Disputes have arisen on these points. Again, the report states that "spiegeleisen" occurs sometimes in a faceted form and sometimes in acicular bundles of a various iridescent colour; it should not contain more than 30 per cent. of manganese. Beyond this limit it is to be

classed among metallic alloys not specified. In the latter case it pays 5 lire per quintal for the crude state and 100 lire per quintal for the manufactured articles. The Direction of the laboratory do not agree with the distinction drawn by Ledebur (Die Metallurgie, vol. 290) between spiegeleisen containing less than 25 per cent. of manganese and ferromanganese containing more than 25 per cent.; the former shows a characteristic fracture consisting of regular lamellae and of a silver-white colour, which is made up of small prisms of rhombohedral system, while the fracture of the latter is completely granular. It is pointed out that these distinctions are not valid, and for the decision in case of disputed samples the Direction adopted the process proposed by Volhard, confirming their results by those of Kessler or Pattinson.

Controversies also arose concerning various manufactured articles, especially those which were supposed to contain silver or gold. Thus certain gilded tea-trays were found to be gilded with alloy of tin and lead. Certain spectacles supposed by the Custom house officials to be covered with silver, did not contain this metal but only tin, though other spectacles declared as gilded silver were composed entirely of gold.—V. H. V.

## REPORT ON ANIMAL AND IVORY BLACK.

*Report of the Central Laboratory of the Italian Customs Department, 381—385.*

Under this heading the tariff includes bone-black, lamp-black, and other blacks prepared by the carbonisation of wood, tar, &c.; the first named pays 50 lire, and the last 5 lire per quintal. Residues, however, from sugar refinery, and residues of animal black, are classed as "concime" (refuse), and are then free of duty. According to the instructions it is necessary to distinguish between lamp- and bone-black; the Direction, therefore, determines the amount of ash, the power of absorbing colouring matters, and the amount of volatile matters by calcining in a porcelain crucible in a current of hydrogen (Köhler, Die Fabrikation des Russes und der Schwärze, 102).

## IMPORTATION INTO ITALY OF CHEMICAL PRODUCTS NOT SPECIFIED IN THE CUSTOMS TARIFF.

*Report of the Central Laboratory of the Italian Customs Department, 343—349.*

In this report an account is given of attempted frauds on the Italian Customs, some few of which are selected. A consignment labelled "concime" (refuse), and thus free from duty, was found to consist principally of naphthalene, which is a substance specially mentioned among the unclassified chemicals. A double oxalate of potassium and antimony was introduced as tartar emetic, iodoform as picric acid, silver nitrate as caustic potash, chloral as sodium sulphate, santonine as boric acid, magistery of bismuth as calcium phosphate, reduced iron as antimony sulphide, and ealomal as corrosive sublimate.

Frequently mixtures intended for the manufacture of fireworks were declared among the unspecified chemicals, and not as explosive matters. Examples are given of mixtures of barium nitrate and resin and eye-straw containing some nitro product which is used for the preparation of a certain kind of dynamite.—V. H. V.

## NOTES FROM BAKU.

Chem. Zeit. 14, 1330.

It is reported that a large quantity of platinum ore has been found in the neighbourhood of Werchoturje, near Jekaterinenburg. An increase in the duty on imported soda is expected, and consequently the price of soda in Russia has already experienced a rise. The Minister of Finance has received a deputation representing the Baku naphtha industry, which urged (1) that those Imperial lands that produce naphtha shall be "free" in order to increase the production of the raw material and lower the price; (2), that the rate of freight shall be lowered on the Baku-Batoum railway; (3), that a duty shall be placed on all exported raw naphtha oil.—J. W. L.

## THIRTEENTH MEETING OF THE SOCIETY FOR PROTECTING THE INTERESTS OF GERMAN CHEMICAL INDUSTRY.

(Chem. Ind. 1890, 13, 403—430.)

The chemical industry of Germany, which has been constantly improving for several years, has made further progress during the past year, owing to the fact that the volume of trade was considerably greater than in the preceding year. No great profit was made by the rise in prices which took place in several branches, this being counter-balanced by higher wages and higher prices of materials, especially coal. The number of chemical establishments was 4,809, against 4,464 in the previous year; and 90,585 workpeople were employed, against 84,315, an increase of 7.44 per cent. This corresponds to a total of 27,379,200 working days, against 25,100,024 in 1888; and 71,611,050 marks wages against 65,644,013 in 1888. The average wages increased from 773 to 783 marks. Another indirect proof of the progress of German chemical industry is an increase of 147,000 tons in the imports of raw materials for the production of chemical products; whilst the export of such raw materials was reduced by 29,000 tons. The price of exported chemicals was about 10 per cent. higher than in the preceding year (561 marks per ton against 508 marks), and the increased production was not due to larger export, but to an increased home demand. In 1889 the number of chemical establishments carried on by limited companies was 85, and these, on a capital of 188,203,600 marks, earned 19,922,168 marks in dividends equal to 10.58 per cent. (compare this Journal, 1890, 121).

The average dividends paid since 1882 by limited companies manufacturing chemical products were as follow:—

	Per Cent.
1882 .....	12.82
1883 .....	10.02
1884 .....	7.96
1885 .....	6.37
1886 .....	7.17
1887 .....	8.92
1888 .....	9.78
1889 .....	10.58

In the different industries the results were as follow:—

## ALKALI INDUSTRY.—The average dividends were:—

	Per Cent.
1885 .....	5.86
1886 .....	6.06
1887 .....	6.95
1888 .....	7.71
1889 .....	7.44

The slight decrease in 1889 was due chiefly to a falling off in the export of *Stassfurt salts*. The quantity of kainite obtained at Stassfurt last year was 324,500 tons, against 318,500 tons in the previous year, but the other salts showed a decrease from 916,750 to 861,300 tons. The Stassfurt industry used:—

	Tons.
Carnallite .....	769,947
Kainite .....	69,700
Sylvanite .....	15,883
Schoenite .....	41,917

and manufactured from these:—

	Tons.
Potassium chloride .....	123,748
Potassium sulphate .....	6,221
Calcined potassium—magnesium sulphate .....	9,215
Crystal potassium—magnesium sulphate .....	671
Chemical manures .....	2,238
Calcined kieserite .....	31,824

The *Potassium Industry* showed no progress during last year. This was due, partly to the introduction of smokeless powder, partly to the competition in potash manufacture, and partly to the low prices of potassium chlorate. The *Soda Industry* much improved, although costs were considerably higher, owing to the higher prices of coal and raw

materials; but a large demand stimulated sales, and it is hoped that the English "Chemical Union" will have the effect of steadying the prices in the German soda trade. The use of hydrochloric acid decreased during the last year, more sulphuric acid being used instead. The bleaching powder manufacture was very unfavourably influenced by the collapse of the English convention.

The *COAL-TAR COLOUR INDUSTRY* showed much more progress, as the following average dividends indicate:—

	Per Cent.
1885 .....	7.05
1886 .....	9.94
1887 .....	13.25
1888 .....	15.44
1889 .....	17.50

The higher profits were due chiefly to the increased production. Many new colouring matters, some of great value, were manufactured, and the chemistry of the azo-compounds showed especial progress. This was the case too with alizarine colours, the export of which was higher by 1.7 millions of marks than in the previous year.

*PHARMACEUTICAL, PHOTOGRAPHIC, AND OTHER TECHNICAL CHEMICALS* gave the following average dividends:—

	Per Cent.
1885 .....	10.13
1886 .....	10.60
1887 .....	12.50
1888 .....	8.90
1889 .....	9.00

The diminution in the last two years was caused by higher prices of raw materials and reagents (soda, sulphuric acid, &c.) as well as by a rise in wages and keen competition. Pharmaceutical substances suffered a further disadvantage, in that many new articles manufactured were of little therapeutical value, and disappeared after a short trial, while others were required in such a state of purity that their manufacture could not possibly pay. A considerable increase however in the export of fine chemicals took place, the export of alkaloids alone increasing by 1½ millions of marks. *Alcoholic Substances* are still suffering from the new brandy taxation bill. The manufacture of *Inorganic Colouring Matters* has made good progress in spite of competition. The exports of copper and lead colours, zinc white and zinc sulphide showed an increase, and even the ultramarine trade, which has been in an unsatisfactory state for some time past, improved a little, as the dividends show a rise of from 1.38 to 2.04 per cent., and it is hoped that this progress will continue, as the manufacture is now syndicated, thus putting an end to unsound competition.

The *SOAP INDUSTRY* suffered greatly from the increased prices of raw materials, this increase being due partly to the import of cattle being prohibited. Competition has however been regulated by a number of conventions which were formed in the last two years in different parts of Germany.

The trade in *EXPLOSIVES* showed the following dividends:—

	Per Cent.
1885 .....	10.65
1886 .....	16.09
1887 .....	14.08
1888 .....	15.41
1889 .....	13.00

The reason that last year's dividend was lower than in the three previous years was due to the leading dynamite establishments having formed a so-called "cartel" with several powder mills. The result, however, was unsatisfactory to the dynamite trade; but the trade will probably succeed better in the current year. The trade in *nitro-glycerin explosives* was not bad, there being more competition and also a greater demand than hitherto, and the prices were better than in 1888, while the export was large. In *percussion-cap manufacture* small profits were the rule, owing to sharp competition produced by the opening of three new establishments. The convention for protecting the trade in *blasting powder* was not renewed, and the result was that prices receded considerably, leaving hardly

any profits. The invention of the new smokeless powder, and its introduction into the different armies, had of course a very bad influence upon the trade in ordinary gunpowder. *Gun-Cotton* will probably be only in temporary demand, as the various Governments are anxious to manufacture their own article, and to become as independent as possible of private industry.

The ARTIFICIAL MANURE TRADE showed the following average dividends:—

	Per Cent.
1885 .....	3'14
1886 .....	2'41
1887 .....	5'14
1888 .....	7'97
1889 .....	9'98

This improvement, chiefly due to the splendid results obtained by the experimental stations ("Versuchsstationen") caused a general inquiry for artificial manures and a rise in superphosphate prices; but it is feared that this improvement will not last.

The above-mentioned progress in the different branches of the German chemical industry is further shown by the fact that during the last year nine new limited companies were formed with paid-up shares of 10,100,000 marks, and the capital of ten other companies was increased by a total sum of 7,044,000 marks, whilst four other companies reduced their capital by 1,615,142 marks. The following figures show that notwithstanding the high average dividends of the last year, a considerable number of establishments yielded no profits, or hardly any.

The total number of German chemical establishments was 85.

Twenty-four of these, or 28·23 per cent. of the whole, yielded no profits at all.

Four, or 4·71 per cent., divided 2½ per cent.

Seven, or 8·24 per cent., divided from 2½ to 5 per cent.

Eight, or 9·41 per cent., divided from 5 to 7½ per cent.

Twenty-two, or 25·88 per cent., divided from 7½ to 10 per cent.

Thirteen, or 15·29 per cent., divided from 10 to 15 per cent.

Seven, or 8·24 per cent., divided more than 15 per cent.

Compared with previous years, the progress of the last two years is striking, as the following table shows:—

Year.	No Dividend.	Under 5 per Cent. Dividend.	5 to 10 per Cent. Dividend.	Over 10 per Cent. Dividend.
	Per Cent.	Per Cent.	Per Cent.	Per Cent.
1885	34	22	28	16
1886	38	24	18	20
1887	38	12	30	20
1888	32·5	16·25	26·25	25
1889	28·23	12·95	35·29	23·53

(Compare this Journal, 1890, 121.)

The proposed alterations of the GERMAN PATENT LAW, as published in a bill (see *Reichsanzeiger*, March 17th, 1890), were investigated by a Commission, which reported favourably on its main features. The main features in these alterations, are: Firstly, it has not been thought advisable to give an exact definition of what is called a patentable invention, the decision being left in each case to science and law. The position of chemical inventions in this respect will be seen below. Secondly, for some time past it has been uncertain how far a German patent for a chemical process protected the products manufactured by that process, and inventors suffered considerable loss by chemical products imported which had been manufactured in foreign countries by processes patented in Germany. In 1886, and again in 1888, this importation was declared illegal; but to remove all doubt it is proposed to add a clause to the new Patent Law Bill, to the effect that nobody shall be allowed to import into Germany and sell therein chemical products

manufactured in other countries by processes patented in Germany, unless the special permission of the patentee has been obtained. Further, that *novel* chemical products are to be regarded as having been manufactured by the said processes, unless otherwise proved. Thirdly, the bill also contains the clause that, if a new process is patented, all products manufactured by this process are likewise protected. As this definition is rather vague, and may cause considerable trouble and uncertainty, it is proposed to change the clause to the effect that all products *directly* obtained by patented processes are protected by the patents. Fourthly, by the new bill not only mechanical apparatus, but also the technical use of new chemicals, dyeing materials, &c. will be within the sphere of the patent law. It is therefore hoped that chemical inventions will greatly benefit by the firmer control which will thus be possible. Fifthly, hitherto it has been necessary for the German inventor to prove that a new chemical product has been manufactured by his patented process if he wishes to prevent its import from a foreign country. In many instances this is either extremely difficult or altogether impossible; and it is therefore proposed that in future no such product should be imported, unless it is proved by the importer that it has been manufactured in a different way. Another proposal, with the same object, is to grant patents not only for new chemical processes, but also for such chemical products manufactured by these processes as are entirely new. But as soon as these products are proved to have been manufactured by other methods they may be freely sold anywhere. Also, by this second proposal, German inventors would be well protected against foreign import, without being subjected to such restrictions as might prevent progress. The bill proposed by the Government is however not favourable to either of these proposals, owing to foreign competition being considered too unimportant in this direction. Sixthly, it is proposed that the new patent law should contain a precise definition of what is termed a "special chemical process" as follows:—A special chemical process is the application of one or several combined chemical reactions, in order to obtain a special industrial purpose by certain means or analogous ones which may be substituted for them. It follows from this definition that for every patent application a clearly defined description of the whole process, from beginning to end, is required. Substances or methods which can plainly be substituted for others need not be specially mentioned in the patent specifications. Seventhly, in view of all these points, the Commission recommends the following clause:—If a patent for a special chemical process is granted, the corresponding chemical products shall be regarded as being manufactured by this process, unless otherwise proved.

The bill further intends to define clearly the authority of the Patent Office concerning the granting of patents, as follows:—(a.) If the examination by the Patent Office authorities shows that the invention is not new, the patent must be refused. (b.) If an invention is found to be partially new, the relations of the previous to the subsequent patents have in every case to be defined as clearly as possible, so that the value of the former patents is not affected. (c.) If two patents are found to be totally or partially equal, the Patent Office shall, according to the proposed new bill, be entitled to declare the second totally or partially void, whilst under the existing law no remedy is possible if the second patent has been granted before the publication of the first. (d.) The existing examination of patent applications in the German Patent Office has long been found insufficient, owing to the enormous amount of labour connected therewith. It has often been remarked that it would be much simpler to let time and competition decide the value of patents, many of which are always perfectly valueless. The difficulties in examining the patent applications are especially great in patents referring to chemical industry. But on the other hand the chief object of the Patent Office is not to eliminate valueless applications, but to promote valuable inventions. A reorganisation in this direction which is recommended by the new bill, requires firstly that in future patent applications shall be examined exclusively by especially selected authorities of the central office, and, secondly, that the department for appeals which

is at present united with the application department, shall be entirely separate from it. Thirdly, appeals shall be verbal, if requested by one of the parties. Such verbal discussions would be a most valuable guarantee for the correctness of the final decisions of the Patent Office, especially with reference to chemical processes, if no models, drawings, &c., can be given.

The above-mentioned features of the new Patent Bill will doubtless exert an influence for good upon the chemical industry of Germany; but it cannot be overlooked that the bill also has drawbacks and omissions. The first of these is, there is no sufficient provision for protecting the inventor by granting him patents which had been granted in error to others. Such patents are void, but the real inventor has no right to apply for the patent instead, and this is decidedly unfair. The Government is of opinion that nothing can be done until the introduction of the new Civil Code now in preparation. Secondly, errors in the official examination of patents can never be entirely excluded, even if the examination be made in the most minute and careful way. It is therefore wrong that there should be no appeal against the decision of the Patent Office, if an application is refused by mistake. Thirdly, it is proposed that foreign inventors should not be granted patents or patent rights in Germany, if their countries do not grant the same patent rights to German subjects as to the subjects of other countries. Such a clause must be most decidedly objected to, for while it would have no practical value, it would be sure to produce international friction.

In the further course of the meeting it was strongly urged that Germany should join the international union for the protection of industrial property. The right of priority for six or seven months in case of patent applications in the countries of the union would be a great boon for inventors compared with the present system; and in many cases it would then be very convenient to make a foreign application prior to the German one, owing to the great number of refusals (58 per cent. last year) of the German Patent Office. Germany's joining the union would further be a great advantage for the international regulation of the trade-mark question.—H. S.

### BOARD OF TRADE RETURNS

#### SUMMARY OF IMPORTS.

	Month ended 31st October	
	1889.	1890.
Metals.....	£ 1,735,623	£ 2,073,732
Chemicals and dyestuffs.....	560,897	458,458
Oils.....	753,432	636,067
Raw materials for non-textile industries.....	4,735,798	4,105,988
Total value of all imports ....	38,230,614	37,746,236

#### SUMMARY OF EXPORTS.

	Month ended 31st October	
	1889.	1890.
Metals (other than machinery) ....	£ 4,182,774	£ 4,276,275
Chemicals and medicines .....	745,244	792,073
Miscellaneous articles.....	3,372,850	3,191,234
Total value of all exports.....	23,080,856	23,673,090

#### IMPORTS OF METALS FOR MONTH ENDED 31ST OCTOBER.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Copper:—			£	£
Ore..... Tons	9,014	10,480	75,529	82,700
Regulus..... "	12,364	6,984	333,100	225,936
Unwrought .... "	2,782	5,315	123,663	314,152
Iron and steel:—				
Iron ore..... "	318,572	272,507	245,395	210,772
Iron bolt, bar, &c. "	13,030	8,642	123,153	91,079
Steel, unwrought.. "	1,007	647	10,502	6,975
Lead, pig and sheet ..	11,266	14,916	143,794	208,977
Pyrites..... "	44,581	43,861	78,516	81,998
Quicksilver..... Lb.	70,750	174,360	8,596	23,250
Tin..... Cwt.	30,611	36,417	135,451	181,187
Zinc..... Tons	5,892	7,965	117,873	185,505
Other articles...Value £	..	..	340,051	461,201
Total value of metals	..	..	1,735,623	2,073,732

#### IMPORTS OF RAW MATERIALS FOR NON-TEXTILE INDUSTRIES FOR MONTH ENDED 31ST OCTOBER.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Bark, Peruvian .. Cwt.	6,876	9,333	£ 22,544	£ 24,522
Bristles..... Lb.	315,467	300,742	43,867	41,538
Caoutchouc..... Cwt.	19,615	23,676	213,530	302,421
Gum:—				
Arabic..... "	3,286	2,621	12,334	8,821
Lac, &c..... "	6,380	7,605	24,092	34,790
Gutta-percha .... "	5,417	9,175	82,128	99,267
Hides, raw:—				
Dry..... "	35,914	39,273	97,308	102,264
Wet..... "	57,321	51,992	131,737	117,269
Ivory..... "	1,317	1,489	63,815	77,025
Manures:—				
Guano..... Tons	2,153	891	16,299	7,502
Bones..... "	4,764	3,099	24,137	16,646
Paraffin..... Cwt.	31,046	36,405	38,680	47,222
Linen rags..... Tons	3,333	2,058	35,182	22,647
Esparto, &c. .... "	11,461	14,633	57,901	75,136
Pulp of wood .... "	12,058	7,628	64,187	40,643
Rosin..... Cwt.	43,130	89,145	10,898	21,596
Tallow and stearin ..	111,052	103,105	144,510	133,485
Tar..... Barrels	19,562	21,184	20,320	17,337
Wood:—				
Hewn ..... Loads	283,765	212,054	628,407	481,432
Sawn ..... "	711,539	538,704	1,685,439	1,168,643
Staves ..... "	22,065	12,341	71,974	50,923
Mahogany ..... Tons	5,973	2,525	53,196	25,172
Other articles....Value £	..	..	1,193,313	1,189,787
Total value .....	..	..	4,735,798	4,105,988

Besides the above, drugs to the value of 67,217, were imported, as against 75,636, in October 1889.

## IMPORTS OF CHEMICALS AND DYESTUFFS FOR MONTH ENDED 31ST OCTOBER.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Alkali..... Cwt.	2,996	3,042	£ 2,738	£ 2,444
Bark (tanners, &c.) "	34,500	36,861	10,646	10,580
Brimstone .....	51,341	41,956	11,187	9,220
Chemicals..... Value £	..	..	110,607	146,177
Cochineal .....	704	620	4,413	4,239
Cutch and gambier Tons	1,664	2,236	49,909	38,423
Dyes:—				
Aniline..... Value £	..	..	27,103	26,468
Alizarine .....	..	..	25,969	22,166
Other .....	..	..	1,220	2,598
Indigo .....	1,535	907	22,739	14,186
Madder .....	2,156	1,256	2,316	1,918
Nitrate of soda.... "	254,849	92,246	106,986	38,392
Nitrate of potash. "	38,009	18,772	33,078	17,764
Valonia .....	296	927	3,821	20,431
Other articles... Value £	..	..	149,165	97,149
Total value of chemicals	..	..	560,897	458,458

## IMPORTS OF OILS FOR MONTH ENDED 31ST OCTOBER.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Coccol-nut..... Cwt.	28,344	17,668	£ 38,279	£ 27,965
Olive .....	1,233	872	44,766	34,663
Palm .....	102,024	98,857	113,109	130,764
Petroleum .....	13,855,717	10,150,958	340,235	225,071
Seed .....	1,695	2,565	48,285	67,675
Train, &c..... Tons	2,389	1,368	47,532	27,417
Turpentine .....	33,311	28,557	57,626	42,060
Other articles .. Value £	..	..	63,600	80,152
Total value of oils ...	..	..	753,432	636,067

## EXPORTS OF DRUGS AND CHEMICALS FOR MONTH ENDED 31ST OCTOBER.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Alkali..... Cwt.	580,521	535,872	£ 153,355	£ 191,585
Bleaching materials "	133,630	148,638	48,134	43,811
Chemical manures. Tons	28,374	26,621	175,593	171,772
Medicines..... Value £	..	..	106,204	92,340
Other articles ... "	..	..	261,958	292,558
Total value .....	..	..	745,244	792,073

## EXPORTS OF METALS (OTHER THAN MACHINERY) FOR MONTH ENDED 31ST OCTOBER.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Brass..... Cwt.	10,883	10,567	£ 48,518	£ 46,963
Copper:—				
Unwrought .....	60,200	78,964	156,283	248,511
Wrought..... "	39,142	26,356	110,807	95,455
Mixed metal .... "	41,003	32,662	100,909	97,156
Hardware .....	..	..	278,506	248,525
Implements..... "	..	..	137,209	122,760
Iron and steel.... Tons	420,195	354,970	2,836,646	2,811,512
Lead .....	5,211	4,520	74,676	67,117
Plated wares... Value £	..	..	57,534	44,642
Telegraph wires... "	..	..	205,603	335,064
Tin .....	13,765	9,804	64,888	49,750
Zinc .....	11,906	13,030	10,015	14,203
Other articles .. Value £	..	..	101,030	94,614
Total value .....	..	..	4,182,774	4,276,275

## EXPORTS OF MISCELLANEOUS ARTICLES FOR MONTH ENDED 31ST OCTOBER.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Gunpowder..... Lb.	902,000	994,100	£ 21,932	£ 24,409
Military stores.. Value £	..	..	131,013	155,096
Candles..... Lb.	1,316,500	1,174,200	24,559	23,896
Cautehouse .... Value £	..	..	106,461	117,889
Cement..... Tons	67,874	51,577	133,603	106,288
Products of coal Value £	..	..	100,463	133,156
Earthenware ... "	..	..	207,404	185,976
Stoneware .....	..	..	19,310	11,249
Glass:—				
Plate..... Sq. Ft.	430,486	214,237	29,627	15,150
Flint..... Cwt.	13,023	10,862	32,693	24,833
Bottles..... "	104,900	83,729	50,556	39,381
Other kinds.... "	16,212	17,839	12,241	14,867
Leather:—				
Unwrought .... "	14,221	15,191	128,292	136,628
Wrought ..... Value £	..	..	50,683	43,308
Seed oil..... Tons	6,331	6,451	143,311	149,082
Floor cloth .... Sq. Yds.	1,424,000	1,240,200	70,229	60,650
Painters' materials Val. £	..	..	166,026	140,341
Paper .....	100,396	80,362	179,220	116,538
Rags..... Tons	6,009	3,997	46,385	31,818
Soap .....	50,180	34,154	51,197	41,175
Total value .....	..	..	3,372,850	3,191,284

## Monthly Patent List.

\* The dates given are the dates of the Official Journals in which acceptances of the Complete Specifications are advertised. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the said dates.

### I.—GENERAL PLANT, APPARATUS, AND MACHINERY.

#### APPLICATIONS.

16,679. W. H. Le Mesurier. Improvements in apparatus for refrigerating air, also applicable to the evaporation of liquids. October 20.

16,761. W. P. Thompson.—From A. Mason and J. Wilson, United States. Improvements in and appertaining to effecting and maintaining combustion in secondary chambers of boiler and other furnaces, and in apparatus therefor. Complete Specification. October 21.

16,881. J. F. Pease. Improvements in furnaces. Complete Specification. Filed October 22. Date applied for April 7, 1890, being date of application in United States.

16,924. M. Edgar and W. J. Warnock. Improvements in and relating to furnaces. October 23.

16,935. E. Edwards.—From J. Langheck, Germany. Improvements in furnaces. October 23.

16,940. L. C. Riggs. Improvements in refrigerating apparatus. Complete Specification. October 23.

17,113. W. Bireb. Improved apparatus for filtering sewage and other liquids. October 27.

17,117. D. Rylands. Improved means of manufacturing glass vessels and adapting the same for the storage and conveyance of compressed gases, and for similar purposes. October 27.

17,218. J. Zeller. Process for obtaining a new material from bituminous slate, and its application as a filtering medium, and as a colouring matter. October 28.

17,237. P. Callibures. An improved and completed system of apparatus for evaporation and distillation. Complete Specification. Filed October 28. Date applied for July 15, 1890, being date of application in France.

17,385. J. G. Galley. Improvements in apparatus for burning refuse, calcining ores or other substances, burning cement, and other purposes of the like kind. October 30.

17,446. R. Wood. Improvements in means or apparatus for filtering, more especially applicable in the manufacture of varnish and japan. October 31.

17,702. C. J. Copeland. Improvements in apparatus for feeding fuel to gas producers. November 4.

17,718. H. H. Lake.—From E. Bolton, United States. Improvements in distilling and condensing apparatus. Complete Specification. November 4.

17,815. F. Ellershausen. Improvements in reverberatory furnaces. November 5.

17,820. R. Gubbins. Improvements in retorts. November 5.

17,855. W. Blagg. Improvements in or appertaining to furnaces or heating apparatus for salt pans and other like purposes. November 6.

17,970. J. Donald. Improvements in charring or drying retorts. November 8.

18,050. T. Thorp. A new and improved apparatus for the clarifying of liquids. November 10.

18,099. T. P. Hinde. See Class XVII.

#### COMPLETE SPECIFICATIONS ACCEPTED.\*

1889.

17,938. J. G. Chamberlain and G. E. Davis. Apparatus for evaporation of liquids. November 5.

18,108. J. Ruseoe. Apparatus for charging and drawing retorts mechanically. November 19.

20,784. R. A. Robertson and W. J. Mirrlees. Apparatus for evaporating, concentrating, and distilling liquids. November 5.

20,954. J. Wright. Apparatus for distilling, evaporating, or concentrating liquids, &c. November 5.

1890.

210. T. Thorp. A frictionless metallic gas-pressure gauge. November 12.

398. W. Wild. Apparatus for evaporation of liquids. November 19.

828. A. Smith. Filters. November 19.

1239. D. Bethmont. Apparatus for instantaneous production of vapour from water or other liquids. November 12.

5419. Transferred to XV. and XVIII.—B.

6032. M. W. Dewey. See Class XI.

12,325. J. Jackson. Pressure and vacuum gauges. November 12.

13,084. C. J. Cario. Thermometers and pyrometers. November 19.

14,846. W. H. Kennedy. See Class X.

### II.—FUEL, GAS, AND LIGHT.

#### APPLICATIONS.

16,738. F. J. Jones. An improvement in the manufacture of coke and gas. October 21.

16,760. W. P. Thompson.—From A. Mason, United States. New or improved method of and apparatus for burning coal and hydrocarbon fuel. Complete Specification. October 21.

16,762. W. P. Thompson.—From A. Mason and J. Wilson, United States. Improvements in or relating to burning petroleum and other hydrocarbons, and apparatus connected therewith. Complete Specification. October 21.

16,846. F. E. Ross. Improved method of and apparatus for producing gases from bituminous fuel. October 22.

17,000. J. J. Thomas. Coal-gas generating apparatus. Complete Specification. October 24.

17,030. J. Lyon-Field. An improved manufacture of night-lights. October 24.

17,154. B. F. Field. Improvements in and relating to a process or mode of manufacturing fuel, and in and to the product obtained by said process. Complete Specification. October 27.

17,159. T. G. Springer. Improvements in the manufacture of gas. Complete Specification. October 27.

17,182. G. Love. Improved coke-oven doors. October 28.

17,183. J. F. Craddock. An improved fire-lighter. October 28.

17,201. L. Van Vestrout. Improvements in setting inclined carbonising or gas retorts. October 28.

17,287. W. Smith. Improvements in the manufacture of peat fuel and peat-moss litter. October 29.

17,321. J. Morris. The manufacture of an improved artificial fuel, and of briquettes. October 29.

17,466. H. H. Lake.—From The Standard Coal and Fuel Co., United States. A compound for treating fuel. Complete Specification. October 31.

17,470. H. H. Lake.—From The Standard Coal and Fuel Co., United States. An improved compound for treating fuel. Complete Specification. October 31.

17,484. H. H. Lake.—From The Standard Coal and Fuel Co., United States. An improved compound for treating fuel. Complete Specification. October 31.

17,710. W. Wren. Improvements in or additions to retorts such as are used in the manufacture of coal-gas. November 4.



17,745. T. Cooke. Improvements in the method of setting clay retorts for the manufacture of coal-gas, to be called "The Bristol Retort Setting." November 5.

17,921. C. Eidle. Machine for charging gas retorts. Complete Specification. November 7.

18,047. Unsworth and Richmond, Lim., and W. Unsworth. Improvements in material for use in incandescent gas fires. November 10.

18,115. S. Pointon. Improvements in apparatus connected with the manufacture of gas for heating purposes. November 11.

18,175. A. J. Boulton.—From H. K. Flagler, United States. Improvements in solutions for facilitating the combustion of fuel. November 11.

18,176. A. J. Boulton.—From H. K. Flagler, United States. Improvements in apparatus for burning liquid fuel. November 11.

18,230. L. Van Vestrant and M. Graham. Improvements in the construction of shoots for charging inclined gas retorts. November 12.

18,360. H. Guyon and L. Métai. Improvements in the manufacture of combustible gas for heating, lighting, and generating of motive power. November 14.

18,419. G. B. Falconer. Improvements in or relating to apparatus for charging inclined gas retorts. November 15.

18,457. M. M. Brophy. Improvements in the utilisation of gas for heating purposes, and in apparatus therefor. November 15.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

18,163. F. Pritchard. Enriching and increasing the volume of gas from coal, and utilising the waste heat of retort settings. November 12.

19,721. F. J. Jones. Process and apparatus for the manufacture of illuminating gas. November 5.

19,763. F. Morris. Apparatus for charging inclined gas retorts. October 29.

20,551. J. West. Machines for charging gas retorts. November 5.

1890.

67. T. Parker. Distillation of coal, shale, &c., and production of a special coke. November 19.

343. W. T. Cotton and E. F. B. Crowther. Improvements in the manufacture of gas. November 19.

788. W. W. Horn.—From C. W. Jarrell. Apparatus for washing gas. November 19.

6553. A. Longsdon.—From F. Salomon. Obtaining oxygen gas from atmospheric air. November 12.

13,574. B. H. Thwaite. Method and apparatus for producing combustible gases. November 12.

15,012. W. B. McClure, E. Corning, T. Hodgson, J. E. McWilliams, and J. W. White. Artificial fuel, and manufacture of same. November 12.

#### III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

##### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

18,823. L. Zwillinger. Process and apparatus for production of charcoal, and recovery of acetic acid, wood-spirit, tar, ammonia, and gas. November 19.

1890.

67. T. Parker. See Class II.

1407. M. Lange. Separating the isomers contained in crude nitroalcohol. November 19.

#### IV.—COLOURING MATTERS AND DYES.

##### APPLICATIONS.

16,666. J. R. Geigy. Production of a yellow colouring matter or dye. October 20.

16,868. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik, Germany. The manufacture and production of a new blue basic dyestuff. October 22.

17,168. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik, Germany. The manufacture of sulpho acids of a red basic naphthalene colouring matter. October 27.

17,195. D. Dawson. Improvements in the production of blue colouring matters. October 28.

17,218. J. Zeller. See Class I.

17,538. C. A. Collin. Improvements in obtaining colouring matters. November 1.

17,712. B. Willecox.—From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture and production of new derivatives of alizarine and its analogues. November 4.

18,024. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. Production of a novel pynazon. November 8.

18,025. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. Production of sodium formylphenylhydrazine and of symmetric alkylphenylhydrazine. November 8.

##### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

14,304. A. G. Green and T. A. Lawson. New azo-colouring matters. November 19.

1890.

39. R. J. Friswell. Manufacture of raw materials for dyestuffs. November 5.

134. R. J. Friswell and A. G. Green. Production of azo-colouring matters. November 12.

735. A. Bang.—From Dahl and Co. Production of dioxynaphthalene-monosulpho acid, and of diazo-dyestuffs therefrom. November 19.

1688. T. R. Shillito.—From J. R. Geigy and Co. Producing brown dyes. November 19.

1771. T. R. Shillito.—From J. R. Geigy and Co. Producing a yellow dye. November 19.

14,432. J. Dawson and R. Hirsch. Production of a substantive cotton colour. November 19.

#### V.—TEXTILES, COTTON, WOOL, SILK, Etc.

##### APPLICATIONS.

16,660. J. Menzies and G. Mitchell. A new process for treating textile fabrics and certain manufactured articles, so as to render them sun and heat proof. October 20.

16,985. D. Barnett. Improvements in the treatment of textile vegetable substances to obtain fibres therefrom, and in apparatus therefor. October 24.

17,342. B. Tettweiler. Improvements in and relating to fabrics composed mainly of fibrous material. October 30.

17,469. A. Grupe. A new or improved product suitable for the manufacture of floor cloth and imitation leather goods, and process of preparing the same, partly applicable to the manufacture of linoleum. October 31.

17,531. R. W. Thom. Improvements in backing or back stretching and finishing textile fabrics. November 1.

18,018. A. J. Boulton.—From G. Rudel, Germany. Improvements in the treatment of vegetable materials to convert them into textile fibres. Complete Specification. November 8.

18,119. C. Brodbeck. Improvements in the method or process for silkyfying, or rendering silky tissue threads or textile fibres by utilising every description of waste silk. Complete Specification. November 11.

18,284. P. W. Nicolle and J. Smith. Improvements in the treatment of vegetable fibrous matters to obtain fibres therefrom. November 13.

18,375. C. Campbell and H. D. Shaw. Improvements in scouring or treating wool and other fibrous materials or fabrics, and in means or apparatus employed therein. November 14.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1890.

200. J. Walker and W. Brown. Apparatus for drying or carbonising fibrous substances. November 12.

725. G. Broadbridge.—Partly from M. J. de Sant Anna. An improved fibrous material, and manufacture of same. November 5.

5482. G. W. Arnott, P. A. Olivier, and G. Seagrave. Washing and scouring wool and fibrous materials containing grease. November 19.

15,170. G. Schuler. Manufacture of linoleum, linerusta, eere-cloth, &c. November 19.

### VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

#### APPLICATIONS.

16,751. W. Laidlaw. Improvements in the method of and apparatus for dyeing yarns, tops, and the like. October 21.

17,074. W. H. Clegg and G. Kirby. Improvements relating to the production of wall papers and the like. October 25.

17,326. C. D. Abel.—From M. M. Rotten, Germany. Processes for dyeing textile materials of all kinds in the hyposulphite vat. October 29.

17,642. G. P. Gardner. An improved composition for fixing or mordanting colours, or for padding paper or other like material. November 4.

17,694. E. W. Foxlee. Printing with primuline. November 4.

17,769. T. F. Naylor and A. Naylor. Improvements in the construction of dye vats. November 5.

17,872. T. Salzmann. Improved process for the production of dyed cotton yarns or threads. Complete Specification. November 6.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1890.

2195. I. Braithwaite. Dyeing with indigo. November 12.

13,184. I. F. Peck. Apparatus for dyeing yarn. November 5.

14,897.—W. P. Thompson.—From F. Kornfeld. Dyeing, bleaching, and treating cops of yarn, &c., and apparatus therefor. October 29.

### VII.—ACIDS, ALKALIS, AND SALTS.

#### APPLICATIONS.

16,676. F. Ellershausen. Improvements in the treatment of soda waste. October 20.

16,842. C. Hope. Improvements in the manufacture of sulphuric acid. October 22.

17,150. W. P. Thompson. Improvements in or relating to the manufacture of bicarbonate of soda. October 27.

17,341. W. P. Thompson. Improvements in or relating to the manufacture of bicarbonate of soda. October 30.

17,392. M. Blumrich. Improvements relating to the manufacture of hydrofluosilicic acid and fluoride of silicon. October 30.

17,765. J. Simpson. Improvements in the employment of sulphuretted hydrogen in the manufacture of alkali and certain by-products. November 5.

17,836. S. A. Sadler. Improvements in the process employed in the manufacture of sodium bichromates. November 6.

17,855. W. Blagg. See Class I.

18,152. M. Goldschmidt. Method for producing alkaline nitrites. November 11.

18,187. L. D. Armstrong. Improvements in apparatus for recovering soda. Complete Specification. November 11.

18,240. F. M. Lyte. Improvements in the production of chlorine, and in the manufacture of soft burnt magnesia. November 12.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1890.

858. C. Roth. Manufacture of ammonium nitrate and sulphate, or chloride of sodium and of potassium. November 12.

9793. F. W. A. Frerichs. Manufacture of carbonates of alkalis and acetone. October 29.

14,022. C. Négrier. Furnaces for concentrating sulphuric acid. November 5.

15,544. H. J. Allison.—From M. M. Monsanto. Purifying brines. November 12.

### VIII.—GLASS, POTTERY, AND EARTHENWARE.

#### APPLICATIONS.

16,629. G. L. Schultz. See Class X.

16,901. W. Woodall. An improved combination of electro-coated and enamelled-coated surfaces for salts, dishes, and other similar articles. October 23.

17,968. J. P. Guy. Improvements in machines for pressing and making hollow earthenware articles. October 25.

17,117. D. Rylands. Improved means of manufacturing glass vessels and adapting the same for the storage and conveyance of compressed gases, and for similar purposes. October 27.

17,204. W. W. Pilkington. Improvements in kilns or tunnels for annealing plate glass, and in appliance used in connexion therewith. October 28.

17,255. H. Baggaley. Improvements in the manufacture of tiles and other porcelain, earthenware, and similar goods. October 28.

17,407. T. G. Webb. Improvements in the manufacture of glass retorts as used in chemical works for the concentration of acids, and other analogous purposes. October 31.

17,489. W. T. Croke. An improvement in the manufacture of "black pottery." November 1.

17,505. E. Cable. Improvements in and relating to the decoration of glass. November 1.

17,573. J. Dunnachie. Improvements in kilns for firing firebricks, earthenware, and the like. Complete Specification. November 3.

17,952. A. J. Boulton.—From H. Thumler, Germany. Improvements in the manufacture of glass chains. Complete Specification. November 7.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

17,468. D. Rylands. Manufacturing hollow glass-ware and tubes by machinery. October 29.

19,734. D. Rylands. Manufacture of glass tubes, sheets, and ware. November 5.

20,234. J. J. Candlish and W. Hall. Manufacture of hollow glass-ware, and apparatus therefor. November 12.

20,619. J. G. Sowerby. Cutting glass cylinders, &c. November 5.

1890.

738. C. F. Bailey. Method for supporting pottery-ware whilst being fired. November 12.

8917. C. T. Maling. Manufacture of china or earthenware vessels. November 19.

12,794. G. A. Marsden. Decoration of ceramic ware. November 19.

14,628. J. Sherwin. Devices for supporting earthenware while being fired. November 5.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

### APPLICATIONS.

16,648. H. J. Allison.—From A. Manara and A. Guglielmina, Italy. Improvements in paving stones. October 20.

16,737. J. A. Archer. An improved material for covering roofs, and manufacturing the same. October 21.

16,777. P. C. Biron and C. Roswag. Improvements in the manufacture and production of refractory materials. October 21.

16,789. R. Stobe. Improvements in the manufacture of fireproof plastic material for cement, also applicable to smelting and various other useful purposes. October 21.

17,153. M. Hirsch and H. Reinisch. Improvements in macadam pavement. October 27.

17,795. F. Oschwald. New or improved building material. Complete Specification. Filed November 5. Date applied for July 5, 1890, being date of application in Belgium.

18,459. C. S. Haessler. Improvements in fire and weather-proof roofing. Complete Specification. November 15.

18,468. F. Jurschina and R. Ritter von Gunesch. A new or improved manufacture of artificial stone or composition, and building blocks and other articles thereof. November 15.

### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

19,092. W. S. Akerman. Boilers for utilising the waste heat of cement, &c. kilns. November 5.

1890.

13,384. J. Jungbluth. Composition or concrete flooring. October 29.

15,678. W. A. Robinson and J. Y. Terry. Plastering compositions. November 12.

15,784. G. A. Wright. Brick, block, tile, or slab for building purposes. November 12.

15,900. L. Sterne. Manufacture of cellular non-conducting blocks. November 12.

16,231. P. Crean. Method of, and kiln for, burning bricks. November 19.

## X.—METALLURGY, MINING, Etc.

### APPLICATIONS.

16,629. G. L. Schultz. Extraction of iron from minerals used in manufacture of glass, china, earthenware, and similar goods. October 20.

16,685. P. G. Biron. Improvements in treating cupreous materials, and obtaining copper and other useful products therefrom. October 20.

16,694. J. Harris. Improvements in machinery or apparatus to be used for the extraction of gold and silver from their ores by the amalgamation process. October 20.

16,724. J. B. Renshaw. An improvement in the manufacture of cast iron. Complete Specification. October 21.

16,802. E. Herz. Improvements in cupola and such like furnaces. Complete Specification. October 21.

16,887. T. S. Lindsay. Improvements in the preparation of aluminium fluoride and double fluorides, and in the manufacture of aluminium from fluorides. October 23.

17,036. J. C. Mitchell. An improved method of extracting metals from their ores, and appliances connected therewith. October 25.

17,953. W. Truran. Improvements in machinery or apparatus for cooling and discharging slag. October 25.

17,981. J. Harper and Co., Lim., and H. Devonport. Improvements in annealing furnaces. October 25.

17,165. H. Lobausen. A process for producing copper blocks with steel core for the manufacture of telegraph and telephone wire. October 27.

17,173. J. C. Bull. Improvements in refining copper. October 27.

17,186. S. B. Evans. A gas ingot-heating furnace, with improvements for iron or steel works. October 28.

17,191. F. H. Varley. Improvements in smelting furnaces. October 28.

17,279. The Manchester Oxygen (Brin's Patent) Co., Lim., and B. H. Thwaite. Improvements in the manufacture of steel and iron, and in apparatus therefor. October 29.

17,380. J. von Langer and L. Cooper. Improvements in or connected with reverberatory furnaces for puddling or smelting iron, and for other purposes. October 30.

17,613. P. M. Justice.—From J. Meyer, Germany. Improvements in the manufacture of steel and ingot iron. Complete Specification. November 3.

17,621. H. Grafton. An improved process of case-hardening or steelifying. November 3.

17,623. J. Garnier. Process for the manufacture of metallic nickel, together with alloys of nickel and iron, utilisable either direct or for the manufacture of nickel steel or nickel and carbonised iron castings. November 3.

17,625. B. Richards. Improvements in furnaces for smelting copper and other ores. November 3.

17,663. E. P. Martin and E. James. An improved method of and means for preparing moulds for casting pig iron, and for breaking the pigs and separating them from the "sow." November 4.

17,935. N. Lébédoff. Improvements in and means or apparatus for the extraction of metals from metallic ores or matters containing metals. November 7.

18,066. Davies Bros. and Co., Lim., and M. Bayliss. Improvements in and connected with the galvanising of sheet metal. November 10.

18,080. C. Hopfner. An improved method of treating cupreous liquors for the purpose of utilising the same. November 10.

18,171. W. P. Thompson.—From M. F. Coomes and A. W. Hyde, United States. Improvements in or appertaining to the manufacture and tempering of steel. Complete Specification. November 11.

18,235. W. D. Bohm. Improvements in the separation of gold and silver from ores or materials containing them. November 12.

18,276. J. Riley. Improvements in and connected with ingot moulds, such as are used in the manufacture of steel. November 13.

### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

19,148. J. C. Bull. Manufacture of metallic alloys and compounds. November 5.

19,632. T. H. Johns. Manufacture of tin andterne plates, and apparatus therefor. October 29.

19,653. T. H. Johns. Means and apparatus for coating and finishing sheet iron. November 12.

1890.

877. D. Elliott. Furnaces for heating iron or steel. November 19.

13,259. C. T. J. Vautin. Apparatus for extraction of gold. November 5.

14,837. C. E. Bernard. Production of dissolving and reducing agent, and employing same for coating metals with metals or combinations with metalloids, extracting metals from ores or combinations, or in arts in which dissolving or reducing action is required. November 5.

14,846. W. H. Kennedy. Method and apparatus for producing mineral wool. November 5.

15,833. E. Tweedy. A tempering fluid for treating steel. November 19.

15,859. J. L. Hopper. Process and composition for softening and subduing refractory ores. November 19.

## XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

### APPLICATIONS.

16,747. J. R. Thame. Improvements in the manufacture of electrical insulating materials. October 21.

16,837. J. E. Douglass. Improvements in the manufacture, construction, and building up of secondary battery elements, electrodes, or plates. October 22.

16,844. H. Weymersch. An improvement in the construction of accumulators or secondary batteries. October 22.

16,934. M. Azapis. A new electric battery. October 23.

17,277. R. Wotherspoon.—From H. Rowley, Australia. An improved method of and apparatus for the electrical treatment of sewage, wines, oils, air, gases, and other fluids. October 29.

17,444. M. Muthel. An improved filling for galvanic batteries. October 31.

17,954. H. L. Maugras. Improvements in electrical accumulators or storage batteries. November 7.

18,096. V. Baron von Alten. Improvements in galvanic elements. November 10.

18,180. Sir C. S. Forbes, Bart. Improvements in primary batteries, and in apparatus connected therewith. November 11.

18,248. J. A. Mays. Improvements in electrolysis. November 12.

18,339. W. R. Lake.—From A. Dehuard, France. An improved process and apparatus for the production of oxygen and hydrogen by electrolysis. November 14.

### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

20,856. D. G. FitzGerald and A. H. Hough. Elements for voltaic batteries. October 29.

1890.

232. J. Hopkinson, E. Hopkinson, and G. A. Grindle. Electric motors and dynamo-electric machines. October 29.

761. O. Dahl. Generating electricity by gas batteries. November 12.

6032. M. W. Dewey. Electric heating apparatus. October 29.

8242. W. R. Lake.—From H. Lemp. Electric welding apparatus. October 29.

12,830. H. H. Lake.—From G. A. Johnson and S. L. Holdrege. Secondary batteries and electrodes therefor. October 29.

13,421. W. P. Thompson.—From C. L. Coffin. Method and apparatus for working metals electrically. October 29.

15,456. W. P. Thompson.—From C. L. Coffin. Welding metals electrically. November 5.

15,482. H. H. Lake.—From The Crosby Electric Co. Electric batteries. November 5.

16,322. S. C. C. Currie. Insulating cells for electric batteries. November 19.

16,415. M. W. Dewey. Method and apparatus for utilising electricity in the formation of sheet metal articles. November 19.

## XII.—FATS, OILS, AND SOAP MANUFACTURE.

### APPLICATIONS.

17,160. E. Hermite, E. J. Paterson, and C. F. Cooper. A process for bleaching palm oil and other vegetable oils. October 27.

17,275. C. R. Illingworth. Improvements in soaps for diseases of the skin. October 29.

17,624. C. Moritt. Improvements in the treatment of linseed oil. November 3.

17,792. E. L. Downing and S. Hern. Improvements in cleansing or washing compositions. November 5.

18,031. A. Gutensohn. An improved compound for use in lieu of palm oil. November 8.

### COMPLETE SPECIFICATIONS ACCEPTED.

1890.

4137. F. Piekenbroek. Producing ozonised oils. November 12.

13,803. C. A. Jensen.—From P. Möller. Manufacture of cod-liver oil. November 12.

## XIII.—PAINTS, PIGMENTS, VARNISHES, AND RESINS.

### APPLICATIONS.

16,654. S. Banner. Improvements in compositions applicable in the manufacture of oilcloth, varnish, pigment, menstrua, cement, binding material for so-called asphalt for giving body to oil, and for other like purposes, and method of manufacturing the same. October 20.

16,757. T. W. Just, R. Weiler, and O. Heidepriem. An improved ink. Complete Specification. October 21.

16,786. W. A. Hall. Improvements in the manufacture of paints. Complete Specification. October 21.

16,916. T. Drake. A new turpentine, and means of producing same. October 23.

17,197. C. A. Fawsitt. Improvements in compounds to be used in vulcanising rubber and other substances or compounds. October 28.

17,313. A. O. Watkins. Improvements in the production of uranium yellow, or compound oxides of uranium. October 29.

17,346. J. Haake. Improvements in the preparation of colours for distempering. Complete Specification. October 30.

17,446. R. Wood. Improvements in means or apparatus for filtering, more especially applicable in the manufacture of varnish and japan. October 31.

17,597. G. Chapman. Improvements in the method of manufacturing white lead. November 3.

17,839. J. MacHale. Anti-fouling paint. November 6.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

20,830. C. Huelser.—From G. Callmann. Process and composition for producing printing ink. November 12.

1890.

16,017. W. Dyshko. Transferred to Class XIV.

## XIV.—TANNING, LEATHER, GLUE, AND SIZE.

## APPLICATIONS.

16,793. J. Patterson. Improvements in fertilising compounds or fertilisers. Complete Specification. October 21.

17,853. T. O. Butler. An improved glue compound. Complete Specification. November 6.

18,385. L. A. Groth. Improved process and apparatus for tanning hides and skins. November 14.

## COMPLETE SPECIFICATION ACCEPTED.

1890.

16,017. W. Dyshko. Preparing and waterproofing skins. November 19.

## XV.—AGRICULTURE AND MANURES.

## COMPLETE SPECIFICATION ACCEPTED.

1890.

5419. J. Guillaume. Treatment of organic matters for production of manure, and apparatus therefor. October 29.

## XVI.—SUGARS, STARCHES, GUMS, Etc.

## APPLICATIONS.

17,232. H. H. Leigh.—From F. T. Romiguières, France. Improvements in refining sugar. October 28.

17,557. A. Wohl and A. Kollrepp. Improvements in the production of invert sugar. November 1.

## XVII.—BREWING, WINES, SPIRITS, Etc.

## APPLICATIONS.

16,746. E. D. Alexander. Moss beer, the ideal drink for invalids, dyspeptics, and ladies nursing. October 21.

16,942. F. T. Simson and J. Cox. Kola or caffenin, or theine malt liquors, fermented wines, and spirits. October 23.

16,951. H. Bates, jun. Improvements in corn product for brewers' use, and in the manufacture thereof. Complete Specification. October 23.

17,329. J. Rose. Brewing oatmeal stout and porter. October 29.

17,640. R. H. Leaker and J. H. Howell. Improvements relating to brewers' and distillers' fermenting vessels and yeast receivers. November 4.

18,099. T. P. Hinde. The boiling of malt and other liquors by coal and coke fire, gas, or other refined heat most applicable. November 10.

18,123. A. G. Hopkins. Charging fermented liquids in cask with carbonic acid gas, and for introducing carbonic acid gas into casks on draught, in lieu of atmospheric air. November 11.

18,212. E. J. Mills and R. Barr. Improvements in the treatment of alcoholic liquids. Complete Specification. November 12.

18,340. F. S. Willoughby and A. Mudie. Improved method of maturing spirits. November 14.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

20,434. B. Hoff and J. Frommel. Apparatus for rectifying spirits, &c. October 29.

20,630. L. Haas. Apparatus for collecting for use the excess of carbonic acid generated during brewing. November 12.

1890.

667. W. P. Thompson.—From J. A. H. Hasbrouck. Process and apparatus for ageing liquors. November 19.

678. E. Edwards.—From F. Hofmeister. Method and apparatus for obtaining accelerated fermentation and producing sparkling drinks. October 29.

3606. E. Carez and La Société Générale de Maltose. Saccharification of amylaceous substances. November 19.

## XVIII.—CHEMISTRY OF FOODS, SANITARY CHEMISTRY, AND DISINFECTANTS.

## APPLICATIONS.

*A.—Chemistry of Foods.*

16,713. S. J. P. Pellatt. Improvements in the method of treating and preparing hops, and blending the same for infusion together with tea, coffee, cocoa, and other substances for which such prepared hops are applicable. October 21.

16,828. T. Eleoate. An improved method of preserving uncooked meats. October 22.

16,857. M. A. E. Schneider and R. H. Coall. The treatment of a certain fruit in order to convert it, either alone or combined with other substances, into food and drink for infants, invalids, and others. October 22.

17,104. L. Bremond. Electrically destroying insects and parasites, and preserving animal and vegetable foods and drinks. October 25.

17,245. B. Wilcox.—From The Rumford Chemical Works, United States. Improvements in baking powders and like preparations. Complete Specification. October 28.

17,427. B. G. Hadnut. New or improved corn products, and process for manufacturing the same. Complete Specification. Filed October 31. Date applied for April 5, 1890, being date of application in United States.

18,368. W. Paterson. The manufacture of a new or improved sweet or sweet food, or preserve. November 14.

*B.—Sanitary Chemistry.*

16,728. G. E. Davis and A. R. Davis. The treatment of sewage, and utilisation of the by-products. October 21.

16,836. J. Lowe. Precipitating and filtering sewage. October 22.

16,972. F. P. Candy. Improvements in and in the preparation of material for use in the treatment of sewage and other polluted water and liquids. Complete Specification. October 24.

17,113. W. Birch. See Class I.

17,453. J. W. Slater and The Native Guano Co., Lim. Improvements in the preparation of agents for use in the purification of sewage and other polluted water by precipitation, and improvements in the treatment of sewage and other polluted water, in order to effect the purification thereof. October 31.

17,686. E. W. Cracknell. An improved furnace for the incineration and destruction of house garbage, town refuse, and such like noxious matters. Complete Specification. November 4.

17,687. E. W. Cracknell. Improvements in the treatment of night soil and such like noxious matters, and in apparatus therefor. Complete Specification. November 4.

*C.—Disinfectants.*

17,943. J. A. Phillips. Improvements in disinfectants and deodorisers. November 7.

## COMPLETE SPECIFICATIONS ACCEPTED.

*A.—Chemistry of Foods.*

1889.

19,281. E. Edwards.—From A. Seidensticker. Method and apparatus for preserving milk. November 12.

1890.

14,888. G. F. Meyer. Method and apparatus for extracting salt from victuals by osmose. November 12.

*B.—Sanitary Chemistry.*

1889.

18,291. Destructors for combustion of refuse, and disposing of or utilising the products of combustion therefrom. November 12.

1890.

472. C. Jones. Destructors for treating refuse. November 19.

480. E. Devonshire. Purifying water, and apparatus therefor. November 19.

5419. J. Guillaume. See Class XV.

7840. W. C. Young. Purification of water by distillation. November 12.

*C.—Disinfectants.*

1889.

18,512. J. Price. Disinfectants and deodorisers. October 29.

20,231. A. Artmann and H. W. Kufcke. Producing disinfectants. November 5.

## XIX.—PAPER, PASTEBOARD, Etc.

## APPLICATIONS.

16,910. C. R. F. Schloesser and J. M. Campbell. Improvements in the manufacture of paper and in apparatus therefor. October 23.

16,928. H. B. Watson and J. S. Watson. Improvements in the manufacture of strainer or knotted plates for straining paper pulp, wood pulp, and the like. October 23.

17,012. G. L. Anders and C. H. Elliott. Improvements in the use and application of xylonite, celluloid, and the like. Complete Specification. October 24.

17,268. A. Wilbaux. Improvements in or relating to the treatment of vegetable parchment and the like. Complete Specification. October 28.

18,169. W. P. Thompson.—From P. R. Thom, United States. Improvements in apparatus for straining or screening paper pulp. November 11.

18,215. R. E. Fraser. The improvement of paper for the use of water-colour artists and others. November 12.

## COMPLETE SPECIFICATIONS ACCEPTED.

1890.

795. R. Stone. Treatment and utilisation of peat, sawdust, and other fibrous and vegetable materials. November 12.

15,995. F. V. L. Hiorth. Apparatus for boiling wood pulp. November 12.

## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES, AND EXTRACTS.

## APPLICATIONS.

17,221. C. Kolbe. Improvements in the manufacture or production of salols. October 28.

17,547. G. de Laire. A manufacture of iso-engenol and of certain of its derivatives and their application to the manufacture of vanilline. November 1.

18,021. B. Willeox.—From The Farbenfabriken vormals F. Bayer and Co. Improvements in the manufacture of periodides of isobutyl phenols, and of isobutyl cresols. November 8.

18,022. B. Willeox.—From The Farbenfabriken vormals F. Bayer and Co. Improvements in the manufacture of methyl- and ethyl-phenacetin. November 8.

18,246. O. Imray.—From The Amsterdamsche Chinine-fabrik, Holland. Process for the production of anthranilic acid. November 12.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

20,392. J. A. Kendall. Manufacture of hypophosphites. November 5.

1890.

5940. H. H. Lake.—From La Société Anonyme des Parfums Naturels de Cannes. Method and apparatus for extracting and purifying perfumes, oils, &c. November 12.

## XXI.—PHOTOGRAPHIC PROCESSES AND MATERIALS.

## APPLICATIONS.

17,189. J. E. Thornton. Improvements in connection with flexible films for photographic and other purposes, and in the manufacture, and in apparatus used in the manufacture of the same. October 28.

17,339. R. Fowler. Improvements in and appertaining to cameras and sensitive plates for photographic purposes. October 30.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

17,773. E. H. Farmer. Improvements in photography. October 29.

1890.

401. T. H. Redwood. Means or apparatus for producing flash light for photography. November 12.

## XXII.—EXPLOSIVES, MATCHES, Etc.

## APPLICATION.

18,472. G. Trench. Improvements in explosives. Nov. 15.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

17,430. A. Walter. Percussion and time fuses. Nov. 5.

18,738. A. Martin. Percussion fuses. November 19.

19,935. A. T. Cocking. Treating and using explosives so as to control and quench the flame. November 12.

19,971. A. Martin. Percussion fuses. November 19.

20,104. The Roburite Explosives Co., Lim., C. Roth, and W. J. Orsman. Treatment or preparation of nitrate of ammonium. November 12.

# THE JOURNAL

OF THE

# Society of Chemical Industry:

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Members who require extra sets or back numbers of the Journal are requested to make application to the General Secretary only, to whom also changes of address should be communicated.



Authors of communications read before the Society, or any of its Local Sections, are requested to take notice that under Rule 41 of the bye-laws, the Society has the right of priority of publication for three months of all such papers. Infringement of this bye-law renders papers liable to be rejected by the Publication Committee, or ordered to be abstracted for the Journal, in which case no reprints can be furnished to the author.

Notice is hereby given, for the information of members and advertisers, that the advertisement columns of this Journal have been contracted for by Messrs. EYRE and SPOTTISWOODE, the Society's printers and publishers, to whom all communications respecting them should be addressed.

The Secretary is instructed to negotiate for the purchase of copies of the Society's Journal for January and May 1883, and January, February, and April 1886. Members possessing odd copies of these numbers are particularly requested to communicate at once, stating price required, with Mr. Cresswell. The stock of all other numbers is at present sufficient for the Council's requirements.

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Fielding, A., 1/o Dantzic Street; George Street, Salford, Manchester.

Findlay, J. T. J., Journals to c/o Messrs. Chapman and Messel, Silvertown, E.

Gray, G. Watson, 1/o 12; 14, Argyle Road, Garston.

Harvey, E. W., 1/o Clapham Common; Hilsrig, Alderbrook Road, Balham, S.W.

Hay, Alex., 1/o Lincoln; Heath Villa, Shaftesbury Avenue, Bristol.

Hedley, Jno., jun., 1/o Fenchurch Street; 11, Crooked Lane, Cannon Street, London, E.C.

Kitamura, Y., 1/o Berlin; 72, Camden Road, London, N.W.

Kleeman, Dr. S., 1/o Manchester; 52, Mill Lane, Old Swan, Liverpool.

Lagerwall, Dr. Ivar, 1/o Brussels; c/o Nobel Bros., St. Petersburg.

Orr, Alex., 1/o Sydney; 1, Park Terrace, Irvine, N.B.

Pugh, Geo., 1/o Mark Lane; 71, Milson Road, West Kensington Park, W.

Reid, W. G., Journals to c/o Reid and Mackay, Old De Beers, Kimberley, South Africa.

Roberts, F. G. Adair, 1/o Bethune Road; Lion House, Amburst Park, Stamford Hill, N.

Stahl, Dr. Karl F., 1/o Johnstown; 57th Street and A.V.R.R., Pittsburgh, Pa., U.S.A.

Thomson, J. S., Journals to Uphall Oilworks, Uphall Station, N.B.

Tyrer, Thos., 1/o Battersea; Stirling Chemical Works, Abbey Lane, Stratford, E.

Warne, Thos., 1/o Wallsend; 118, Wednesbury Road, Walsall.

Wood, E. T., 1/o Malvern; Brinseall, Chorley, Lancashire.

Wright, Alf., 1/o Newcastle; 12, Richmond Villas, Seven Sisters Road, Holloway, N.

Wuth, Dr. A., 1/o Dambachthal; 37, Kapellerstrasse, Wiesbaden, Germany.

#### Deaths.

W. Lant Carpenter, 36, Craven Park, Harlesden, N.W.

Charles Marvin, Grosvenor House, Plumstead Common, Kent.

#### CHANGES OF ADDRESS.

Beckett, G. H., Journals to 133, Bewsey Road, Warrington.

Bloxam, A. G., 1/o Cirencester; 16, Bolingbroke Road, West Kensington, W.

## London Section.

### CHEMICAL SOCIETY'S ROOMS, BURLINGTON HOUSE.

Chairman: T. Tyrer.

#### Committee:

W. Crowder.	B. E. R. Newlands.
J. Dewar.	B. Redwood.
A. G. Green.	John Spiller.
S. Hall.	W. S. Squire.
C. W. Heaton.	Wm. Thorp.
D. Howard.	T. E. Thorpe.
C. C. Hutchinson.	C. R. Alder Wright.
R. Messel.	

Hon. Local Sec. and Treasurer:

T. W. B. Mumford, 1, Glendale Villas, Sylvan Road, Wanstead, E.

#### SESSION 1890—91.

1891.

Jan. 5th.—Mr. Percy C. Gilchrist. "The Basic Copper Process."

Feb. 2nd.—Mr. W. C. Young. "On Standard Sperm Candles."

Feb. 16th:—

Mr. W. Macken. "Incandescent Gas Lighting."

Messrs. Cross and Bevan. "The Volumetric Estimation of Alumina."

March 2nd.—Mr. V. H. Veley. "The Chemical Changes between Nitric Acid and Metals."

March 16th:—

Mr. W. Crowder. "Observations made in the Working of Vitrified Chambers."

Mr. A. H. Allen. "The Chemistry of Whisky and Allied Products."

April 6th.—Professor Vivian B. Lewes:—

1. "The Analysis of Illuminating Gases."

2. "The Products of Checked Combustion."

April 20th.—Mr. W. P. Rix. "Stoneware: and its Application to Chemical Apparatus."

May 4th.—Professor Dewar, F.R.S., and Mr. Boverton Redwood, F.R.S.E. "A Process for the Conversion of Heavy Mineral Oils into Lighter Hydrocarbons suitable for Illuminating and other purposes."

June 1st.—Dr. W. S. Squire. "The Artificial Production of Cold theoretically and practically explained."

Meeting held Monday, December 1st, 1890.

MR. THOS. TYRER IN THE CHAIR.

### PURIFICATION OF SEWAGE AND CONTAMINATED WATER.

BY WM. WEBSTER.

THE term sewage many years ago was rightly applied to the excremental refuse of towns, but it is a most difficult matter to define the liquid that teems into our rivers under the name of sewage to-day; in most towns "chemical refuse" is the best name for the complex fluid running from the sewers.

It is now more than 10 years since I first commenced a series of experiments with a view of thoroughly testing various methods of purifying sewage and water contaminated with putrefying organic matter. It was while investigating the action of iron salts upon organic matter in solution and splitting up the chlorides present by means of electrolysis, that I first became aware of the importance of precipitating the soluble organic matter in such manner that no chemical solution should take the place of the precipitated organic matter. If chemical matter is substituted for the organic compounds, the cure is worse than the disease, as the resulting solution in most cases sets up after precipitation in the river into which it flows.

My first electrolytical experiments were conducted with non-oxidisable plates of platinum and carbon, but the cost of the first and the impossibility of obtaining carbon plates that would stand long-continued action of nascent chlorine and oxygen made it desirable that some modification should be tried. I next tried the effect of electrolytic action when iron salts were present, but did not think of using iron electrodes until after trying aluminium. I found that the action of non-oxidisable electrodes was most efficacious after the temperature of the fluid acted upon rose 4° or

5°; but the cost of working made it impossible on a large scale.

After a long series of experiments, iron plates were used as electrodes with remarkable results, for the compounds of iron formed not only deodorised the samples of sewage acted on, but produced complete precipitation of the matters in suspension, and also of the soluble organic matter; the resulting effluents remaining perfectly free from putrefaction. The first part of the process is well illustrated by the small experiments now shown; the organic matter in suspension and in solution separates into flocculent particles, which rise to the top of the liquid and remain until the bubbles of hydrogen which have carried them up escape, when the solid matter will precipitate. In the arrangement adopted on a working scale, the separated particles precipitate readily. As an illustration of the action upon organic matter in solution I take a small quantity of dye, mix it with water, and placing the connected iron electrodes in the mixture, the dye in solution separates into flocculent particles. The electrolytical action is of course easily understood, but the chemical changes that take place need an explanation. At the positive pole, hypochlorite of iron seems to be formed at first, but this is quickly changed into a protochloride, and as at the negative pole an alkaline reaction takes place, the iron salt is precipitated in the form of the ferrous hydrated oxide, together with the organic matters in suspension and solution. Owing to the carbonates that are always present in sewage, ferrous carbonate is also formed.

The success of these laboratory experiments led to a trial of the process on a larger scale, for hitherto only a gallon at any one time had been treated.

Small brick tanks were erected at my wharf at Peckham and iron electrodes fitted to them.

Wrought-iron plates were fixed about an inch apart, and connected in parallel in the tanks, forming one big cell. Sewage to the amount of about 200 gallons was run into the electrode tank and then treated, the results being so satisfactory that larger works were erected, when a supply of sewage equal to 20,000 gallons an hour could be obtained.

After a number of experiments had been carried out it was decided to run the sewage as rapidly as possible through electrodes, six cells or two rows in series fixed in a long channel or shoot, for experience showed that the motion of the liquid acted on reduced the back E.M.F. and hastened the formation of the precipitate.

A channel is kept at the bottom of the electrodes for the silt to collect, with a culvert at side to flush it into, so as to prevent any block occurring; the advantage of this is obvious. The plates in each section may be from half an inch to an inch thick, and can be of any length up to 6 ft. It may possibly be objected that a large number of plates is required. This may be so, but the larger the number of plates the less the engine power required, and the longer they last. In each section the electrodes are in parallel, and any one section is in series with the other, the arrangement being exactly alike that of a series of primary battery cells.

By actual experience I have been able to prove that at least 25 sections of electrodes should be in series, and across any one of these sections the potential difference need not be greater than 1·8 volts, the current being of any desired amount according to the surface of plates used.

The electrical measurements taken by Dr. John Hopkinson during these experiments for the Electrical Purification Association, to whom I had sold my patents, entirely corroborated my contentions as to E.H.P. used, and agreed with the measurements of the managing electrician, Mr. Octavius March.

The process was then thoroughly investigated by Sir Henry Rosevear, who had control of the works for one month. He reports as follows:—

"The reduction of organic matter in solution is the crucial test of the value of a purifying agent, for unless the organic matter is reduced the effluent will putrefy and rapidly become offensive.

"I have not observed in any of the unfiltered effluents from this process which I have examined any signs of putrefaction, but, on the contrary, a tendency to oxidise.

The absence of sulphuretted hydrogen in samples of unfiltered effluent, which have been kept for about six weeks in stoppered bottles is also a fact of importance. The settled sewage was not in this condition, as it rapidly underwent putrefaction, even in contact with air, in two or three days.

"The results of this chemical investigation show that the chief advantages of this system of purification are:

"First.—The active agent, hydrated ferrous oxide, is prepared within the sewage itself as a flocculent precipitate. (It is scarcely necessary to add that the inorganic salts in solution are not increased, as in the case where chemicals in solution are added to the sewage.) Not only does it act as a mechanical precipitant, but it possesses the property of combining chemically with some of the soluble organic matter and carrying it down in an insoluble form.

"Second.—Hydrated ferrous oxide is a deodoriser.

"Third.—By this process the soluble organic matter is reduced to a condition favourable to the further and complete purification by natural agencies.

"Fourth.—The effluent is not liable to secondary putrefaction."

Mr. Alfred E. Fletcher also investigated the process subsequently, and reports as follows:—

"The treatment causes a reduction in the oxidisable matter in the sewage, varying from 60 to 80 per cent. The practical result of the process is a very rapid and complete clarification of the sewage which enables the sludge to separate freely.

"It was noticed that while the raw sewage filters very slowly, so that 500 cc. required 96 hours to pass through a paper filter, the electrically treated sewage settled well and filtered rapidly.

"Samples of the raw sewage having but little smell when fresh, stank strongly on the third day. The treated samples, however, had no smell originally, and remain sweet, without putrefactive change.

"In producing this result two agencies are at work, there is the action of electrolysis and the formation of a hydrated oxide of iron. It is not possible, perhaps, to define the exact action, but as the formation of an iron oxide is part of it, it seemed desirable to ascertain whether the simple addition of a salt of iron with lime sufficient to neutralise the acid of the salt would produce results similar to those attained by Webster's process.

"In order to make these experiments, samples of fresh raw sewage were taken at Crossness at intervals of one hour during the day. As much as 10 grains of different salts of iron were added per gallon, plus 15.7 grains of lime in some cases, and 125 grains of lime in another, and the treated sewage was allowed to settle 24 hours; the results obtained were not nearly as good as by the electrical method."

During the present year a very searching investigation of the merits of various processes of sewage treatment has been made by the Corporation of Salford; amongst others of my electrical process. As the matter is at present under discussion by the Council, I am not in a position to give extracts from the reports of the engineers and chemists under whose supervision and control the work was done, but I may go so far as to say that the results of my system of electrical treatment have proved its efficiency and applicability to sewages of even such a foul nature as that of Salford and Pendleton. The system was controlled continuously for the Corporation by Mr. A. Jacob, B.A., C.E., the borough engineer; Mr. J. Carter Bell, F.I.C., &c., county analyst; Messrs. John Newton and Sons, engineers, Manchester; Mr. Giles, of Messrs. Mather and Platt, electrical engineers, Manchester; Dr. Charles A. Burghardt, Lecturer in Mineralogy at Owens College.

I would also refer you to a paper recently read before the Manchester Section of this Society by Mr. Carter Bell, the borough analyst for Salford, in whose remarks Dr. Burghardt, an independent authority, permits me to add that he concurs. He cannot give details until his report has gone in, which will be very shortly. (See p. 1101.)

Mr. Carter Bell's report has gone in, and although he is precluded also from giving full details, he has kindly put at

my disposal samples sealed by him of the effluents produced by the electrical treatment which I now submit, together with the analyses, as follows:—

	Parts in 100,000.			
	May 15.	June 7.	June 30.	July 25.
Total solids.....	Not filtered. 109	125	141	152
Loss on ignition .....	33	21	29	23
Chlorine.....	32	44	42	43
Oxygen required for 15 minutes .....	2.56	0.76	0.27	0.79
Oxygen required for three hours.....	4.27	0.79	0.50	1.00
Free ammonia .....	2.20	0.88	0.50	0.92
Albuminoid ammonia.....	0.32	0.17	0.092	0.19

The samples are taken at random.

Whether the process will or will not be adopted by the Salford authorities I am of course unable to say, but I think I may safely say that the electrical process has now absolutely proved its case in regard to the solution of the sewage problem. It is simple, efficient, and I am sure more economical than any other known process where duration is taken into account.

In regard to the Salford trials it may be interesting to give the following particulars:—

The electrical shoot was built in brick and contained 28 cells arranged in series.

Each cell contained 13 east-iron plates 4 in. × 2 ft. 8 in. ×  $\frac{1}{8}$  in. thick connected in parallel.

The available electrode surface in each cell was 256 sq. ft.

The ampère hour treatment required for Salford was found to be about 37 ampère hours per gallon, and the I.H.P. per million gallons based on these figures would be 37.

NOTE.—In estimating for the plant necessary for treating the whole of the Salford sewage, a margin was allowed on above figures. The A.H.T. was taken at 44 and the I.H.P. per million at 39 to 39.5.

Mr. Octavius March, electrical engineer, who has followed the process from the commencement, and who superintended the electrical details both at Crossness and Salford, will give you on the blackboard a rough sketch of the above trial plant.

The Salford tanks are admirably adapted to the application of the electrical, or in fact any process of precipitation. They are 12 in number, and it is proposed to take two end tanks for the electrical channels, in which the iron electrodes would be placed.

The total I.H.P. required for treating the whole of the Salford and Pendleton sewage, taken at 10,000,000 gallons per 24 hours, is calculated at 400 I.H.P., based on the actual work done during the trial. The electrical plant would consist of four engines and dynamos, any three of which could do the whole work, and three boilers, each of 200 I.H.P.

The total cost of plant, including alterations, is estimated at 16,000*l.*, to which must be added the cost of about 5,000 tons of iron plates—ordinary cast iron—at say, 4*l.* per ton. These plates would last for several years.

If filtration were required, there would be an extra expenditure for this, but it will be remarked that as the treated sewage is practically purified when it leaves the electrical channels, these filters would be only required for complete clarification, which for most places would not be a necessity.

The filtering material used could be gradually prepared from the sludge obtained after electrical treatment, unless it could be more profitably sold as a manure, and I am not a believer in the value of sewage sludge in large quantities. This sludge, a waste product, is converted into *magnetic oxide of iron*, of which I have here two small samples. This magnetic oxide is a good filtering material, but, like

every other filtering material, it would of course require renewal. There would, however, always be a supply of the waste product—sewage sludge—on the spot, and the spent magnetic oxide recarbonised could be used indefinitely.

The annual cost for dealing with the Salford sewage is estimated at in round figures 2,500*l.* for coal, labour, maintenance of engines, boilers, and dynamos. To this must be added the consumption of iron and its replacement, which would have to be written off capital expenditure.

If a colourless effluent were required, absolutely free from suspended matter, the additional cost is estimated at from 1,200*l.* to 1,500*l.*

#### DISCUSSION.

Dr. GUSTAV BISCHOF wished to emphasise the great difference with which the sewage question was approached 20 years ago as compared with the present time. Twenty years ago one used to hear of the *utilisation* of sewage; now the question had become the *disposal* of sewage. In other words, we were satisfied now if we could get rid of our sewage with a reasonable expenditure of money. With regard to the process so ably brought before the notice of the meeting, he was under the disadvantage of having no practical experience with it. He could only come forward as a theorist, referring to some researches, and leaving the meeting to judge of their bearing. Sewage was a very complicated body, containing matters in solution and in suspension, both organic and mineral. Amongst the matters in suspension were the living organisms, and if he had understood the paper correctly, the author attached great, perhaps the greatest, importance to these. The question thus arose, what became, from a biological point of view, of the sewage after it had passed Mr. Webster's sewage works? Was it still capable of causing nuisance? This question was essential in considering the effect of the process in purifying sewage. The first experiments on the action of electricity on microphytes were, he believed, made by Schiel in 1875. The criterion was then the suspension of motion, which was now known to be a mistake, since a great number of organisms were capable of resuming motion after its temporary absolute cessation. The next and only other experiments on the subject that he was aware of were made about 1883 by Drs. Ferdinand Cohn, of Breslau, and Mendelssohn. Their first attempts were with a "constant" Daniel's cell, which they soon found to be constant only in name, and discarded for the Marié-Davy cell. Though this was a strong cell, the first experiments with one cell only produced no perceptible effect. Then they had recourse to two cells; but although the result was an appreciable diminution of organisms, life was by no means finally extinguished. If some of the same nutritive medium—which was a purely mineral one—was sterilised and then inoculated with a drop of the liquid resulting from the experiment, the mixture soon became full of life. Only when using two strong Bunsen zinc carbon cells together with the two Marié-Davy's all life ceased, as inoculation with the liquid thus treated was ineffective. In all the experiments the electrical treatment was continued for 24 hours, and the temperature was maintained at 30° to 35° C. He would leave the members to draw their own conclusions from these facts whether any such conditions existed in Mr. Webster's process. He believed that Mr. Webster attached some importance to the elimination of chlorine?

Mr. WORTH dissented.

Dr. BISCHOF, continuing, said: As Mr. Webster did not attach importance to that point, he would not pursue it, but would conclude by wishing the author all success with his method, feeling sure that he fully recognised that criticism was the backbone of success.

Mr. W. C. SILLAR considered that the sewage problem, though not the greatest difficulty of the age, was nevertheless of such importance that one ought to hail with pleasure any genuine attempt to solve it. He would like to ask the author, firstly, whether the electrical process removed the dissolved as well as the suspended impurities of the sewage; secondly, whether the impurities so separated lay so exclu-

sively at the bottom of the tank that the effluent could be run off without fear of disturbing it or carrying some of it into the river. Thirdly, he would like to ask whether the electrical process was applied merely to the "disposal" of sewage, or whether it provided for that "utilisation" of it which, as the last speaker had said, was the idea 20 years ago. He was strongly impressed with the idea that every evil that came under his notice had in some way or other a duplicate: that if the evil took the form of matter out of place, there was a place for it void somewhere. The same matter which polluted our rivers, if properly utilised, would become the solution of our agricultural difficulties. No solution of the sewage difficulty could be considered satisfactory unless it provided for its appropriation in whole or in part to agricultural purposes. The precipitation process at Kingston did so provide, and he thought it would be possible to compare the two processes in very few words so as to ascertain whether the one now before them possessed any advantages. First of all, the effluent water from the A.B.C. process was sufficiently purified to satisfy the requirements of the authorities, and was not at all deleterious to fish life in the river. He presumed that Mr. Webster claimed this point, but it was quite certain that the electrical process could not be superior to the Kingston process in that respect. Secondly, the area required for the A.B.C. treatment was very small, and he supposed that Mr. Webster would also claim that. Thirdly, no subsequent filtration or irrigation was required. On that point he understood Mr. Webster to say that his effluent required subsequent filtration to render it clear. This would be found to be a great disadvantage in many localities, where it was essential that the sewage treatment should be begun and ended in the sewage works. Lastly, and most important of all, at Kingston the sludge was prepared and dried, and had both an agricultural and a market value, in proof of which he might state that upwards of 10,000 tons had been made and sold at 70*s.* a ton. Did Mr. Webster claim that his sludge had any agricultural or market value? Had he ever sold any of it? If not, he had only half solved the difficulty.

Mr. P. BIRCH would be glad to be informed as to the cost per head per annum of dealing with sewage completely under this system; precipitating, drying, pressing, and getting it into such form as to be carried away. All sewage processes consisted largely of precipitation. One method might be cheaper than another in that respect, but it might involve more expense for drying. For example, they had just heard of another process which depended upon precipitation, namely, the A.B.C. process. No doubt that process gave a clear, sweet effluent, but it differed from the method under notice in that whereas the electrical process involved the addition of a very small amount of chemicals, Mr. Sillar's process required a large amount. In days like the present, when it was difficult to get rid of the sludge, and when they had to dry it in a cleanly manner, the quantity of material to be dealt with was an important consideration. He would ask whether it was not a fact that Mr. Sillar had been 20 years getting rid of his manure. They knew that there was a large fortune at his back pushing the manure on the market, and he believed that a great amount of skill was employed; but, as far as he could make out, the sludge which was dried and sold at 70*s.* a ton was the material which they saw depositing in the phial before them, plus a quantity of material which was infinitely less valuable. Did Mr. Webster base any recommendation of his process on the fact that when dried he could get 70*s.* a ton for his sludge? If not, no sound agriculturist would continue to give 70*s.* a ton for such matter mixed with clay or charcoal, worth only a few shillings a ton. With regard to the process immediately before the meeting, he would ask Mr. Webster to take upon himself the task of putting before them a little more information as to the outside cost of ridding sewage of suspended matter, and drying it, and also, when necessary, of obtaining a greater degree of purity. If they could get some figures of that nature, say a statement in pence per head of population, it would greatly assist members in forming an opinion of the value of the process. He knew that Mr. Sillar was very keen upon the question of agricultural depression and the prevention of

waste, but for his part, he thought that a greater sin than wasting a thing was spending more than it was worth in attempting to use it.

Mr. C. F. CROSS said that, apart from the economic question, there were some points connected with what actually took place in the electrolysis which he would be glad to have further explained. So far as he had understood the reader, the only information given respecting the actual operation of the current was that a hypochlorite of iron was formed at the anode, and they were left to guess what took place at the cathode. He presumed that in this electrolysis the operators relied upon the chlorides present in the sewage. Chloride of sodium being electrolysed in solution, the effect of the acid would be to dissolve the iron in the form of ferrous chloride, which would afterwards meet the base separated at the cathode, and thus ferroso-ferric hydrate would be precipitated. This would remove the dissolved oxygen of the dilute sewage. The presence of ferric oxide had been alluded to as a product of the action, but nowhere was it stated how the ferric condition was arrived at. He failed to see where the oxygen could be obtained to raise the iron from the ferroso-ferric to the ferric state otherwise than from the atmosphere. Assuming that the water was electrolysed, hydrogen and oxygen would be formed, but the only effect would be to fix the latter on the iron plates and simply oxidise the electrodes *in situ*, without producing any effect on the sewage. Mr. Webster would, he thought, confer a benefit on the Society if he dissected some of the elements of the problem and told them what took place at the electrodes, and how the products acted on the dissolved organic matters of the sewage. The organic matter should be selected with a view to determine precisely what this action of the electrolysis effected. Sir Henry Roscoe did not appear to have examined the condition of the discharged effluent in regard to its condition of aëration, and that was a matter which he (Mr. Cross) regarded as being very important, for if a lot of liquid deprived of oxygen were run into a sluggish stream, the abstraction of oxygen from the stream itself was likely to affect fish life in it. Seeing that the general interest in the paper was economic rather than scientific, he would not take up further time with such points as he had already raised; but he thought that in a matter of this kind, where a process departed in an essential particular from general practice, they had a right to ask that the elements of the problem should be laid before them. He would limit himself therefore to inquiring how the iron was raised from the ferroso-ferric to the ferric state, and whether in fact that was not done at the expense of the due aëration of the effluent.

Mr. B. E. R. NEWLANDS considered the process before the meeting to be a practical suggestion for dealing with the great sewage difficulty. They had all heard of the electric method of purifying sugar; and he had expected to find Mr. Webster's process something of the same kind, and was agreeably surprised to find himself mistaken. At the same time, he did not think the idea was quite new. About 20 years ago Mr. C. H. GILL passed electric currents through sugar solutions to free them from salts and organic matters, and in 1880 Messrs. Hodge, Hodge, and Eastick took out a patent for decomposing organic and inorganic substances by electrolysis, for the purpose of destroying colouring matters and other impurities contained therein. That patent did not relate to the purification of sugar solutions only, but was very wide in its claims. The inventors used metal plates and passed currents through them so to make them alternately anodes and cathodes, the impurities being eliminated in the form of a slime.

Mr. ALFRED E. FLETCHER rose in obedience to the invitation of the Chairman, though he could add nothing material to his opinion quoted in the paper, and which had been given after careful investigation of the process. He might, however, remark in reply to Mr. Cross that the process did not produce ferric oxide. The oxygen in the sewage water had been taken up by the organic matter itself, and, as might be expected, a ferrous salt was produced. The after-oxidation, if any, was due to exposure to the

atmosphere. An effluent was produced which was bright in appearance, innocuous, and not subject to decomposition. The question of cost could only be tested by lengthened experiments carried out on a large scale. He himself was awaiting with interest the results of the Salford experiments, which would determine the economic value of the process. As to the process yielding an innocent effluent, rapid subsidence and easy filtration, there could be no doubt.

Mr. CASSAL asked in what parts the results given in the paper were stated?

Mr. WORTH replied that they were in parts per 100,000.

Mr. CASSAL continued that in that case the results were certainly remarkable, so far as reduction of organic matter was concerned. It had been asserted by Mr. Webster that the crucial test of a sewage process was the reduction of the amount of organic matter in solution; but short of the *beau idéal* of practically complete removal, such reduction was of minor importance, since there would still be enough organic matter to putrefy and cause a nuisance. The only crucial test which could at present be applied to so-called innocuous effluents on the large scale in any given place, was that of turning the effluents into a river and observing the results. Mr. Fletcher and the reader of the paper had stated that the effluent produced by this process was an "innocuous" one, and he had no reason to doubt the assertion. But if it were so, he would be glad to know upon what grounds so important a statement had been based. The sewage treated originally contained putrefactive and probably disease organisms, and such organisms were either not present in the effluent or, being present, were not capable of producing their specific effects. If this were so, then the result obtained was so important as to put any reduction of the organic matters in solution—short of complete removal—entirely into the shade. It was obvious to anybody who had had to do with sewage that it must be treated in some way before it was either applied to land or run into a stream. Irrigation with raw sewage, except under very special circumstances, was most objectionable and highly dangerous. Like Mr. Sillar, he would be glad to find that the process under consideration was one which would go far towards attaining the end which should be kept in view; but they had yet to learn that the effluent it produced was absolutely innocuous when turned over a sewage farm. So little was the necessity of treating sewage understood, that in a paper recently read before the Society of Arts, it had actually been suggested that the sewage of London should be taken in a conduit from Crossness and Barking to the sea, and there discharged. That would mean an immense quantity of untreated sewage flowing for some 70 or 80 miles at the rate of about one or two miles an hour; and he could only say of it that no more monstrous idea had ever been put before the public even in connexion with the sewage difficulty. Few things could be worse, from a scientific point of view, than the method at present followed in London, which had not succeeded in producing anything except a costly nuisance in the Thames. The case of London afforded an illustration of the fact that most sewage processes must be judged by the effects produced in a reasonable time after the effluent had been discharged into a stream. When Mr. Webster had carried out experiments on a scale sufficiently large or in a manner otherwise decisive to prove that his effluents and sludges were innocuous, he would have taken the only step necessary for forcing his process on the most sluggish of authorities. Mr. Webster had stated that he was in possession of favourable reports from various eminent authorities. In regard to most reports upon questions of this kind, it was necessary to go through them very carefully, and to be able to read between the lines in order to have a clear idea of what their authors had in their minds when they wrote them. It was unfortunate therefore that the reports were not before the meeting. The greatest mistake that could possibly be made in connexion with the sewage problem was that of supposing that corporations could make money out of it. The only way of dealing with sewage was to get rid of it without producing a dangerous nuisance, and in the most economical way that existing circumstances would permit, regarding as the true economy that which placed the health of the community above all other considerations.

Colonel JONES said that the process before the meeting was not new to him, as Mr. Webster had read a paper on it before the Institution of Civil Engineers about a year and a half ago, and illustrated it with experiments on a fuller scale than he had shown that evening. On that occasion he was astonished at the greediness with which a scientific body of men, accustomed to the sewage question for many years, jumped at electricity. It was a quality of human nature to like anything that was magical and startling, and that would account for the reception which the process had met with on the occasion he had referred to as well as by the present meeting. Having been grinding away at the sewage problem for the last 20 years, he himself approached the subject with more caution, and was not very much startled by what he had seen. If he were given a bottle of London sewage, he would merely shake it up violently, and in a few minutes they would see the deposit going to the bottom just as it had done in the experiment made by the reader of the paper. It might be said that he was not a chemist; but he had seen Dr. Tidy do the same thing in the witness-box. It must be remembered that aeration had a great deal to do with sewage treatment. If one took a bottle half filled with sewage and half with air, and agitated it thoroughly, somehow or other a disturbance was created, and in about 20 minutes a great deal of matter went down. That simple effect of deposition was taken credit for by all the patent sewage precipitation people. They put their pinch of lime, 3 grains or 3·7, into a gallon of sewage, agitated it, and then claimed that the deposition was the effect of the 3 grains. Let them try it on the sewage that had stood stagnant for 20 minutes. They did not do that, but they compared the analysis of their effluent with the raw sewage which contained all the matter which by 20 minutes' stagnation would go to the bottom. Was it scientific, he would ask, when a thing was brought to you for analysis, of which more than one cause might have produced the effect, to attribute it to one cause off-hand, or should not one try and eliminate all save one agency. He contended that if one wanted to try the comparative merits of Mr. Dibdin's 3·7 of lime and 1 of iron and this electric process, or any other, one ought first of all to give the sewage some 20 minutes' stagnation, and then compare the analysis of the supernatant sewage with the effluent of the processes in question applied to the same supernatant sewage. He thought it important to consider this view, because during all the years that people had been fighting over precipitation processes, they had neglected all that could be done by simple deposition; and that view had been equally neglected in all the blue books on the subject, until the appearance of the excellent report of Lord Bramwell's commission. That was the first time that simple deposition was officially noticed, and it was referred to in the carefully worded conclusions which the Board of Works ought to have carried out, in the following words: "after the separation of the solids by simple deposition or precipitation."

Mr. DAVID HOWARD thought that the views put forward by the last speaker had a bearing which should not be overlooked. Time was an important element in the question, and if only the electrolytic process would aggregate the suspended matter and cause it to deposit faster than would be the case where merely allowed to stand, it would be valuable on that account alone. He did not share the views of those gentlemen who expected to find sewage anything but a nuisance; but if it was possible by this process to get a rapid deposition of a sediment that could be dealt with practically, then he believed that the sewage problem was solved, as far as in this world of trouble one could hope to solve it. One could not hope to get rid of all the organic matter, nor to obtain an effluent fit to drink.

Dr. R. MESSEL said that, assuming Sir Henry Roscoe's report to be borne out in practice, the question seemed to resolve itself into one of cost, and upon that point it was difficult to form a reliable opinion in the absence of further information—the more so as, if the process mainly depended on oxidation, electrolytic oxygen or chlorine was the most expensive form in which those chemicals could be employed. It would therefore be highly interesting to have some more

information both from an electrical and a chemical point of view. He would also like to know whether cast iron answered well for the electrodes.

Mr. WORTH answered in the affirmative.

Mr. CASSAL wished to explain that he did not intend to say that the *beau idéal* of sewage treatment was to remove the whole of the organic matter in solution until a water equal to distilled water in organic purity remained. His object was to convey that such effluent should be freed from organic pollution to such an extent as to render it equal in purity to the stream into which it was to be discharged.

Mr. A. P. TROTTER had gathered from the reading of the paper that the flow of the sewage would be at the rate of about 16½ ft. per minute, which would allow about 12 minutes action for the whole process. He would like to ask whether that was correct, and also what time was allowed for the subsidence?

Mr. C. C. HUTCHINSON regarded the question of sewage treatment as being altogether a practical and economic one, and mainly one of cost. However perfect a process might be from a scientific point of view, it was well known by everyone who had any experience of the sewage question that as soon as it came into practical operation the best possible results became subordinated to questions of cost. He did not rise for the purpose of criticising the results in the rationale of the elaborate process before the meeting. With the amount of information which the author had given them, accurate criticism was impossible and out of the question. They had no figures, and hence they could not really say whether the electrical process came within the range of practical politics, if he might use such a term in connexion with sewage treatment, or not. He would like to ask the reader one question with a view to elucidating the question of cost. He had told them that for a flow of a million gallons per 24 hours, power equal to 37 indicated horse-power would be necessary. Was that 37 indicated horse-power to be taken at per hour in the usual way? And was the treatment of the Salford sewage to take 400 indicated horse-power reckoned in the usual way for cost per hour? Perhaps Mr. Worth would answer those questions at once.

Mr. WORTH replied that it was per hour.

Mr. HUTCHINSON, continuing, said that that being so, the figures given for the production of 400 horse-power for 24 hours were remarkably low, and, in his opinion, quite inadequate. He had had some experience connected with the production of power, as no doubt many of the members present had, especially Mr. Newlands, and they would doubtless be able to support him when he said that if a horse-power could be produced on that scale much, if anything, under 1*d.* per indicated horse-power per hour it would surprise him. If 400 indicated horse-power were needed for the treatment of 10 million gallons of sewage per day of 24 hours, that would come to something like 400 pence per hour, say 33*s.* per hour, which for a day would be 40*l.*, and for a year about 14,600*l.* for power alone. On the other hand, if the cost of power were taken at 1½*d.* per horse-power per hour, a figure more often the case in practice with ordinary appliances and attention, the cost of power would become 21,900*l.* per annum, added to which would be the cost of sludge disposal, &c. If to that they added the cost of the 500 tons of iron which was to be converted into the active iron salt, the cost of the treatment of Salford sewage would be brought up to something like 24,000*l.* per annum on the highest estimate and 17,000*l.* on the lower scale. Now, it was well known that sewage could be treated at a total inclusive cost of about 1*s.* per head per annum on the population. This would make the cost of the treatment of the sewage of Salford about 15,000*l.* per annum, including everything. He was aware that the value of any particular sewage process had reference not only to the actual results obtained, such as were shown on the table before the meeting, but also to the location of the particular works. If the effluent from the Salford works was to be turned into such a large stream as the Ship Canal, a very rough treatment would be ample, and so long as suspended and



offensive introgressive matters were removed, absolute purity of effluent was not requisite. So far as Salford was concerned, what they had to consider, therefore, was whether the Webster process would yield better results as regards cost alone than any of the other well-known processes, and if it did not compare in cost, he should like to know in what would the advantage of the process consist? It seemed to him that in the case of such a town as Salford, other things being equal, the only point upon which the Webster process could show any advantage was of cost. The A.B.C. process alluded to by Mr. Sillar was very good as a treatment process; but whilst thinking it gave good results as far as purification was concerned, he did not, like Mr. Sillar, claim that it was a perfect panacea for the sewage nuisance, or that it would yield the users of it large, or indeed, any profit; but he did say that he believed that the sewage of Salford could be treated by the A.B.C. process at a cost of something like 15,000*l.* per annum. He would like to hear from Mr. Webster whether he claimed that his process would yield better results at Salford than could be got by the A.B.C. process, or by any one of half a dozen other well-known processes equally as good, and if so, whether he based his claim on anything like equivalence of cost?

The CHAIRMAN, before calling on Mr. Worth to reply, observed that it was interesting to note that several of the members who had taken part in the discussion had borne testimony to the salutary efficacy of certain salts, chiefly salts of iron, which had long been popular in connexion with sewage treatment. The main point, however, to which he would direct Mr. Worth's attention, was the economic aspect of the question. As so many speakers had referred to that point, he trusted that it would receive the very specific attention of the author of the paper, to whom, as he was absent, notes of the discussion would be sent.

Mr. F. G. WORTH said that he would do his best in Mr. Webster's absence, and as his substitute at the eleventh hour, to reply to the various points raised during the discussion, and if he was unable to reply clearly to any particular questions, he would undertake that Mr. Webster himself should do so if the members concerned would submit them to him in writing. He referred especially to one or two chemical queries, as to which he was bound to admit that he had not the proper knowledge to enable him to reply satisfactorily. The subject was a complicated one, and he must confess himself to be as unable to define the exact chemical reactions which take place as many eminent chemists had declared themselves to be. But he would consult the inventor.

Dr. Bischof had spoken about the presence of organisms, and he should state at the outset that the electrical process did not claim in actual work to destroy the whole of the organisms present in sewage. That aspect of the question had been very seriously considered; in fact it was during Mr. Webster's bacteriological study of the problem that he (Mr. Worth) first became connected with the process. He was then living in Paris, and had the opportunity of going to M. Pasteur's laboratory. He took some samples of treated sewage both to M. Roux at Pasteur's and to M. Marié-Davy for examination. He was told that the electrical process had succeeded in reducing the number of organisms from something like 5,000,000 per cubic centimetre to 600, but that notwithstanding this reduction there might be several millions again by the following day. From that moment he had felt that it was useless in actual practice to devote attention to the bacteriological aspect of the question, the more so as M. Roux had remarked that bacteriologists were as yet only on the threshold of the science. In fact most bacteriologists would, he thought, admit that they could not say what organisms were beneficial and what harmful; and therefore by destroying all they might do positive harm.

In reply to Mr. Sillar's remarks as to the existence of the A.B.C. process for a quarter of a century, the accuracy of which he quite admitted, he would merely say that the Electrical Company was as yet quite young. It was feeling its way, honestly and straightforwardly, as one gentleman

had said; but it had not yet got to that stage at which they could say that they had a town which had been working for a long time with practical results. He hoped, however, that they would soon be in that happy position, and long before 25 years had passed he trusted the Electrical Company, more fortunate than Mr. Sillar, would have proved its case otherwise than by adoption in one town only; at any rate if they had not, he should long before that time expired have nothing more to do with the electric process. He could only compliment Mr. Sillar on the pluck with which he had stuck to a process which so many had condemned. He said that honestly, for he felt that Mr. Sillar's courage deserved more success than it had met with. Mr. Sillar had asked a question with regard to the running off of the effluent without disturbing the sludge. They had always been able to do that; further, there could be no question as to the process removing a very large percentage of dissolved organic matter as well as matter in suspension. But as he wished to tell them the defects of the process as well as its advantages, he would admit that with certain sewages they sometimes got iron in a very finely-divided state in the effluent, and in that case a little organic matter was sometimes hung up with the iron, which they had to get rid of. In certain sewages that might be a source of annoyance. Of course the electric process by itself might not be a panacea for everything. The Electrical Company would have to deal with acid sewages, and sewage containing sulphates, and so on. In such cases, after treating the sewage by electricity, they might, to produce a clear effluent, have to pass it through some filtering material or otherwise treat it in a manner covered by the Webster patents. He would, however, ask members to bear in mind that the sewage was practically purified after electrical treatment, and that they simply relied on filtration, where considered necessary, as a mechanical agency. As to the disposal or utilisation of sludge, that was a delicate question, and in big towns a very disagreeable question. He held that it should be shelved as well and cheaply as possible, but that it must be done at any necessary cost. He did not believe in the manurial value of sludge or in obtaining value from farmers for it. One might, as Mr. Sillar had done, sell 10,000 tons of it in 25 years; but in the case of such towns as London and Salford that quantity would be a mere nothing. In the case of London and Salford, few agriculturists were at hand to take the sludge; and if there were enough, they would only require manure during about three months of the year. During the rest of the year the sludge would have to be stored, and if rain got to it during that period what little ammonia it contained would go off. Considering the cost of carriage, &c., he personally thought it would be much better for farmers to buy sulphate of ammonia, nitrate of soda, and phosphates. But if sludge had any manurial value, the small addition of iron which the electrical process put into it would, for many soils, increase that value. It was possible, too, that it might prove to be valuable in another way, if the experiments which were being carried out succeeded. Those experiments consisted in filter-pressing the sludge, and then carbonising it to such an extent as to produce the red magnetic oxide shown in one of the phials before the meeting, or carrying on the carbonisation far enough to produce the black magnetic oxide, a sample of which was also shown. If those experiments were successful, Mr. Webster would communicate the results to the Society at a later date. It was impossible to compare the effluent produced at Kingston with that which was obtained by the electrical process at Salford, the two sewages being so different in character. The only way of arriving at a comparison of the two processes would be for both of them to deal with the same sewage at the same time. With regard to the effect of the effluent on fish life, he paid very little attention to that point; fish were shown in most effluents. An alderman who visited the electrical works, on being shown some fish alive in their effluent, said: "Well, what does that prove? When a boy I always got most fish in the dirtiest water." The fact remained that fish did live in the electrical effluent. He thought he had answered most of Mr. Sillar's remarks, and he thanked him for the spirit in which, as a competitor, they were made.



Mr. Birch had asked for information with respect to the cost per head of population for completely treating sewage by the electric process. He was sorry to say that at the present moment he was not in a position to give the whole of the data. At Salford the electrical process was engaged only in producing a satisfactory effluent, the sludge was considered to be a separate and subsidiary question. The company had asked to be allowed to carry out experiments with the sludge, but they were met with the reply: "We do not think such experiments necessary. We shall always have sludge, though less by your process than any other, and we do not wish to waste time on them at present." For that reason only he was unable to give the information which Mr. Birch so naturally asked for. So far as they could give figures, both Mr. Webster and himself were willing and anxious to do so. But sewages differed in the treatment they required, and it was best therefore to deal with a case in which the experiments had been carried out as at Salford, not by the company alone, but by a corporation who had controlled the whole thing, and whose report would be issued shortly. Perhaps on that account the reading of the paper had been somewhat premature, and it might have been better to wait until the report was actually out, and so be able to give other people's figures, which might naturally be considered more reliable than any of those which he could bring forward. Mr. Webster had felt, however, that it was due to the Society that the paper should not be further postponed, and had it not been for his illness, which he had fought against to the last moment, the meeting would have had full replies on the chemical points raised.

Mr. Cross had put some chemical questions which were quite beyond his (Mr. Worth's) power to answer, but he was sure that Mr. Webster would be happy to do so if Mr. Cross would state them to him.

Mr. Newlands had spoken in such a manner as to leave him absolutely nothing to say in reply, except to thank him for his reference to certain patents which he would take the first opportunity of looking into; for, as Mr. Newlands had said, everyone can learn something from other people's patents.

He must also thank Mr. Fletcher for his remarks; his report was the result of independent investigation, and it was all the more satisfactory therefore to find him corroborating everything that Mr. Webster and himself had said.

Mr. Cassal had made a speech which he would be glad to answer more in detail than time would then permit. He had raised the question of organisms, and on that point he (Mr. Worth) had expressed his personal views in replying to Mr. Bischof. With regard to taking out the whole of the organic matter in solution, of course Mr. Cassal meant the whole of the putrefying matter, for to take out the whole would be of no great advantage; and it was very difficult to decide what was the exact quantity that should be taken out. A pertinent question had been asked as to the meaning of the term innocuous, and on what it was based. Chemists and others could only do what was humanly possible to ascertain whether the effluent was innocuous or not. They had taken effluents produced on a working scale from different sewages; had kept them in stoppered bottles and in unstoppered bottles; had mixed them with water and kept them without water; and none of them had gone bad. He would expect that if they ran such an effluent as that produced containing a small proportion of organic matter into a stream, the oxidising action would rapidly take away the objectionable portion of the effluent. With regard to sending sewage to the sea, that was rather outside the limits of the question; but he quite agreed with Mr. Cassal that nothing could be much worse than the system at present followed in London. The sewage had to be dealt with and got rid of somehow, and though by electricity they did not profess to do it so economically as some processes professed to do, they did claim to do it efficiently and at a reasonable cost. Neither did they profess that their effluent was to be regarded as a drinking water; at the same time it was a thoroughly harmless effluent, and independent chemists had stated that after submitting it to the severest tests there were no signs of after-putrefaction. If anyone else could do better,

Mr. Webster and himself would have no course but to retire from the field, for their continuance in it could only be maintained by commercial success. He might add that the full reports of the chemists were open to inspection to anyone desirous of fair investigation.

Colonel Jones had made a characteristic speech, repeating what he had seen 18 months ago, when the electrical process was in its infancy. But it must have been a very promising child for the engineers to jump at it as Col. Jones had said. The experiments shown that night were certainly not on so large a scale as those shown on the occasion referred to by Col. Jones; but he hoped that they had been sufficient for the purpose, especially as they were now backed up by practical results. He was sorry to see that Col. Jones had left the meeting. If he were still present, he would be happy to submit a sample of sewage to the test he had proposed, asking Col. Jones, or any other gentleman who would now take his place, to shake it up for 20 minutes and compare the result. The analyses of the effluent were not compared with unsettled sewage only, but also with settled sewage. It was to be remembered that sewage must be taken as it came, it being generally impractical on a large scale to have separate tanks for it to settle in before treatment. To determine chemically what degree of purification any process had attained, one must compare its results with settled sewage. That had been done in their case.

In reply to Mr. Howard's inquiry as to whether the sewage deposited quicker by the electrical method than by mere standing, there could be no doubt that it did. If sewage were allowed to stand for 24 hours, there would be no material difference effected; practically no organic matter in solution would be removed. If it stood for any length of time it would become as clear as the effluent before them, but that would prove nothing.

Mr. Hutchinson's criticism of the process would be serious, if he had not unintentionally erred on the side of exaggeration. He had said that all other points of the process would be subordinate to the question of cost, and he had criticised the figures given for the production of the power required at Salford. But he would remind Mr. Hutchinson that the figures for horse-power would not be the same everywhere, but would vary with the sewage. Salford, for instance, would require more per million gallons than London. Mr. Hutchinson estimated the cost of producing an indicated 400 horse-power per hour at 16,000*l.* per annum. The company's engineers had worked it out themselves, and their estimate was before the meeting. Perhaps their figure for coal was low; but with the highest class of electric machinery they had a right to expect that they would obtain results as good as had yet been got. They could, he thought, calculate on using not more than 2 lb. of coal per indicated horse-power per hour. If they were wrong, if the actual quantity would be 3 lb., then the estimate for that item must be increased by one-half. But he was told, and he believed that he could have a guarantee on the point, that it could be done with 2 lb. of coal.

Mr. HUTCHINSON, interposing, said that the cost for fuel was only a small portion of the total cost for horse-power. If they reckoned their fuel at 2 lb. per horse-power, they would have to use Welsh coal, which at present was 18*s.* per ton. And if to that they added the charges for wear and tear of machinery, interest on cost of plant, labour, &c., they would find the cost amount to quite a penny per horse-power.

Mr. WORTH said not; but there was not much use in continuing the discussion on that point then. It would be best that the figures verified by the engineers at Salford should be put before the members. At present the figures which he had given were the estimate of the company, and he would stand by them. To them, however, must be added the loss for consumption of iron, which, at the rate of 3 grains per gallon, would equal 690 tons per annum, worth 2,760*l.*; and of course there were sundry other items of expense. As to the advantages of the electrical over other processes, and its superiority to chemicals, he thought it best for this question to be answered by the independent reports obtained and those to be submitted.

Dr. MESSEL remarked that they would have to renew their iron plates long before they were completely worn out.

Mr. WORTH admitted that, and said that the question was, how soon? They would certainly last several years. Cast-iron plates were used at Salford.

Mr. HUTCHINSON said that, at 3 grains per gallon, the loss would be 740 tons per annum.

Mr. WORTH replied that he adhered to 690 tons, which he thought was correct for 10 million gallons a day. Dr. Messel had said that they had no information as to cost. They had information, but it was the company's, and he thought that it was best that the information should come from others.

In answer to Mr. Trotter's question, the rate of flow was difficult to give off-hand in feet per minute. The rate of flow was about 3,000 gallons per hour with a shoot 18 in. wide. The time of subsidence varied, the maximum being three hours. A continuous flow was, he thought, better than intermittence for electrically-treated sewage. With regard to the Chairman's closing remarks, he could only repeat that he would get Mr. Webster to put before the Society, as early as possible, the estimates made by independent men, as well as the full chemical reports, which would be far more valuable than any he could lay before them at that moment. He would conclude by thanking the members for the very courteous reception they had accorded to him as Mr. Webster's substitute, and much regretted that he had only had a few hours to put Mr. Webster's rough notes into a readable form, which necessarily caused the paper to be incomplete on many interesting points. In consenting to act at such short notice as Mr. Webster's substitute, he was animated by the desire not to disappoint the meeting altogether, and claimed their indulgence for present shortcomings.

At the close of the discussion Mr. WATSON SMITH exhibited a new form of india-rubber stamp enclosed in a locket, the lid of which closing upon a spring handle attached to the stamp, presses the latter upon a pad. The stamp is thus always ready for use, and whilst by gentle pressure upon paper it gives a sharp and clear impression of the benzene ring in single lines, by stronger pressure alternate double and single lines appear. By gentle pressure twice performed and at points in close juxtaposition the naphthalene ring is delineated. This convenient aid to the writing of abstracts, notes, papers, &c., on aromatic organic chemistry was sent for exhibition by, and is the invention of, Mr. C. S. S. Webster of Bristol.

## Manchester Section.

*Chairman:* Ivan Levinstein.

*Vice-Chairman:* Edw. Schunck.

*Committee:*

J. Augell.  
G. H. Bailey.  
R. F. Carpenter.  
G. E. Davis.  
H. Grimshaw.  
Harold B. Dixon.

J. Grossmann.  
P. Hart.  
A. Liebmann.  
Sir H. E. Roscoe, M.P.  
C. Truby.  
D. Watson.

*Hon. Local Secretary:*

J. Carter Bell,  
Bank House, The Cliff, Higher Broughton, Manchester.

Notices of Papers and Communications for the Meetings to be sent to the Local Secretary.

*Meeting held on Friday, 7th November 1890.*

MR. IVAN LEVINSTEIN IN THE CHAIR.

THE CHAIRMAN called attention to the resolution passed by the Committee with regard to discussions being held at the ordinary monthly meetings previous to the reading of papers. It was thought that discussions not exceeding half an hour would afford a means to the members of interchanging their ideas on those subjects in which they were more especially interested.

The CHAIRMAN introduced for discussion—

"AMENDMENTS ON THE PATENT LAWS AS SUGGESTED BY THE CHEMICAL COMMITTEE OF THE MANCHESTER CHAMBER OF COMMERCE."

THE CHAIRMAN said it would scarcely be necessary for him to read through the whole of the report of the committee, but he would lay before them the proposals which the Manchester Chamber of Commerce intended to introduce to the House of Commons next session, and which he hoped might receive the support of our Section, as representing the chemical and allied industries of Lancashire. The report and the proposed amendments had not only been approved by the Board of the Manchester and also of other Chambers of Commerce, but several members of Parliament had also expressed their sympathy with them and promised their support. He would read to them the following letter which he had received from Sir Henry Roscoe:—

"I have carefully read through the draft report of your committee to the Manchester Chamber of Commerce. The subject is one in which I feel much interest, and your report has my hearty sympathy. I quite agree as to the necessity of some change in the law of chemical patents, as you point out. Section 18 opens the door to abuses of a serious character, and the proposals of your committee seem to me to meet the difficulty, inasmuch as they do not act as restrictions to honest inventors, but only prevent patentees from abusing their privileges, &c. If the Chamber decide to take action in the matter I need scarcely say that I shall be glad to give it every assistance in my power in the House of Commons."

THE CHAIRMAN said it might interest the members to know that the Chemical Committee of the Manchester Chamber of Commerce has at present under consideration the new tariff proposed by the French Government, as far as chemical products came into question. The proposed tariff was by no means a general advance on the duties at present charged and compared with other products. The French Government had wisely taken care not to ballast too heavily chemical produce, which, to a large extent, served as raw material in the production of textiles. Still, in some few instances, the proposed increase was such as to make import to France from England prohibitory. For instance, alum (ammonia or potash) and sulphate of alumina, which now paid approximately 7s. 4d. per ton, were proposed to be taxed at 30s. 6d. per ton, which was more than four times the duty at present charged. There was, further, an increase of about 25 per cent. on iodide of potash and ammonia. Oleic acid, which was now free, would pay 1l. per ton; the duty on stearic acid was more than doubled, and oxide and carbonate of lead, cobalt salts, calcium citrate, which were hitherto exempt from duty, were proposed to be charged respectively per ton, 16s., 14l., and 3l. There was also an enormous and absolutely prohibitory increase on salts of quinine. On the other hand there was proposed a reduction of duty on the following products: Bromine and its salts, oxalic acid, borax, aluminium, chloride and chlorate of potash. Out of 117 chemical products there would be a change in the proposed tariff in the case of 16 products, whilst 101 products would be taxed as hitherto. The intermediate products from coal tar used in the manufacture of coal-tar colours, such as nitrobenzol, carbolic acid, and those not enumerated, were under the present tariff free; whilst under the proposed tariff

there would be a duty of 6*l.* per ton, a move no doubt intended to further stimulate the development of the coal-tar industry in France. He only intended to give them a few illustrations of the proposed tariff. The Chemical Committee of the Chamber would be pleased to receive any suggestions or observations from members of the Society. The committee was at present engaged in preparing a comparative table of the duties at present charged, and those proposed by the new tariff. Finally, he again wished to urge upon them the advantage of joining the Chemical Section of the Manchester Chamber, which was charged to look after the interests of those engaged in the chemical and allied trades.

The CHAIRMAN then referred in detail to the report (see this Journal, June 1890, 676).

The CHAIRMAN proceeded to say that he thought it was very desirable that the meeting should support the proposals of this Section of the Chamber of Commerce.

MR. CARPENTER wished to know whether, under section 22, any case had been presented to the Board of Trade, asking them to fix a licence for any patent taken out by a foreign country, or if any application had been made for a licence on behalf of a manufacturer who finds himself unable on account of the foreign patent laws to work the process he has found out.

MR. WM. THOMPSON was of opinion that any one taking out a patent should be called upon to work it within a certain time, otherwise it would become invalid.

MR. GRIMSHAW thought the two most important points for consideration were section 18, and the one relating to foreign patents in this country, otherwise we should be handicapped in the industrial race. He had introduced this matter at the council meeting in London, and he was sorry to say that the members declined to entertain it. If they wished to make the alterations suggested by the proposals brought forward by the Chairman they would have to put into motion a very sluggish body—the Commissioners of Patents—who knew nothing about the interest which the industrial communities at large take in these matters, and the only way to get them to move would be by repeated agitation, inasmuch as those who were at the head of these Departments of State were not technical in the sense of being acquainted with the details, and consequently the one fact which has any weight with them is public opinion.

The CHAIRMAN in reply to Mr. Carpenter said, as far as he knew there had been hitherto no test case submitted to the Board of Trade under section 22. One part of this section, viz., "The patent is not being worked in the United Kingdom," is absolutely meaning less in its present form. No doubt Legislature intended to compel a patentee to grant licences in case he does not work his invention in this country, but without a proper definition of the word "worked," the principal object of this part of the section has been defective. Under this section a patentee is neither bound to work on a manufacturing scale nor is he tied to any time. He may either work his invention in his parlour or in his stable or any other sham way, or he may commence to work his patent a few weeks before its termination. The Manchester Chamber has also at present under consideration this important section, and he would not fail to communicate the result at a future meeting.

The following resolution was then put to the meeting and unanimously carried:—"That this meeting approves of the proposals for amendments of our Patent Laws as submitted by the Chemical Committee of the Manchester Chamber of Commerce."

## ON RIVER POLLUTION, AND THE PURIFICATION OF SEWAGE BY ELECTRICITY AND OTHER METHODS.

BY J. CARTER BELL, A.R.S.M., F.I.C.

As the consulting chemist to the River Irwell Conservancy Committee appointed by the council of the county borough of Salford, it has been my duty during the past two years to give special attention to that most interesting and important problem, "the purification of sewage, and the prevention of the pollution of rivers." The following resolution of the Salford council will clearly show the immediate cause of my investigations:—"Resolved, That in view of the approaching completion of the ship canal docks, and the consequent impounding therein of the waters of the Irwell, the River Conservancy Committee be instructed to present a report to the next meeting of the council, showing what improvement in the condition of the river has been attained during the past two years, and further showing what prospect there is of such purification as will render the water fit to be impounded without serious detriment to the public health." The Conservancy Committee, in reporting in accordance with the instruction of the resolution, remark:—"Your committee are pleased to find that the anxiety they have felt for some time past with reference to the condition of the river, not only in view of the early completion of the ship canal, but on the broader grounds of the sanitation of the borough as affected by the condition of the river, is shared by the council."

It is this larger question, viz., the "broader grounds of sanitation," which renders the experience of the Salford committee of use to other corporate bodies, and which forms the object and justification of my paper.

As will be readily understood, it has been my duty to examine many hundreds of samples of river waters, crude sewage effluents, &c. It is not my intention to inflict upon the Society a mass of figures. I shall confine myself to giving an average of the various waters, &c. brought under investigation. I wish to mention that the whole of the samples submitted to me were taken by the inspector acting under the direction of the River Irwell Conservancy Committee of the county borough of Salford.

I suggested to the committee that samples of water should be taken every week from the Irwell as it entered and left the borough of Salford. My idea was that we should have some record to show if there was any improvement in the state of the river. These weekly samples have been taken for something like 18 months. As I was anxious to obtain a standard of what the Irwell should be, I asked the inspector to take a sample of water before it had passed any works. This was done, and the total solid matter at Irwell springs is only 8 parts in the 100,000, and this sample was taken at the most unfavourable time of the year, when the water was charged with suspended matter equal to 2 parts in the 100,000. But immediately on passing the first works the total solids and suspended matter increased very rapidly, total solids 37, suspended 7, and so on until it reaches the borough. The variation of the total solids and suspended matter is very remarkable, for in one month it may be as low as 30, and at another time mount up to over 100 parts in the 100,000.

This variation will, to a great extent, depend upon the rainfall, the total solid matter being high in times of drought and low in the rainy season; but this rule does not always hold good. For instance, samples of water were taken on November 7, 1890, from the Rivers Irwell, Irk, and Medlock, at a time of flood, and the amount of total solids and suspended matter is given below:—

	Parts per 100,000.			
	Irwell as it enters Salford.	Irwell as it leaves Salford.	Irk.	Medlock.
Total solid matter ..	80	97	81	78
Suspended matter ..	57	68	40	27

This enormous quantity of suspended matter will be a serious inconvenience to the Manchester Ship Canal Company, and therefore it is imperative upon manufacturers to keep their solid matter out of the river.

I thought it would be interesting if a sample of water were taken on some Sunday, as I had the impression that the river would be clearer on that day than on any other day of the week; but, strange to say, the river was in a worse state than on any day during the 18 months upon which samples have been taken. This particular Sunday was July 7, 1889. The total solid matter was 106 as the river entered the borough, and 93 when it left. These very high numbers may be due to certain manufacturers who make a point of having a general "clear up" on the Saturday, and getting rid of all their objectionable matter by passing it on to their neighbours.

The question may be asked, who are the great polluters of the river? Unfortunately, their name is legion.

There can be no doubt that the paper-makers are responsible for an immense amount of pollution, both solid and liquid. The liquid, consisting chiefly of the resinous matters from the straw and esparto used, dissolved in the alkali employed. This is undoubtedly a difficult matter for the manufacturers to contend with, as the plant necessary for the utilisation of this refuse is very expensive, and not altogether satisfactory, owing to the enormous quantity of water to be evaporated, and the peculiar properties of the residue.

The solids from a paper mill are also very large, and this pollution consists of the debris of the materials used, minute portions of paper and mineral matter escaping from the washing machines, the dregs from the bleaching-powder and caustic pans. In well-conducted works the second item mentioned is reduced to a minimum, manufacturers being fully alive as to its value. The matter passing from the machines may be considered fairly inert.

The bleaching-powder and caustic dregs, however, constitute a most serious source of pollution, and the amount produced in the various paper works using the Rivers Irwell and Roach must be something like 150 to 200 tons a week.

The whole of this material can, and must be kept out of the river; this serious pollution can be prevented by very simple means, indeed several large works are already making attempts to deal with this refuse by constructing large settling tanks, into which the dregs are collected and settled. In one works which I visited, this solid matter was collected, made into bricks, then dried and burnt in a kiln, and then sold for the manufacture of cement, and as this burnt residue consists largely of soluble silica, it would have the tendency to form a good cement.

This method of collecting the solid matter in tanks, in the absence of anything better, may be effectual, if loyally carried out, but as the sludge produced is very bulky and difficult to deal with, I am afraid the river becomes after all the common settling tank; and it has come under my own observation where tanks with suitable plugs have been used, and at some convenient season the whole of the solid matter is shot into the river.

I may here remark that in days gone by, when large profits were made from the production of paper, manufacturers did not consider it necessary to attend to the waste product of their mills. The habit of waste has become a confirmed one, and it appears to me that unless compulsion is brought to bear upon these manufacturers to keep their refuse out of the river, it will not be an easy matter to teach them, and to prove that this prevention of the pollution of the river by paper-makers is not an Utopian idea. I have had a process brought under my notice, by which these solids can be wholly kept out of the river in a cleanly and convenient manner, all connexion between the settling tanks and the river is cut off, and which makes it impossible for the manufacturer to draw the plugs and send the refuse into the river. Instead of this easy and

wasteful plan, the solids are removed in a portable form, and the valuable materials, now lost, are again utilised. There is no doubt that for the expenditure of a few hundred pounds, sufficient saving could be effected to return a very handsome dividend upon the outlay.

With such a process, which is as ingenious as it is simple, paper-makers will be enabled to relieve the rivers of this enormous amount of solid matter with considerable advantage to themselves. I understand that the process of which I speak will shortly be at work in one of the largest mills in the neighbourhood of Bury.

My attention has also been directed to an ingenious invention of one of our members, Mr. W. Birch, of the Milton Iron Works, Lower Broughton, Manchester. This is described as an "improved continuous filter," and is intended for the treatment of large masses of impure water. It consists essentially of an endless band of thick felt carried over suitable rollers; the water conducted on to the surface of the felt passes readily through the same, leaving the suspended matter coating the surface of the felt. The felt, in its movement over one of the carrying rollers, is suddenly compressed by a counteracting roller, and this sudden compression of the water contained in the pores of the felt causes the coating of suspended matter, the sludge, to be driven off into a receiving box. The action of the machine is very simple, and from what I have seen of its working I conclude that it will answer to the claims of the patentee. One machine, costing 200*l.*, will filter from 100,000 to 150,000 gallons per day, and by its use filtering and settling tanks are entirely dispensed with.

It has been found that on the majority of days, when the river leaves Salford it is more impure than when it enters the borough.

I asked the inspector to obtain samples of water from every works in the borough draining into the Irwell, and the following figures, in parts per 100,000, will give you some idea of the amount of suspended matter that is being discharged into the river: 456, 246, 154, 282, 135, 159, 477, 135, 134, 264, 376, 590, 127, 184, 148, 125, 131, and 12,280.

These heavy numbers must not be all laid upon the backs of the paper-makers, for some of the sinners in this respect of pollution are india-rubber manufacturers, dyers, soap-makers, brewers, and sundry other firms; there is one number which you may think is an error, and that is 12,280. This solid matter consisted of fat, glycerin, and alkaline salts. It is difficult to believe that a manufacturer can be so reckless as to throw his profits into the river in these days of fierce competition. I feel certain that this gentleman will thank the River Conservancy for reclaiming him from the evil of his ways. The numbers I have given represent but a very small portion of those manufacturers who collectively are discharging thousands of tons of solid material into the river annually. The River Conservancy Committee of Salford is doing all that lies in its power to purify the river, and to try and bring back once more into the lovely Irwell valley the long-lost crystal streams. Yet it is sad to say in these days of enlightenment, that the committee is met by opposition, in an "Association for the Protection of Manufacturing and Commercial Water Rights." These gentlemen who form this association seem to prefer the waters of Lethe to those clear and crystal streams which flow through Elysian fields; can these gentlemen who form this association justify such samples as I place before you. I am glad to say that there are now more than 40 firms on the banks of the river who are putting up settling tanks and filters, so as to prevent the suspended matter flowing into the river; ultimately this must result in a great gain to the manufacturers, for at the present time large sums of money have to be spent to purify the water before it can be used in the processes of various manufactures. Another reason why the River Irwell is more impure when it leaves Salford than when it enters, is, because it has received the waters of the Irk and Medlock.

## Comparison of Salford sewage with the three Manchester rivers :—

	Parts in 100,000.				
	Salford Sewage.	Irwell as it enters Salford.	Irwell as it leaves Salford.	Irk.	Medlock.
Total solids .....	142	70	85	120	112
Suspended matter .....	15	7	12	26	24
Suspended mineral .....	8	2	3	8	14
Suspended organic .....	7	5	9	18	10
Total solids in solution .....	127	63	73	94	88
Mineral solids in solution .....	106	46	58	72	76
Organic solids in solution .....	21	17	15	22	12
Chlorine.....	38	23	30	29	31
Oxygen absorbed in 15 minutes.....	3.4	2.3	2.8	2.7	2.4
Oxygen absorbed in 3 hours.....	5.8	3.9	4.2	5.4	7.2
Free ammonia .....	1.5	0.24	0.72	1.44	0.24
Albuminoid ammonia .....	0.64	0.29	0.36	0.72	0.51

On comparing the above analyses one need not be surprised to find the Irwell so polluted when it leaves the borough of Salford, for the Irk and Medlock are nothing better than open sewers, and what flows along is about equal to ordinary Salford sewage, and in the future when the history of Manchester is written it will hardly be believed that a people could spend a million of money on a town hall, and yet allow three lethal streams to meander through their midst.

From the pollution of rivers we naturally come to consider their purification, and we thus come on to the sewage-question, and I need not say that this is one of the great problems to be solved. During the last 12 months the authorities in Salford have invited various gentlemen to try experiments at Mode Wheel sewage works, and I thought it would be interesting to bring before you some results of these experiments.

The first is the electrical; this is certainly the most novel of all the experiments which have been tried on sewage.

The electrical process, which is generally thought complicated, is in reality particularly simple; the system consists in passing an electric current through the sewage, which is caused to flow through a shoot or channel in which iron plates or electrodes are fixed, these electrodes being connected alternately with the positive and negative conductors of a dynamo driven by steam or other motive power.

In travelling along the shoot, every particle of the sewage comes in contact with the iron plates, and is then run into settling tanks, where the precipitation takes place.

The treated sewage as it leaves the electrical shoots is practically purified, and is free from danger of putrefactive change, but this effluent though harmless is not colourless. If a perfectly colourless effluent be required absolutely free from suspended matter, filtration is all that is necessary. The exact chemical reactions which take place are not in my opinion clearly understood. I think that the active agent is hydrated ferrous or ferric oxide. The changes which take place are no doubt very complex, rapid change and interchange going on in such a way that definition is almost impossible.

Mr. Webster is, or was, of opinion that hypochlorite of iron is formed, but this I have been unable to trace. It is, however, certain that nascent gases are liberated, such as oxygen, hydrogen, and sometimes chlorine if the sewage were very strong in chlorides. There can be no doubt as to the valuable results obtained by this process; the effluents which are now before you are as clear as spring water. With reference to the sample which has not been filtered, you will notice a reddish suspended matter; this is nothing more than hydrated oxide of iron which in my opinion is really beneficial, for as long as you have an excess of this

material, you cannot have free sulphuretted hydrogen; and time after time when I have been analysing the waters from the Irwell I have found free sulphuretted hydrogen, and I need not say that is one of the principal causes of the bad odours which proceed from the river in warm weather; therefore, to have suspended oxide of iron in the water, though it gives the water an unsightly appearance, is not without some advantage.

In regard to Salford sewage, I can confirm the statements of Sir Henry Roscoe and Mr. A. E. Fletcher, who have both reported on the process (see pp. 1093—1094), though whether the effluent produced would be considered by the authorities sufficiently clear and colourless to be sent into the Ship Canal without some sort of filtration, I am unprepared to say. Whatever process be adopted, I believe the Local Government Board insist on filtration through land, though without specifying the area, and I am certainly clear that a very small area would suffice for producing not only an innocuous but a colourless effluent from the electrical process.

I am, of course, not prepared to go into the question of cost, this mainly depends upon the consumption of coal, where water power is unobtainable, and the waste of iron from the plates; these points we shall shortly be enlightened upon, as the experiments lately carried out at Mode Wheel were subjected to the most searching investigation and control. The experimental works constructed by the Electrical Association at Mode Wheel sewage works were on a scale of operation sufficiently large to demonstrate the system.

The crude sewage was passed through a channel of brickwork 90 ft. long, 4 ft. 9 in. deep, and about 14 in. wide. In this channel were suspended the iron plates which form the poles or electrodes, 364 in number. They were made of cast iron of a common quality,  $\frac{3}{8}$  in. thick, 4 ft. deep, and 2 ft. 8 in. in width. For electrical reasons they were divided into 28 sections, each containing 13 plates, connected by copper strips to a dynamo. The plates were fixed longitudinally in the channel so as to present their edges to the flow of the sewage, and as the space between the plates is five-eighths of an inch, every particle of the sewage in transversing the length of the channel was subjected to the action of the electric current.

The impurities separate themselves in the form of a flocculent mass, which at first is buoyed up to the surface as a froth or scum by the various liberated gases, but after being allowed to remain quiescent in a tank set apart for the settlement of the impurities, they gradually fall to the bottom, and the remaining liquid is the purified effluent.

For dealing with the whole of the Salford sewage, say 10 million gallons per 24 hours, the arrangement would be practically on the same lines as for the experiment, and

the control has shown that its application presents no material difficulty inherent to the process even with such a large volume of liquid. I am informed that the indicated horse-power would not exceed 400 for the whole quantity. The chief outlay would be, probably for cast-iron plates used as electrodes, but they would certainly last from seven to 10 years, according to their thickness. The loss of iron in the experiments was about three grains to the gallon of sewage. I need not say that the sludge by this process is less than by any other, because with the exception of the little oxide of iron, nothing is added to increase the sludge, and when this sludge is dried and ignited magnetic oxide of iron is formed.

So confident are the directors of this company that success must follow their efforts, that they offer to put down all the necessary plant for the purification of any quantity of sewage, and they do not ask for payment until it has been proved that the desired end has been accomplished. I have made many analyses of the effluents, but it would be tedious to give them all, and therefore I have thought it better to give a fair average from each process, and append it to this paper.

The second process is that called the Polarite or International System. In this method the sewage is run along a shoot into a tank, and on its way it receives a small portion of a substance called "ferrozone;" this is composed of sulphate of iron, alumina, and magnesia, together with some fine magnetic oxide of iron.

#### ANALYSIS OF "FERROZONE."

Moisture.....	20.00
Ferrous sulphate.....	16.28
Ferric sulphate.....	6.07
Alumina sulphate.....	22.20
Carbon.....	1.47
Insoluble in water.....	15.20
Water of constitution and other matters....	15.78

When the sewage has been thoroughly treated with ferrozone, the treated sewage is allowed to run into a tank and to remain there for about four hours; at the end of that time most of the suspended matter will have settled, and the supernatant liquid is run on to the filter. This is composed of sand and the substance called polarite, which is a magnetic oxide of iron.

Magnetic oxide of iron.....	54.52
Alumina.....	6.21
Magnesia.....	7.24
Silica.....	24.92
Water.....	6.13
Lime, carbon, and alkalis.....	0.98

This is mixed with sand and put into the filter, about 1 foot in thickness. The purifying power of this filter is very remarkable; it reduces the organic matter about 80 per cent. and even more. At first I was very sceptical about this filter, as I could not understand how this filter could be worked day after day without losing its filtering power. I thought it would be interesting to compare an ordinary sand filter with a polarite one. For this purpose I had two ordinary 9-inch drain-pipes cemented together in an upright condition. One was filled with sand and the polarite mixture and the second was filled with sand alone. Various gallons of sewage, to which 2, 4, 6, 8 and 10 grains of

ferrozone were added. These were passed through the filters in one-gallon lots, and the difference between the two filters was very great.

In the case of the sand filter, the whole of the free ammonia was taken out and converted into nitrates.

#### SAND FILTER.

100,000 Parts.	1.	2.	3.	4.
Free ammonia.....	Nil.	Nil.	Nil.	Nil
Albuminoid ammonia.....	.08	.11	.14	.13

#### POLARITE FILTER.

100,000 Parts.	1.	2.	3.	4.
Free ammonia.....	.03	.02	.04	.06
Albuminoid ammonia.....	.02	.07	.08	.03

The appearance of these two effluents was very marked after passing through the respective filters.

The sand effluent was cloudy, with matters in suspension, while the polarite effluent was bright and clear, and could not be distinguished from spring water by the appearance. The great question is, how long will the filters possess this filtering power? At Acton, near London, the Local Board have these filters at work, and I understand that these filters have been in use for over three years, and some weeks since I visited this place and saw the effluent running away like clear spring water. The following are the analyses of the sewage and effluent:—

Albuminoids.	Acton Sewage.	Polarite Effluent.
Total solids at 212° F. in 100,000 parts	361	77
Suspended matter.....	306	..
„ mineral.....	190	..
„ organic.....	116	..
Solids in solution.....	55	77
„ mineral.....	45	50
„ organic.....	10	27
Chlorine.....	5	6
Oxygen for 15 minutes.....	1.28	0.040
„ 3 hours.....	2.55	0.240
Free ammonia.....	6.10	0.220
Albuminoid ammonia.....	0.70	0.035

Samples of water from the Irwell, Irk, and Medlock were analysed and then passed through polarite filters, without any addition of ferrozone; the following shows the purification effected by the polarite filters:—

100,000 Parts.	Irwell before Filtering.	Irwell after Polarite.	Irk before Filtering.	Irk after Polarite.	Medlock before Filtering.	Medlock after Polarite.
Oxygen required for 15 minutes.....	1.76	.34	2.07	.52	2.1	.41
„ „ 3 hours.....	3.70	.62	5.40	.81	9.3	1.50
Free ammonia.....	.72	.12	1.44	.40	2.4	.30
Albuminoid ammonia.....	.36	.14	.72	.11	.51	.12

The effluents which have been produced at Salford have been characterised by a freedom from colour and odour, and when one considers the highly-coloured Salford sewage, the results of such an effluent cannot but be considered as highly satisfactory. After quoting Sir H. Roseoe and Mr. Fletcher upon the electrical effluents, it will only be fair to quote what Dr. Frankland says of this polarite process.

"The effect upon the dissolved organic matter in the subsidence tank is very remarkable, its amount being reduced to little more than one-tenth of that present in the original sewage."

"In its subsequent passage through the filter, the dissolved organic matter is still further reduced to nearly

one-sixteenth of that present in the original sewage. It is now in a state of purity greatly exceeding that prescribed by the standards of the Rivers Pollution Committee."

In my investigations I have considered as a standard for a good effluent that the free and albuminoid ammonia shall be low, that the quantity of oxygen required for the 15 minutes and three hours shall be small, and that the appearance of the effluent shall be bright and clear.

Comparative table of Salford sewage and effluents resulting after treatment by indicated processes. It must be clearly understood, however, that the various effluents are not the result of the treatment of the sample of sewage of which the analysis is given.

	Salford Sewage.	Effluents.					
		Electrical.	Polarite.	Porous Carbon.	Alumino- Ferric.	Barry.	Lime.
Total solids at 212° F. in 100,000 parts ...	142	109	111	78	130	132	111
Suspended matter .....	15	..	..	..	6	5	3
Suspended mineral .....	8	..	..	..	4	3	2
Suspended organic.....	7	..	..	..	2	2	1
Total solids in solution.....	127	109	111	78	124	127	108
Total mineral .....	106	24	24	61	114	111	55
Total organic.....	21	85	87	17	10	16	23
Chlorine.....	38	32	30	19	38	39	48
Oxygen for 15 minutes .....	3.4	0.74	0.55	0.070	1.47	3.5	0.120
Oxygen for three hours .....	5.8	1.10	0.60	0.132	2.60	3.9	1.66
Free ammonia .....	1.5	0.89	1.0	0.026	2.25	1.3	0.72
Albuminoid ammonia .....	0.64	0.16	0.12	0.021	0.30	0.22	0.27

Whatever process may be adopted for the treatment of sewage, one important fact has to be considered, viz., the amount of the resultant "sludge." The following experiments were undertaken in 1883 in view of this important phase of the subject:—

Sulphate of alumina and lime have been tried with the following results:—

No. 1.—Sulphate of alumina was used in the proportion of 5 cwt. to 1,000,000 gallons of sewage; the weight of the precipitate dried at 212° F. was 1 ton 18 cwt.

No. 2.—6 cwt. of sulphate of alumina was used in the same amount of sewage; weight of dry precipitate was 1 ton 18 cwt. 1 qr.

No. 3.—7 cwt. of sulphate of alumina; weight of dry precipitate was 2 tons 3 cwt.

No. 4.—8 cwt. of sulphate of alumina; weight of dry precipitate was 2 tons 1 cwt.

The effluent water from No. 4 was very clear; there was a very marked difference between this and No. 1.

No. 5.—Freshly burnt lime was used in the proportion of 5 cwt. to 1,000,000 gallons of sewage; the weight of the dry precipitate was 2 tons 13 cwt.

No. 6.—6 cwt. of lime; weight of dry precipitate was 2 tons 14 cwt.

No. 7.—7 cwt. of lime; weight of dry precipitate was 2 tons 15 cwt. 3 qrs.

No. 8.—8 cwt. of lime to 1,000,000 gallons of sewage, gave a precipitate weighing 2 tons 17 cwt. 2 qrs.

The effluent water from the lime treatment had a very noxious odour, and was not so clear as that from the sulphate of alumina. The weight of the wet precipitate I should calculate at about 6 tons to 1,000,000 gallons.

In the following experiments the sewage was much more foul than the previous sample. This sample was quite black. In consequence I found that very small quantities of lime and sulphate of alumina had very little effect in producing a good effluent water.

No. 1.—A quantity of lime was used equal to 2 tons to 1,000,000 gallons; in five or six hours the precipitate had subsided; the effluent water was quite clear.

The weight of the precipitate dried at 212° F. was 3 tons 8 cwt. 2 qrs.

The effluent water contained in 100,000 parts:—Ammonia, 1.2; albuminoid ammonia, .1; total solid matter, 125.

No. 2.—A quantity of lime was added to equal 1 ton per 1,000,000 gallons; also 5 cwt. of sulphate of alumina. Precipitate weighed 3 tons 9 cwt. 2 qrs. The effluent water contained in 100,000: Ammonia, .80; albuminoid ammonia, .17; total solids, .63.

No. 3.—Sulphate of alumina was added 12 cwt. to the 1,000,000 gallons. In a few hours a clear effluent water was obtained. The weight of the precipitate dried at 212° F. was 2 tons 4 cwt. 3 qrs. Effluent water in 100,000 parts: Ammonia, .70; albuminoid ammonia, .05; total solids, .47. The effluent water, even after many days standing in the sunshine, did not give forth any bad odours; it was comparatively speaking odourless. I cannot say so much for the effluent water from the lime treatment: there was a very disagreeable smell even after some days standing.

Results of experiments showing the difference in weight of dried sludge by the lime and the ferozone processes, August 6th, 1889:—

Experiment.	Weight of Dried Sludge per Million Gallons of Sewage.		Relative Proportions in Weight of Dried Sludge.	
	Ferozone.		Lime.	
No.	T.	C. Qr. Lb.	T.	C. Qr. Lb.
1	5	19 0 0	11	6 0 0
			1.00	1.90
2	1	15 2 24	3	9 1 12
			1.00	2.22
3	1	1 2 4	3	9 1 24
			1.00	3.22
4	0	17 3 12	2	4 2 16
			1.00	2.50
	Average		-	-
				2.46



## DISCUSSION.

MR. GEORGE E. DAVIS writes:—"I am sorry that I am not able to be present at the adjourned discussion on Mr. Bell's paper, as I wished to say that he has been too hard upon the paper-makers. The paper manufacturers have been nearly the only people on the river who have seriously done anything to prevent their waste liquids from entering the water courses. In juxtaposition to them, let us place the bleachers: What have they done to prevent river pollution from their waste soda-ash liquor, from their lime-stews, and from their partly spent 'chemick'? Why do they not follow in the footsteps of their brethren, the paper-makers? In most cases the prevention of river pollution may be made a source of much private profit combined with public advantage, but the work must be done properly.

"The process of river purification must undoubtedly commence by prohibition of the entrance into watercourses, of solid matters in suspension in water. This by and bye will be recognised as *sine qua non*; but curiously enough section 20 of the Rivers Pollution Prevention Act of 1876 distinctly states that 'solid matter' shall not include particles of matter in suspension in water," so that by inference it is competent to put any quantity of solid matter into a river, provided it be in suspension in water. If any polluter, however, elects to do this, he will find that the Act of 1876 will not protect him, at common law, if it can be proved that he injures his neighbour lower down. The Act of 1876 is a dead letter, and a writ of the High Court is a 'premunire' to offenders who seek its aid.

"I am very pleased to find that Mr. Bell has emphasised the remarks, made in our book on 'The Irwell and its Tributaries,' respecting the Rivers Irk and Medlock. It is a disgrace to a city like Manchester to have two such open sewers flowing through such populous districts, but it is pleasing to know that the requirements of the Manchester Ship Canal will force a speedy cure for this."

MR. G. SISSON (Messrs. Peter Spence and Sons) said he had recently made a great many analyses of sewage and experiments on sewage purification dealing with large quantities, and consequently being able to obtain good average samples. He thought that it would be interesting to lay before the Society a few results on that more or less neglected portion of the analyses not mentioned in Mr. Carter Bell's otherwise exhaustive paper read at the last meeting, namely, the gases held in solution in fresh sewage and in the same sewage after keeping, with and without treatment by chemicals or otherwise, especially as regards the oxygen gas contained in solution, as this had a very important bearing upon any secondary or putrefactive changes taking place in the purified effluent from the sewage.

The following figures were obtained during the treatment of the Salford and Pendleton sewage by the aluminio-ferrie process:—

Fresh untreated sewage contained—

	Cc. per Litre.
Carbonic acid gas.....	18
Nitrogen.....	12
Oxygen.....	6

Some of the same sewage, after standing 24 hours *without any treatment*, contained—

	Cc.
Carbonic acid gas.....	21
Nitrogen.....	13
Oxygen.....	Nil.

The oxygen being completely absorbed by the organic matter.

After treatment of fresh sewage by the aluminio-ferrie process, samples of the continuous effluent were taken, and

kept in full glass-stoppered bottles from 16th October to 5th November last.

On 5th November these samples contained 5·5 cc. oxygen per litre.

Some of the same samples, kept as before, from 16th October to 1st December, contained 5 cc. oxygen per litre.

That is, whereas the untreated sewage lost all its free oxygen in 24 hours, the treated sewage, after being kept for six weeks, still contained five-sixths of its free oxygen.

This showed the importance of treating the sewage as soon as possible, in order to remove the organic matter before it had time to take up the oxygen. No noxious putrefaction can take place so long as any free oxygen remains dissolved in the effluent. It showed also the inadvisability of introducing any deoxidising materials, such as ferrous salts, sulphides, &c., which would absorb the free oxygen of the sewage, the oxygen so lost having to be replaced by filtration through oxidising materials, or by the addition of substances such as bleaching powder, manganates, &c. It was found that by treating fresh sewage with some precipitating substance which does not absorb oxygen, such as an aluminous compound, and allowing the precipitated matter to settle either continuously or intermittently, that the amount of dissolved oxygen in the effluent water is more than sufficient to oxidise the small amount of organic matter remaining in solution, the amount of oxygen absorbed by such organic matter being estimated by Tidy and Frankland's acid permanganate method in the usual manner, and this compared with the dissolved oxygen. To demonstrate this in a visible and practical way, samples of effluent were taken from some millions of gallons of sewage, treated as mentioned, kept in full glass-stoppered bottles from 16th October to 1st December, when an ordinary aquarium globe was filled up with a portion, in which were immediately placed some gold fish, where they have lived happily until the present time. The determinations of gas were made by boiling 1 litre of the sewage or effluent, kept boiling for not less than three-quarters of an hour, the gases given off being conducted into a graduated mercurial tube, brought to uniform temperature and pressure, the carbonic acid gas absorbed by the introduction of a small quantity of strong caustic potash solution, the oxygen absorbed by then introducing pyrogallin, the residual gas being taken as nitrogen. Dr. Dupré, in his report to the Medical Department of the Local Government Board, 1884, says: "A water which does not diminish in its degree of aeration during a given period of time, may or may not contain organic matter, but presumably does not contain living organisms. Such organic matter, therefore, as it may be found to contain by chemical analysis (permanganate or otherwise) need not be considered as dangerous impurity."

MR. R. F. CARPENTER asked if Mr. Sisson in his experiments had raised the effluent to anything like summer temperatures?

MR. SISSON: The temperature was raised to 65° F.

DR. G. H. BAILEY: Mr. Bell seemed to be satisfied so long as the effluent appeared clear. Had Mr. Bell made any determinations on microscopical examinations of the effluent after long exposure to the atmosphere.

MR. H. L. TERRY had frequently analysed the Medlock in Holme and found that the contamination was due to manufacturing refuse rather than to sewage. The total soluble solids varied from 30 to 80 grains per gallon, a large proportion of which was organic matter. This organic matter and the general greasy state of the solids in suspension was the great drawback to the adoption of the water for steam raising or other purposes in factories. He thought that the chemicals in the river neutralised the sewage, as the smell of the river in his district was rarely objectionable.

MR. CARTER BELL, in reply to Dr. Bailey, said he had taken determinations of the effluent, and had also made microscopical examinations, and when he found that the

albuminoid ammonia was in the second place of decimals, then he came to the conclusion that it must be a good effluent, especially if it was clear at the same time. They had not to seek impossibilities in these matters, all that was necessary was an effluent clear enough to turn into the Ship Canal. Alluding to Mr. Terry's remarks, he was of opinion that the Medlock was nothing more than an open sewer, and on many days was worse than the Salford sewage.

## Newcastle Section.

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Notices of Papers and Communications for the Meetings to be sent to the Local Secretary.

Meeting held in the College of Science, Thursday,  
December 4th, 1890.

MR. JOHN PATTINSON IN THE CHAIR.

## ESTIMATION OF AVAILABLE SODA IN COMMERCIAL CAUSTIC SODA.

BY JOHN WATSON.

As it has been my daily work for the past three years to conduct and supervise the testing in the caustic soda department of the Newcastle Chemical Works (which works now, I believe, produce more caustic soda than any other in the trade), I naturally feel much interested in Dr. Lunge's remarks on the estimation of available soda in commercial caustic soda.

I read the paper by Messrs. Cross and Bevan when published; but was previously acquainted with the fact that the alumina (as well as the soda) of sodium aluminate acts as a base when methyl orange is used as indicator, on titration. Whilst agreeing with the main contention of Cross and Bevan, in their paper on "The economy of pure caustic soda" (this Journal, 1889, 252) I felt at the time that these authors exaggerated the real error caused by the use of methyl orange as indicator for titrating ordinary samples of caustic soda. They mention one case where a sample contained three per cent. of alumina; now this is most exceptional for white caustic, many samples of caustic "bottoms" containing no more alumina.

From a great number of full analyses of white caustic, made in the past three years, I give the following tabulated result:—

Newcastle Chemical Company's Caustic Soda. (Average of 21 analyses.)		Other Makers' Caustic Soda. (Average of 17 analyses.)	
$\text{Na}_2\text{Al}_2\text{O}_4 = \text{Al}_2\text{O}_3$		$\text{Na}_2\text{Al}_2\text{O}_4 = \text{Al}_2\text{O}_3$	
	Per Cent.		Per Cent.
·07	·04	·17	·10

In one exceptional case we had a sample of white caustic made by a Lancashire firm which contained aluminate equal to 1·73 alumina per cent.; but this I have not included in the above average.

It is obvious, therefore, that in most cases the alkali in white caustic soda may safely be titrated in the cold (or just warm) with methyl orange. I tried the method with phenolphthalein, as used by Cross and Bevan, shortly after reading their paper, and with our 77 per cent. caustic, got practically the same result as by the test made with methyl orange in the ordinary way; but I considered the boiling, subsequent cooling and back-titration too complicated, at least in our case.

With caustic "bottoms," methyl orange should not of course be used as indicator. Over three years ago I tried experiments on this subject, the result of which was that we adopted the following method: after dissolving a weighed portion of the bottoms in boiling water, filtering from the insoluble residue, and washing thoroughly, the filtrate was boiled, litmus added and the solution titrated in the way usual when this indicator is used.

## Estimation of Available, or Soluble, Soda in Alkali Waste.

There is one point in this portion of Professor Lunge's interesting paper to which I feel I must take exception.

The Professor describes briefly the method, used at some Lancashire works, communicated to him by Dr. Hurter.

This method, I gather, is practically the same as has been in use for over 10 years (to my knowledge) in the laboratory of the Newcastle Chemical Works. In the brief description, on the proof sheet which we possess, there is, I think, a misprint: "boiling the solution to decompose the calcium carbonate" should read, I take it, "boiling the solution to decompose the calcium bicarbonate."

The chief point, however, that I wish to draw attention to is that, according to Dr. Lunge and Mr. Zalscecki, less than one hour's treatment with gaseous carbon dioxide is not sufficient. Possibly they have worked on a very much larger quantity of waste than we do. With a view to some member, or Dr. Lunge himself, helping to clear up this point, I will explain our *modus operandi*:—

Twenty grms. of waste are treated with about 150 to 200 cc. warm distilled water; after stirring and allowing to stand for about an hour, the clear liquid is decanted, and a stream of carbon dioxide passed through it, for three to five minutes.

The effect of this may be seen if watched: First, the carbon dioxide causes a milkiness, owing to precipitation of calcium carbonate, then the solution clears as this calcium carbonate is dissolved as bicarbonate, in the excess of carbonic acid, sulphuretted hydrogen is then evolved, and at this point we consider that enough  $\text{CO}_2$  has been passed through to convert lime existing as hydrate, or soluble carbonate, into bicarbonate.

We then evaporate to at least half the original bulk, generally lower, filter off, and wash the calcium carbonate, titrating the alkali in the filtrate with a weak standard acid, using methyl-orange as indicator. As our average for the past year has only been ·025 soluble soda per cent., there cannot be much, if any, alkaline lime present in the filtrate. That lime is present in this filtrate, I am aware; but it is, I believe, in a neutral state combined mostly as calcium sulphate. My reasons for this opinion are, that tank waste in the moist state contains about 1 per cent. of calcium sulphate. (An average of five analyses gave 1·09  $\text{CaSO}_4$  per cent.). Part of this will no doubt be dissolved on treating the waste for available soda, and I have tried an experiment which shows that an aqueous solution of calcium sulphate is not decomposed by a stream of carbon dioxide passed through it for 15 minutes. Calcium sulphate would, therefore, find its way into the filtrate, but, being neutral, would not effect the final titration. Since reading this portion of the paper, on Tuesday, I have had very little time to try experiments on this point; but my assistant, Mr. T. S. Laidler, has kindly made two determinations of the lime and the sulphuric acid in the filtrate from the aqueous extracts from two samples of waste treated as

above, and to-day I made one determination of each in a different sample. The results were:—

	$\text{SO}_3 = \text{CaSO}_4$		$\text{CaO} = \text{CaSO}_4$	
				Per Cent.
I.	·46	·78	·50	1·21
II.	·16	·27	·65	1·57
III.	·33	·56	·49	1·19
Average .....	·32	·54	·55	1·32

These results, it will be seen, do show an excess of lime over and above that required to combine with the  $\text{SO}_3$  to form  $\text{CaSO}_4$ , and so far this favours Dr. Lunge's recommendation; but we cannot base much reliance on these few experiments, and tank-waste has so many constituents that some other neutral lime compound may possibly be present.

This morning Mr. Laidler tried two portions of different samples of waste for soluble soda by our ordinary treatment, and at my suggestion he tried duplicate portions of these, passing a stream of  $\text{CO}_2$  through for a little over an hour. The results were as follow:—

#### SOLUBLE SODA IN TANK WASTE.

Sample.	A.	B.
	Per Cent.	Per Cent.
After passing stream of $\text{CO}_2$ for 3 to 5 minutes	·034	·034
After passing stream of $\text{CO}_2$ for 70 minutes ..	·030	·030

The results were, therefore, practically the same in both cases.

From the fact, stated before, that our average soluble soda in tank waste for the past year has only been ·025 per cent., my opinion is that even with 3 to 5 minutes "gassing" with carbonic acid, and with treatment as described, alkaline lime will be absent, or practically absent, in the filtrate when titrated.

#### EGYPTIAN SALT.

BY H. DROOP RICHMOND AND HUSSEIN OFF, KHEDIVIAL LABORATORY, CAIRO.

THE salt supply of Egypt is almost exclusively obtained from the beds of salt left by the evaporation of the water on the northern coast, and on the borders of the inland lakes of the delta: the salt district stretches from Mex, near Alexandria, to beyond Damietta, including the salt lakes Bourlos, Edko, &c., and going as far inland as Damahour. The collection and sale of this salt is a Government monopoly, and there is a large tax on this industry. As salt is found in considerable quantities on the mountains inland, there are inducements to the sale of the contraband article, and consequently it frequently devolves on the Khedivial laboratory to give an opinion as to whether specified samples are contraband or not. In some cases the quantity of impurities is so great in the contraband salts that the task is easy, but it was considered desirable to examine a large number of salts of the Government to see if the composition was sufficiently constant to enable us to lay down a standard. With this view, we obtained samples from each of the different centres of collection and from the different depôts of Cairo.

As we frequently have a considerable number of samples at one time, we have adopted a method which, while not pretending to be of absolute accuracy, gives a sufficiently exact analysis in a short time; it differs but slightly from the ordinary methods of analysis, but as the differences

economise time, we give it in full; 10 grms. of the salt are weighed out, dissolved in a small quantity of water, and evaporated to dryness with hydrochloric acid on the water-bath; the residue is taken up with dilute hydrochloric acid, and filtered into a 250 cc. flask, the residue being weighed as silica. Of the filtrate 100 cc. (= 4 grms. salt) is precipitated by means of a concentrated solution of barium chloride added drop by drop to the boiling solution, and the precipitate allowed to stand, when practicable, till next day. Another 100 cc. is made alkaline with ammonia, and without filtering off the precipitate of oxide of iron, &c. ammonium oxalate is added, drop by drop, to the boiling solution, and the precipitate of calcium oxalate and oxide of iron, &c. is filtered off; the filtrate and the first two washings are collected in a beaker, and sodium phosphate added to precipitate the magnesia, as usual, and this is allowed to stand till next day. In this way the bulk of the solution from which the magnesia is precipitated does not much exceed 150 cc., and does not contain too large quantities of ammonium salts; and as the amount of magnesia is small, the loss from incomplete washing is quite negligible. The precipitate of calcium oxalate, &c. is thoroughly washed, the washings being collected apart and thrown away, and is burnt at a low temperature and weighed as calcium carbonate and oxide of iron; it is then dissolved in a small quantity of hydrochloric acid, and the iron precipitated with ammonia and weighed, this quantity being subtracted from the total weight. Another portion of about 2 grms. of salt is carefully heated in a covered crucible over a small flame, to estimate the moisture.

In the calculation of the results we have departed from the usual custom of combining all the sulphuric acid with lime as calcium sulphate, but have preferred to express it as a double sulphate of soda and lime,  $\text{CaNa}_2(\text{SO}_4)_2$ ; our reasons for this are (1) we have observed several times that salts containing practically no magnesia, and lime in proportion about equal to the sulphuric acid present, are hygroscopic, a thing which could not be explained, unless we supposed that some of the calcium was combined with chlorine; (2) we prepared three samples of salt from the same liquor, one ( $\alpha$ ) by very slow crystallisation at the ordinary temperature, one ( $\beta$ ) at about  $35^\circ \text{C}$ ., and the third ( $\gamma$ ) as rapidly as possible at a boiling temperature; the results of the analysis of these samples are as follows:—

	Silica.	Oxide of Iron, &c.	Lime.	Magnesia.	Sulphuric Acid.	Moisture.
$\alpha$	·08	·03	·76	·05	·97	1·50
$\beta$	·02	·02	·55	·18	·84	5·26
$\gamma$	·01	·03	·50	·46	·45	3·42

As it may be safely assumed that the moisture, silica, and oxide of iron are accidental impurities, and liable to vary from extraneous circumstances, we have calculated the percentages on the salt as free from them, as follows:—

	$\alpha$	$\beta$	$\gamma$
Calcium sodium sulphate .....	1·71	1·52	·80
Calcium chloride .....	·84	·55	·60
Magnesium chloride .....	·11	·45	1·13
Sodium chloride .....	97·34	97·48	97·38
	100·00	100·00	100·00

It is thus seen that the higher the temperature at which the salt was prepared, the less is the amount of sulphuric acid, and were this all as calcium sulphate, it would be anomalous. We may add that each of the samples consisted of a considerable amount of salt, care being taken that approximately the same amount of salt was deposited in each case, and that the whole amount deposited

was mixed before analysis. So far we have not much evidence to prove that the sulphuric acid is combined as  $\text{CaNa}_2(\text{SO}_4)_2$ , all the facts indicating only that the sulphuric acid is not as calcium sulphate, but we have preferred to state our results as  $\text{CaNa}_2(\text{SO}_4)_2$ , as this salt is found deposited in the places where salt liquors are evaporated.

In the sample  $\gamma$  the proportion of sulphuric acid to sodium chloride is the same as in the liquor, while in  $\alpha$  it is more than twice as great.

The analyses of the salts are as follows, they are calculated as the salts present in 100 parts free from moisture, &c. :—

—	1.	2.	3.	4.	5.	6.
$\text{CaNa}_2(\text{SO}_4)_2 \dots$	·60	·60	·93	·37	1·42	·28
$\text{CaCl}_2 \dots\dots\dots$	·02	·33	·54	·06	..	·74
$\text{MgCl}_2 \dots\dots\dots$	·29	·39	·32	·45	·75	·10
$\text{MgSO}_4 \dots\dots\dots$	..	..	..	..	·26	..
$\text{NaCl} \dots\dots\dots$	99·00	98·68	98·21	99·12	97·57	98·88
	100·00	100·00	100·00	100·00	100·00	100·00

—	7.	8.	9.	10.	11.	12.
$\text{CaNa}_2(\text{SO}_4)_2 \dots$	·47	·14	1·07	1·37	1·65	1·25
$\text{CaCl}_2 \dots\dots\dots$	·90	·31	·02	·21	..	·12
$\text{MgCl}_2 \dots\dots\dots$	·98	·09	·91	·78	1·39	·86
$\text{MgSO}_4 \dots\dots\dots$	..	..	..	..	·80	..
$\text{NaCl} \dots\dots\dots$	97·56	99·46	98·00	97·64	96·16	97·77
	100·00	100·00	100·00	100·00	100·00	100·00

—	13.	14.	15.	16.	17.	18.
$\text{CaNa}_2(\text{SO}_4)_2 \dots$	1·22	1·18	1·20	1·42	1·22	1·09
$\text{CaCl}_2 \dots\dots\dots$	·27	..	·10	·02	..	..
$\text{MgCl}_2 \dots\dots\dots$	·39	·48	1·03	·55	·66	·18
$\text{MgSO}_4 \dots\dots\dots$	..	·12	..	..	·04	·33
$\text{NaCl} \dots\dots\dots$	97·92	98·22	97·67	98·01	98·08	98·40
	100·00	100·00	100·00	100·00	100·00	100·00

—	19.	20.	21.	22.	23.	24.
$\text{CaNa}_2(\text{SO}_4)_2 \dots$	1·41	1·17	·79	1·16	·87	·82
$\text{CaCl}_2 \dots\dots\dots$	·19	·09	..	..	..	..
$\text{MgCl}_2 \dots\dots\dots$	·66	·36	·30	·41	·25	·15
$\text{MgSO}_4 \dots\dots\dots$	..	..	·16	..	·19	·29
$\text{NaCl} \dots\dots\dots$	97·74	98·38	98·75	98·43	98·67	98·74
	100·00	100·00	100·00	100·00	100·00	100·00

—	25.	26.	27.	28.
$\text{CaNa}_2(\text{SO}_4)_2 \dots$	·87	1·15	1·15	1·53
$\text{CaCl}_2 \dots\dots\dots$	..	·21	·06	..
$\text{MgCl}_2 \dots\dots\dots$	·23	·45	·72	·53
$\text{MgSO}_4 \dots\dots\dots$	·25	..	..	·02
$\text{NaCl} \dots\dots\dots$	98·60	98·19	98·07	97·92
	100·00	100·00	100·00	100·00

From an examination of these results it is seen that the variations in the salts are too great to lay down a standard of purity. The moisture, as a matter of course, is of no use whatever in judging of the purity; the silica and oxide of iron, &c. are due to sand and dirt which have got mixed with the salt, and cannot be employed as indications, while the variations in the various salts are too great to give any reliable standard. The sulphate of lime and soda varies from ·14 to 1·65, the magnesium sulphate from 0 to ·80, the calcium chloride from 0 to ·99, and the magnesium chloride from ·09 to 1·39; nor do these different compounds occur in any fixed ratio.

We are therefore forced to the conclusion that the salt of the Egyptian Government has not a composition sufficiently constant to allow of standards being laid down.

### DISCUSSION.

Dr. BEDSON commented on the author's practice of stating the sulphuric acid present in the samples of salt as double calcium sodium sulphate, for the existence of which he did not consider there was sufficient evidence.

The CHAIRMAN pointed out that the plan of dissolving the salt with the aid of hydrochloric acid would cause any calcium present in the original sample as carbonate to be determined as chloride.

Dr. BEDSON afterwards exhibited and gave a demonstration of the working of Pettersson and Palmquist's portable apparatus for determining carbonic anhydride in the air, and also of an application, made by himself, of Lunge's volumeter to Dittmar's gas analysis apparatus.

## Glasgow and Scottish Section.

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Notices of Papers and Communications for the Meetings to be sent to the Local Secretary.

## SESSION 1890-91.

1891.

Jan. 5th (Glasgow).—Mr. H. Ballantyne. "On the Effect of Exposure, under Certain Conditions, upon some Constants of Oils."

Feb. 3rd (Edinburgh).—Dr. J. B. Readman. "The Manufacture of Phosphorus: Part III."

Mar. 2nd (Glasgow).—

(1.) Dr. E. J. Mills. "Destructive Distillation: Part II."

(2.) Messrs. R. T. Thomson and H. Ballantyne. "On the Revision of Constants Employed in the Analysis of Fats and Oils: Part II."

*Meeting held at the Philosophical Institution, Edinburgh, on Tuesday, 2nd December 1890.*

MR. C. BEILBY IN THE CHAIR.

## CONDENSATION OF CARBON PARTICLES IN SMOKE.

BY ROBERT IRVINE, F.C.S., F.R.S., ED.

IN the Society's Journal, May 31st, 1889, there is a short note (communicated by me) on the condensation of carbon particles in smoke; and as it represents the initial stage of the investigation, I make it the preface to the paper I am to communicate this evening on the same subject.

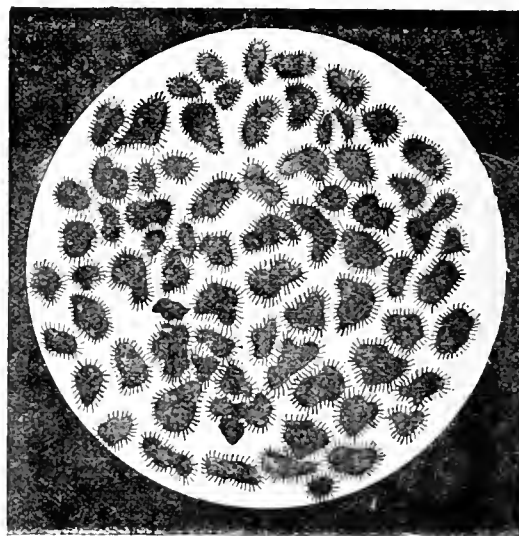
"In the manufacture of lamp black, hydrocarbons are burned in an atmosphere with a limited supply of oxygen; the resulting dense black smoke is conducted into large chambers, where, on account of the extremely sluggish draught, it is allowed to roll about until the particles, by attraction between themselves, gradually coalesce into masses, which after a lengthy period fall on the floor in the form of soot. This process is exceedingly slow, and the product obtained, from even an enormous condensing space, is comparatively very small. Consequently this manufacture is one carried on, on a limited scale, considering the magnitude of the plant required.

"I erected a glass structure 5 ft. x 4 ft. x 4 ft., in which were fixed two malleable iron plates, provided with a great number of points—these facing each other, the plates being separated by a distance of from two to three feet, and all the conducting surfaces, except these points, being carefully insulated with shellac varnish. This chamber was filled with smoke produced by burning pitch oil, which retained its opacity for at least two hours; so much so that on then looking through the chamber, a bright light placed on the other side was totally obscured by the vapour. The chamber was now refilled with smoke, and the whole atmosphere therein was to a greater or less degree electrified by coupling the conductors, which were connected to the plates with a small dynamo. The effect immediately produced was exceedingly striking, the minute particles of carbon at once separated from the opaque smoke, and were attracted to, or driven from, the points of the plates, congregating together in a most extraordinary manner; and in the space of two or three minutes the atmosphere in the glass chamber was almost entirely cleared of smoke. The prohibitory cost of electricity has however prevented the application of this process on a large scale, for the present at least."

This led me to infer that the condensation of the minute particles of carbon in smoke into concrete masses, could be effected by simpler and cheaper means. I carried out a series of experiments by which smoke was rapidly agitated by mechanical means. The results were practically the same as that obtained by electric disturbance.

Before proceeding to explain in detail these experiments, I wish to direct your attention to some of the properties of the products obtained when fuel is burned, more especially that known as coal.

When a microscopic slide is passed quickly over a smoky flame, a very thin semi-transparent film is left on the glass, which, when examined under a powerful microscope, presents the appearance of numerous particles of amorphous carbon, in a very finely-divided condition, each particle surrounded by an areola, or coating of oily matter.



MICROSCOPIC VIEW OF SMOKE PARTICLES SHOWING "AREOLA" OF HYDROCARBONS SURROUNDING THEM.

Here we have an explanation of two things. First, why a black fog may be and will remain persistently a fog while rain is falling. Each particle of carbon being, so to speak, surrounded by a waterproof coating and hence repelling moisture. Second, when the particles composing such a smoke fog are agitated, either by currents caused by changes of temperature, or by a vertical down-draught of air, this oleaginous coating causes the finely divided carbon particles to cohere, forming masses which the air cannot long support in suspension, and their condensation into greasy black smuts ensues, often with startling rapidity, and the fog clears.

If commercial lamp-black, or soot, which is the product of imperfectly burnt coal, is strongly heated, a large amount of empyreumatic matter is given off, which condenses on cooling into a brown greasy mass, consisting of crysene, pyrene, capnomar, &c., which causes ordinary lamp-black to cohere like damp snow when pressed, whilst after calcination (during which it has been deprived of the oily or greasy matter) it loses this property.

If we examine the smoke from a newly-made (or mended) fire, we find at first only light blue and yellow-brown coloured vapours given off (consisting principally of the solid and liquid hydrocarbons before referred to), these passing into the atmosphere, with which, if I may use the term, they "emulsify." This first stage is followed by the combustion of these products, which, as the coal becomes heated, are resolved into gases, which burn with a smoky flame. It is at this stage, owing to the imperfect combustion of these gases, that we have finely-divided particles of carbon formed, and the black smoke so produced is added to the grease-laden air of the first stage, and so long as the atmosphere into which these products are poured remains cold, damp, and still, what is known as a black or brown fog results.

In the last or perfect combustion stage, after all the volatile or bituminous matter has been driven off as gas and burned (represented by a red glowing smokeless mass of burning coke), we have nothing but carbonic acid and

water and some sulphurous compounds given off, which also pass into the air, but black fog cannot result from such products.

Taking the total products from a coal fire, consisting of permanent gases, carbon, greasy hydrocarbons, and water all together, the percentage amount of the carbon is very small; yet so great is its power of obstructing the light, that the amount present in the blackest fogs of our large towns is represented by the insignificant quantity of blacks or smuts which fall to the earth when the fog clears. This is estimated at 3 per cent. of the whole smoke.

We are aware that dense fogs disappear in many cases without apparent atmospheric changes or wind storms to drive away the smoke. This may be as already stated due to electrical disturbances or convection air currents, or to sudden rise of temperature in the atmosphere in which the smoke matter is suspended. In any and all cases however the effect is the same, viz., the agglomeration of the minute carbon particles into masses too heavy for the atmosphere to retain them in suspension; this agglomeration being due to the presence of hydrocarbons, which we have found to accompany or clothe each carbon atom in the smoke, and which causes it to deposit upon and defile buildings, trees, flowers, and other objects of beauty in and around our cities.

In a smoky town, when a breeze prevails, the smoke, in its horizontal passage through the air, gradually parts with its greasy-coated carbon to anything which impedes its progress, so that only a few miles away it loses its dolorous aspect, and assumes the soft dreamy haze so dear to the soul of the painter, but which, after all, is only an air emulsion of finely attenuated particles of grease.

This smoke and fog question, from an economic point of view, presents interesting features. The Hon. Rollo Russell, in a lecture delivered under the auspices of the National Smoke Abatement Institution, states that 20,000 tons of coal are consumed per diem in London alone. We will therefore, taking the solid carbon produced at only 3 per cent. of the coal burned, have 600 tons of smuts which will ultimately descend either on the city or in its immediate vicinity; and taking the volatile hydrocarbons at 10 per cent. of this amount of coal, we will, from the same source, have a daily pollution of the air to the extent of 2,000 tons of tar and other coal products.

As a chemical manufacturer, I sigh when I think of all this valuable material lost to us, either in the form of wasted heat producers or valuable chemical products in the shape of *aniline colours, ammonia, burning oils, paraffin wax, printing ink, &c.*, which, divorced from their natural channels, are floating about in the atmosphere, veiling the sweet sunlight and choking the lungs of both animal and vegetable life.

Of course, if we could overcome our sentimental desire for the cheerful though smoky blaze of the coal fire, and burn carbonised coal in our grates, these solid, liquid, and gaseous hydrocarbons would be saved and made profitable use of. In this case our chimneys might become ornaments to our houses, while the products of combustion would pass from thence as colourless gases.

But if we will insist on burning green coal, it is possible for us to do so and have the enjoyment of the blazing fire by constructing our house grates so that the green coal may be introduced at the bottom (as is now successfully accomplished in some forms of steam boiler furnaces) by which means the hydrocarbons given off from the heated coal are made to pass (with a regulated supply of air) up through the mass of incandescent coke which by this means would occupy the upper portion of the fire instead of the lower.

Many attempts have been made to introduce such a perfect combustion grate, but the usual condition of the atmosphere of our large towns proves that, as yet, either a scientifically constructed grate or one requiring extra care or involving extra attention on the part of the class who minister to the "smoke fiend," is far from general adoption.

In the coke and iron industries much has been done in the direction of utilising the gaseous and liquid products obtained from carbonised or partially coked coal, and

processes are in general use by which these instead of being wasted are now profitably made use of.

Efforts have been and are being made to introduce such carbonised coal (or coke) into use for household firing, and experience has proved its use to be twice as economical as ordinary coal, while it is impossible to produce smoke from it. But sentimental prejudices prevail over all such considerations as health, beauty, and what is more wonderful, money; nor will there be an end of the allied sins of waste and dirt which we so strongly cling to (and which in this connexion Russell in his pamphlet before referred to estimates, costs the city of London alone nearly six million of money sterling) until the law compels us to burn smokeless fuel either in the form of coke or gas.

Consequent upon the failure (from a money point of view) of the condensation of smoke particles by means of electric currents,\* as already explained I tried to obtain the same results as were then observed, by mechanical agitation of smoke, and I constructed a flue or chamber, partly horizontal and partly perpendicular, through which dense smoke, obtained by burning naphtha at the one end, was passed. In this chamber (which was a box about 20 ft. long and 1 ft. wide) was placed two sets of fanners, which were made to revolve near the point where the smoke entered, so that it might be driven about in the confined space, my idea being that under these circumstances the particles of oily-coated carbon would by their impact coalesce into large smuts and condense *in situ*.

On starting these experiments the smoke was allowed to pass through the apparatus undisturbed, and at the exit end of the flue a loose plug of white cotton wool was placed, so that the smoke had to pass through it without its exit being impeded. Two minutes after this plug was applied the amount of carbon condensed among the fibres had blackened it completely. A second plug replaced the first, which was again blackened in the same space of time. The fanners in the box were now set in motion, and what followed was observed through spy-holes in the box and flue, both behind and in front of where the fanners were at work. Before agitation there was the dense brown-black homogeneous cloud which the undisturbed smoke presented. After agitation we had an effect very similar to the breaking up of a mist scene among the hills; large veil-like black streaks taking fantastic shapes, and a continuous shower of large particles of carbon falling within the flue or chamber, a few feet from the point where the agitation of the smoke had taken place. At this stage another plug of white cotton wool was placed at the exit of the flue; after two minutes this presented only a grey appearance. A second and a third plug exposed for the same time to the process showed less and less colouration; whilst the plug applied at the end of eight minutes showed hardly any stain, showing that most of the carbon particles in the smoke had been condensed. The same end can be arrived at by employing air jets, so as to throw the smoke into rapid circulation. Of course the production of smoke was continuous during the experiments.

I have endeavoured to repeat this experiment with smoke free from empyreumatic products, but the difficulty is to get smoke in such a condition. When gas is used for the production of carbon in the shape of fine lamp-black, the smoke does not contain empyreumatic matter in any large proportion, but of course a large quantity of water-vapour accompanies the carbon particles; and as the heated gases and water become cold, each particle of carbon is surrounded by condensed water which acts in the same manner as already described, in causing the particles to coalesce into large masses.

We are all familiar with the result of introducing a cold body into a gas flame. For instance, if a white plate is placed over a gas jet we have at once deposition of carbon on the cold substance, which will not take place if the plate is heated to the same temperature as the flame. Advantage is taken of this fact in preparing a very fine variety of lamp-black. And in America where gas is given off in large quantity from the oil wells, a large trade has sprung up for the manufacture of what is called gas-black.

\* See this Journal, 1889, 377.



Simultaneously with my observations on this smoke cure question, Mr. Elliott of Queen Street, Cheapside, has introduced a means of smoke condensation, using an apparatus which virtually washes the smoke with water. I take the following descriptive account from a London paper.

"Mr. Elliott's apparatus virtually washes the smoke and converts it into innocuous steam, and turns the deleterious parts into lamp-black and other colouring material. It can be attached to any chimney or works without interfering with the ordinary operations. The process is simple. The smoke is drawn from the flue by a fan with a closed chamber in which a rapidly revolving paddle wheel acts like a washing machine. The black thick smoke which enters emerges as colourless steam, which leaves no smell on a cambric handkerchief. The transformation is complete."

Such appliances as Mr. Elliott's washer, and agitating smoke in chambers, may be adopted in factories, when the smoke may easily be deprived of its carbon and hydrocarbons (and even the carbonic and sulphurous acids may thus be profitably utilised). That is to say, there is now no difficulty in preventing the emission of coloured smoke from any factory chimney. Indeed, were the law to insist upon this being done, coal users would in the end find it to be to their profit. This remark applies equally to the largest factories and to the ordinary bakery. Perhaps then this is enough for the fulfilment of our duty as representing the Society of Chemical Industry. As yet we have been wholly unable to deal with the domestic smoke nuisance, which is almost as serious a problem to cope with as that of domestic drainage.

I need not further urge the immense importance of some means by which the enormous waste of valuable material may be prevented. Macanley, in a lecture delivered at Liverpool in February 1888, estimates the amount of coal wasted in this country per annum at 45,000,000 tons, costing 15,750,000*l.* at the pit mouth. We have seen in the experiments I made when smoke was agitated in a confined space, that it could be deprived of its carbon. The same thing can of course be effected by filtering the smoke through a porous material, or washing with water, but the adoption of any such plans as these would necessitate the entire reconstruction of our dwelling-houses, and is consequently not to be thought of, even if it could be practically carried out. The only real resource open to us seems to be in the use of a smokeless fuel in our domestic fire-grates, or a grate which will prove in every respect a successful smoke consumer where coal is used.

Did time permit, I would lay before you statistics as to the additional sum in money which would be saved, added to what at present is lost (in consuming bituminous coal) by adopting such a fuel or grate, and in addition an appalling catalogue of death caused to animals and plants, which would be avoided.

For information on these and other points I refer you to some excellent papers issued by the National Smoke Abatement Society, which deal not only with the evil but with the attempt to cure it.

#### NOTE ON SPONTANEOUS HEATING.

BY ROBERT R. TATLOCK, F.R.S.E., F.L.C., F.C.S.

THE tendency of many carbonaceous substances, both of an organised and structureless character, to combine with oxygen, under ordinary conditions, with evolution of heat, has been made the subject of much patient investigation, and a large mass of information relating thereto is now available. Inquiries in this direction, however, have had reference chiefly to the circumstances under which this phenomenon takes place, and to its prevention, with the view of obviating the disastrous consequences which it involves, when the temperature rises sufficiently high to char or to inflame goods of value. Much obscurity, however,

still hangs round the question of the nature of the products formed at the commencement and during the progress of the chemical action, and any work done with the object of throwing light on this point would probably be productive of valuable results. The observed slowness with which these substances combine with oxygen is more apparent than real, the time occupied being, not what is required for the action to take place, but rather what is necessary for the non-contiguous molecules to be brought into contact. There can be little doubt that if the ultimate molecules of these bodies were in contact with the necessary oxygen molecules in sufficient number, combination would be instantaneous, as in the case of metals in the pyrophoric condition.

Nor is it even known whether the comparatively small proportions of oxygen which take part in such action combine with the entire complex substance—an oil, for example—or only with some part of it, leaving the remainder intact. In some instances, however, the nature of the chemical changes has been very fully ascertained, as, for example, in the heating of barley in the process of malting, by which starch is converted into maltose and dextrin, with evolution of carbonic acid. The conditions which favour this low combustion are well known; they are, first, the presence of the carbonaceous body, which must always be one that would burn in the ordinary sense if heated to redness in air; second, the presence of air or oxygen; third, the presence of moisture; and fourth, at least a moderate temperature. All these being fulfilled, the mechanical condition of the substance is of the greatest consequence, as, if it is either in a porous or spongy state, or in a condition of fine division, the facility for heating is greatly increased, while a calm and still atmosphere aid very materially. The following examples illustrate the varied conditions under which this heating frequently takes place, particularly with regard to the nature of the material. A fire broke out, apparently spontaneously, in a workshop in Glasgow where "fire-lighters" were manufactured. These were prepared by mixing sawdust with melted rosin in certain proportions. On the following day some of the finished goods which had escaped injury were taken away for investigation. One pound weight of these were placed under a glass shade with a thermometer, and in less than an hour the substance burst into flame. An examination of the sawdust showed that it contained a large proportion of moisture when mixed with the resin, and farther experiments confirmed the view that this was the cause of the accident. Again, a quantity of weaver's "heddles," prepared, as usual, with boiled linseed oil, were sent from a factory in this town to Aberdeen. The railway truck which carried the goods took fire, to all appearance spontaneously, and there could be no doubt, from a careful scrutiny of the whole case, that the oil was the cause of the mishap, as the centre portion of the heddles, which alone was prepared with oil, was charred and carbonised, while the rest of the threads in the same bundle were intact. The same accident happened on two other occasions about the same time. Yet another instance:—A few hundred tons of dried fish guano were landed from a vessel at Glasgow harbour, and at once placed in bulk in a store with a stone floor. In a short time the material began to "heat" internally, in consequence of which it was opened out and spread more thinly over the floor by a gang of men, but while this was being done a large quantity of it suddenly took fire, and before the flames could be extinguished, a portion of the wooden beams of the ceiling was destroyed. The spontaneous heating and consequent conflagration of coal cargoes, especially those of large size, are too familiar to us to require any reference. If we assume that a cubic foot of coal weighs 75 lb., and a cubic foot of air 1·23 oz., and that for every three cubic feet of coal in hold, bunker, or store there is one cubic foot of air containing at least ·28 oz. of oxygen, we have conditions equivalent to 2 ewt. of coal, having probably a surface of 50 square feet, practically in contact with the ·28 oz. of oxygen, which, if it combined with carbon to form carbonic acid, would give out sufficient heat to raise the cubic foot of air from 60° F. to 5316° F., which is much higher than the temperature of any blast furnace. Of course in practice this temperature could not be attained by the combustion of the oxygen initially present, as the



heat would be partly communicated to the coal itself, and would partly escape with the warm air, for even if the atmosphere among the coal was dead still to begin with, the warmth imparted to it would cause it to expand, ascend, and pass away.

In order to determine satisfactorily whether it was the sulphur, the compressed or occluded gases, or the non-gaseous carbon compounds in coal that was responsible for spontaneous ignition, I took different kinds of coal—cannel and house coal—pulverised them finely, and after exposing them to the air for 24 hours to allow all contained gases to escape, dried them thoroughly at  $212^{\circ}$  F., and determined the percentage of sulphur existing as sulphuric acid in each. A weighed portion of each was then exposed to a temperature of  $350^{\circ}$  F. for  $1\frac{1}{2}$  hours, when it was observed that both samples had materially increased in weight, due, of course, to their having combined with oxygen. The sulphur in the state of sulphuric acid was then estimated in both, and found to be almost precisely the same as originally.

The following is a summary of the results:—

	Cannel Coal.	House Coal.
Increase of weight between $212^{\circ}$ and $350^{\circ}$ F. ....	2.00	1.20
Sulphur, as sulphuric acid, before heating. ....	0.07	0.04
Sulphur, as sulphuric acid, after heating. ....	0.19	0.10

It will be seen from these experiments that the increase of weight cannot be due to the oxidation of sulphur, as if it were there would have been a corresponding amount of sulphuric acid formed, which was not the case, and it could not arise from the oxidation of compressed or occluded gases, as even if these were not completely expelled by the minute pulverisation of the coal and exposure to steam heat, and had combined with oxygen, their products of combustion, being gaseous, would pass off at the high temperature, which would result in a *loss* rather than a *gain* in weight. It is this gain in weight which is the key to the whole question of the rationale of the process of spontaneous combustion in coal and other carbonaceous substances. The carbon or carbonaceous ingredients of the coal combine with oxygen, forming *fixed* substances, and so increase in weight by the amount of oxygen with which they have combined. Now, the amount of oxygen which entered into combination in these experiments was approximately 2 lb. to every 100 lb. of coal, and was capable, if utilised for the ordinary combustion of carbon, of giving off heat nearly equal to what is evolved when 1 lb. of coal is burned. These results, moreover, make it clear that whatever part the contained gases take in aiding the action at the commencement, they are not required to carry it on, as their combination to form new products could result only in loss of weight, and not gain.

It has often been stated, and with truth, that chemical analysis throws no light whatever on the liability of a coal to spontaneous combustion, but while this statement is correct for such analyses as are usually made, it by no means follows that a chemical examination of the coal carried out on proper lines might not be useful in determining its qualities in this respect. The analysis required for technical and commercial purposes is one showing the percentage of carbon, hydrogen, oxygen, nitrogen, sulphur, ash, and moisture, but the relative proportions of these are not and never were intended to have any reference to the safety or otherwise of a coal for shipment or storage, and it is only such tests as are made with that special object in view that can ever be of value for that purpose. But while the elementary composition of a coal gives us no information on this point, it is extremely probable that a knowledge of its proximate constituents or compounds would be of great service. These are the decomposition products of the woody matter from which the coal was formed, but unfortunately little or nothing is known of them

except that they differ widely in different coals, some of them being unstable in their nature and consequently liable to chemical changes with production of heat, while others are of a stable character, and therefore not prone to such change. The subject affords a wide and interesting field for investigation, both for the scientific and the technical chemist. It is not at all unlikely, however, that experiments on the small scale, such as those described, carried out upon the principal coals intended for shipment might indicate the degree of their liability to heat and ignite spontaneously. There are also other points worthy of attention, such as the amount of moisture which a coal retains after being air-dried. Many hold as much as 10 per cent. retained at  $60^{\circ}$  F. and expelled at  $212^{\circ}$  F., and it is reasonable to suppose that water may either take part in bringing about a chemical change, or that any portion of it which would be expelled by a moderate heat of say  $100^{\circ}$  F. to  $110^{\circ}$  F. would leave the coal to some extent in a spongy or porous condition, which would favour oxidation.

The spontaneous heating of oils in a finely divided state has been made a subject of observation among chemists and others for a very long period, and the conditions under which it takes place are too well known to require any reference. All that is necessary is that the oil should expose a large surface to the air, and if the latter is warm and still, the action commences with great ease and progresses with astonishing rapidity. Numerous and varied experiments have shown that some oils are far more prone to oxidation and heating than others. Among vegetable oils, linseed stands prominent in this respect, and of animal oils, seal shows the greatest tendency, while mineral oils not only show no disposition to heat, but when mixed in comparatively small proportions with heating oils, annul or neutralise the heating proclivities of the latter. Attempts based upon the results of spontaneous combustion experiments have been made to arrange vegetable and animal oils in the order of their liability to heat or to combine with oxygen, but their behaviour under experiment is far too fickle to allow of any but a very rough classification, admitting of certain oils being placed at or near the top of the list, others at the foot, and the remainder somewhere intermediate. It has been observed by Mr. R. T. Thomson, however, that those oils which are capable of combining with the largest proportion of iodine are precisely those which have the greatest affinity for, and combine with, the greatest amount of oxygen under favourable conditions; and there can be no doubt of the correctness of this view.

It has been, and still is, the result of painful experience, that many of the most destructive conflagrations owe their origin to the affinity of certain oils for oxygen, although there must be some instances in which this has been wrongly assigned as the cause. It is clear that any fire having its origin in a building such as a Turkey-red stove, where goods prepared with oil are suspended at a high temperature for a considerable length of time, would in all probability take place in one of three ways:—

*First.*—By the elimination of combustible vapour from the oil by the heat of the stove, producing an inflammable atmosphere, which would at once ignite on contact with a spark or flame.

*Second.*—By a spark escaping from a chink or crack in the heating flue of the stove (or brought into the apartment in some other way) coming in contact with the hot and dry goods.

*Third.*—By the extra heat—over and above that of the stove—generated by the oxidation of the oil; this would promote further action, developing more heat, and so the action would proceed, in an accelerating ratio, till the ignition point was reached, immediately before which a large quantity of inflammable vapour would be distilled off from the highly heated portions of the oil surface.

The first of these causes may be dismissed, as the amount of gas or vapour which could possibly be thrown off from the oiled goods would not be sufficient to form an explosive or inflammable mixture with the air, nor such as would ignite by contact of either spark or flame. The second could not and need not be made the subject of experiment,

as the effect of direct contact with fire is obvious. It is only the third of these probable causes, therefore, that could form the subject of direct inquiry, and for this reason, as well as because it seemed probable that the majority of disasters arose from this cause, I thought it desirable to institute a few experiments, with the object, not so much of throwing light upon this point, as of determining in what direction investigation might proceed.

In order to ascertain whether olive oil gives off an inflammable vapour such as would ignite on the application of a flame, or spontaneously at the temperature of a Turkey-red stove, say  $180^{\circ}\text{F.}$  to  $184^{\circ}\text{F.}$ , I prepared some grey cotton yarn by soaking it in a well-known brand of olive oil (Gioja), pressing out the excess until only 10 per cent. remained, and heated this in an Abel's flash-point apparatus for some hours at  $200^{\circ}\text{F.}$ , but no vapour was given off that would flash or ignite on the application of a light at any period of the trial. The same was tried on the "fatty acids" of olive oil, with the same negative results, which makes it certain that both olive oil and the fatty acid recovered from it are safe as far as this point goes. All the materials, both yarn and oil, were carefully weighed before and after in these trials, in order to ascertain whether there was any increase of weight due to oxidation of the oil at the high temperature, but the results were unsatisfactory, the weights fluctuating on account of the hygroscopic character of the cotton fibre, and the difficulty of getting accurate weighings without absorption of moisture from the air, which vitiated the results. Recourse was then had to heating the dry oil to a temperature of  $194^{\circ}\text{F.}$  alone, without any fibre, but merely spread in a thin film over the flat bottom of a platinum basin, with the result that in 24 hours there was a clear gain in weight of nearly 1 per cent., which steadily increased until a point was reached, at the end of 60 hours, at which it appeared to remain stationary, probably on account of some decomposition and evolution of vapour, thereby causing a loss in weight which would neutralise the gain in weight by oxidation. The oil was purified beforehand by removing from it the free oleic acid, which all olive oils in the market contain more or less. The fatty acids from oil were then tried in the same way, but instead of showing a gain, like the oil itself, they showed a loss nearly to the same extent, but this was no doubt due to a chemical change producing some volatile body which passed away, and that this more than counterbalanced the gain in weight by oxidation, which was really going on at the same time. The fatty acids of cotton-seed oil behaved in the same way, but those of linseed oil showed a gain of over 1 per cent. in 24 hours. Stearic acid, on the other hand, remained almost constant in weight after exposure for 50 hours.

Now, at first sight it may appear that as the fatty acids of olive, cotton, and other oils lose weight on being kept hot in a close place, the vapour or substance given off being inflammable, would give rise to an atmosphere that would "flash" on contact with a spark or flame, and that therefore these would be dangerous from that cause, but a little examination will show that this cannot be the case. Suppose an example in which a ton of fatty acids of olive oil was thinly spread over the surface of cotton goods in a stove  $60 \times 40 \times 30\text{ ft.}$  at  $182^{\circ}\text{F.}$ , and that the weight of volatile vapour given off in 24 hours was 1 per cent., the conditions would be 5,600 lb., or thereby, of air through which were diffused 22.4 lb. of combustible vapour or gas, which would be equal to 0.4 per cent. Such a mixture would be perfectly safe to carry a naked flame through, as it would neither take fire nor explode. About  $2\frac{1}{2}$  per cent. of the vapour would require to be present before a combustible or explosive mixture would be reached. The real danger lies in the accumulation of heat produced by the oxidation (involving increase of weight) of the oil. In the above example approximately 1 per cent. of oxygen would be taken up by the oily matter in 24 hours, which, if combined with carbon by combustion in the ordinary way, would give out heat sufficient to raise the whole air of the stove to a temperature of  $273^{\circ}\text{F.}$ ; but then the heat, as it was generated, if not removed, would at once become available for promoting further action, producing more heat, and so on until finally the ignition point was reached. But it

must not be forgotten that the heat generated would greatly exceed this, as every successive development of heat by the combination of the oxygen with the elements of the fatty acid would facilitate or accelerate oxidation, resulting in the production of more heat, and so on, till the firing point was reached.

## NOTES ON GALLOTANNIC AND GALLIC ACIDS.

BY J. NAPIER SPENCE.

I HAVE ventured to bring these notes before the Society, feeling the want of a test for gallotannic acid in presence of gallic acid.

I used a mixture of pure gallotannic and gallic acids as my reagents.

On addition of lead acetate to a solution of gallotannic and gallic acids, they are both precipitated as lead tannate and gallate, and according to Guyard, on addition of acetic acid the lead gallate is dissolved up, leaving a precipitate of lead tannate. I find, however, that the difference in solubility of lead tannate and gallate in acetic acid is so slight as to make it impossible to dissolve the one without the other.

Lead nitrate precipitates gallotannic acid and not gallic acid, but the lead tannate is soluble in gallic acid. Therefore, if you have a mixture of the two acids you get no precipitate unless the gallotannic acid is in excess, when the excess only is precipitated.

Alkaloids, gelatin, and starch precipitate gallotannic acid in presence of gallic acid only if the gallotannic acid is in large excess, as the precipitates are soluble in gallic acid. Ostermeyer, improving on Wagner's process, uses cinchonine and magenta as an indicator. Now, you get no reaction with cinchonine and gallotannic acid in presence of gallic acid, owing to the solubility of the precipitate of cinchonine and tannin in gallic acid. Magenta precipitates both gallotannic and gallic acids, so cannot be used as an indicator.

Ammonia cupric sulphate will precipitate gallotannic acid in presence of gallic acid and therefore makes a good test, as, by filtering off the cupric tannate you can precipitate the gallic acid from the filtrate.

Gerland's process uses a standard solution of tartar emetic in presence of ammonium chloride, which is supposed to prevent the precipitation of gallic acid. Now, I find that a solution of tartar emetic does not precipitate gallic acid unless the gallic acid solution is very strong, when it is not prevented by ammonium chloride.

Ammonia sulphate of nickel may be used as a test for gallotannic acid in presence of gallic acid, its action being similar to ammonio-cupric sulphate. As a colour test potassium cyanide is very delicate. With a solution of gallic acid it yields a bright crimson solution, and has no action on gallotannic acid if added to separate solutions of the respective acids, but if they are mixed it turns the crimson colour yellow, and by using a standard solution of gallic acid and potassium cyanide and matching the colour yielded by the sample with a standard solution of tannin, a fairly accurate result might be arrived at.

In the Chem. Soc. J. for August there appeared an abstract from the Compt. Rend. 110 (532-534) on the Volumetric Estimation of Tannin, by E. Guenez, which was as follows:—

"When a solution of tannin is added to a boiling solution of tartar emetic mixed with certain coal-tar colours, antimony tannate is precipitated and carries down the colouring matter in the form of a true 'lake.' After a sufficient quantity of the tannin has been added, the liquid above the precipitate becomes quite colourless. Gallic acid does not interfere. Green dyes only are suitable, and Poirrier's green 1JE gives very good results.

"Having found tartar emetic to precipitate gallic acid, I thought perhaps the colouring matter might prevent it, but tried it with the following results:—

"I made up the solutions as directed, following his instructions exactly.

"I took 10 cc. of the dye solution and boiled. I then ran in 1 cc. of the tannin solution when I got a curdy green precipitate.

"I went on boiling and adding 1 cc. as directed, but having reached 20 cc. I thought I would add 10 cc. as I could see a distinct colour in the liquid above the precipitate.

"At this stage the precipitate seemed to change in form (from a curdy mass to a slimy powder), and although I went on adding the tannin solution until I had reached 50 cc., I failed to get the liquid above the precipitate colourless, and on testing I found the solution to contain excess of tannin."

There seem to be two distinct forms of precipitates formed, and they, especially the second form, do not fall readily, as, according to Guenez, they should do.

By using a mixture of gallotannic and gallic acids I failed to get any precipitate, but, on adding the gallotannic acid in excess, the excess was precipitated.

I am indebted to the kindness of Dr. E. Kuecht, of Manchester, for the sample of Porrier's green, 4 J.E., which I used to test the above process.

## Journal and Patent\* Literature.

### I.—GENERAL PLANT, APPARATUS, AND MACHINERY.

#### PATENTS.

*Means for Treating Spent Grains or other Analogous Material for the Separation of Liquid therefrom.* G. Thornley and T. Buxton, Burton-on-Trent. Eng. Pat. 16,891, October 25, 1889. 4d.

THE object of this invention is to provide means for separating liquid from spent grains or other material in a rapid, efficient, and economical manner. This is effected by a double process consisting in first pressing the grain and then in drying it, so that any desired quantity of the remaining moisture is driven off. The press suitable for the purpose is described in Eng. Pat. 13,259 of 1886. In drying, the grains are discharged from the press on to an endless web and are then carried through a chamber which is heated by a current of hot air, the extent of drying being regulated by the speed at which the web travels through the chamber. Millburn's drying machine is suitable for the purpose.—E. S.

*Improvements in Apparatus for the Evaporation of Fluids.* J. G. Chamberlain, Tipton, and G. E. Davis, Manchester. Eng. Pat. 17,938, November 11, 1889. 8d.

THIS patent relates to apparatus for the evaporation of liquid in a partial vacuum, a water-jet condenser being employed for producing the vacuum by the withdrawal and

simultaneous condensation of the resulting vapour. The evaporating liquid is heated by pipes traversed by steam fitted in the lower part of the vessel, the condensed water being collected and emitted through a steam-trap. The concentrated liquid overflows through a pipe from the evaporator into a tank placed sufficiently far below to admit of the vacuum being maintained through the luted descending pipe. Or the overflow may go into a second vessel, and may from that overflow into a third and so on, the operation being repeated as often as required with the aid of appliances for maintaining the desired flow of the liquor. Some variations are described.—B.

*Improvements relating to Apparatus for Evaporating, Concentrating, and Distilling Liquids.* R. A. Robertson and W. J. Mirkles, Glasgow. Eng. Pat. 20,784, December 27, 1889. 6d.

THE improvement consists in reversing the usual method of operating in multiple-effect apparatus, that is, the liquid to be operated upon is led first through that vessel of the series in which the temperature and pressure are the lowest, leaving at that where they are the highest. Steam is admitted at the end of the series, and flows in a direction contrary to that of the liquid. The concentrated liquid and products of condensation are discharged through pipes at a high temperature, and are therefore in a more liquid state, and the tendency to crystallisation is thereby reduced.

—E. S.

*Improvements in Apparatus for Distilling, Evaporating, or Concentrating Liquids or Solutions and the like, and for Condensing Exhaust Steam.* J. Wright, Westminster. Eng. Pat. 20,954, December 31, 1889. 8d.

THIS invention is to provide an apparatus for efficiently utilising the heat of steam for various operations by what is known as "multiple-effects," and can be adapted to the treatment of the liquors of soapworks, or sewage, or salt water, &c., and also for producing pure condensed water for wool-washing, dye-works, and other purposes. Four cylinders, preferably of a vertical form, are united with each other by suitable pipes and valves. Each cylinder has a chamber at the bottom from which inverted U-shaped tubes extend upwards; the cylinders constitute the evaporators, and the tubes the condensers. The upper part of the cylinder is provided with a steam strainer. Steam or vapour from the liquid in each cylinder passes from it into the chamber below the next cylinder, and into the inverted tubes, and so on through the whole series, until it arrives at the surface condenser, where its complete condensation takes place. The mode of construction is fully shown on the two sheets of drawings which accompany the specification.—E. S.

*Improvements in Filtering Machines.* J. A. Crocker, Brooklyn, U.S.A. Eng. Pat. 5446, April 10, 1890. 8d.

THE construction of this filtering apparatus includes a horizontal cylinder having hollow trunnions in the ends. The ends are partly double and have perforated pipes secured to the inner walls thereof reaching from one head to the other and communicating with the central trunnions by separate passages. The trunnions are fitted with two sets of circular valves connected by rods and sleeves in the centre of the cylinder, and so arranged that one passage may be open whilst another may be shut, and that both sets may be worked either simultaneously or separately, for the purpose of cleaning the pipes and passages, without stopping the apparatus. The claims are for the details of the arrangement, and the specification and two sheets of drawings should be consulted for further particulars.—B.

\* Any of these specifications may be obtained by post, by remitting the cost price, plus postage, to Mr. H. Reader Lack, Comptroller of the Patent Office, Southampton Buildings, Chancery Lane, London, W.C. The amount of postage may be calculated as follows:—

If the price does not exceed 8d. ....	½d.
Above 8d., and not exceeding 1s. 6d. ....	1d.
" 1s. 6d., " " " 2s. 4d. ....	1½d.
" 2s. 4d., " " " 3s. 4d. ....	2d.

*Improvements in the Manufacture of Filtering Plates, in Apparatus for Testing the same, and in Filtering Apparatus.* W. L. Wise, London. From A. Egen, Schwerte, Westphalia, and E. Bassege, Berlin, Germany. Eng. Pat. 8614, June 4, 1890. 11d.

THE inventors produce filtering plates from a mixture of powdered coke or "cylinder coal" with tar and pitch, which is heated to plasticity and pressed into flat circular moulds. The mixture recommended consists of 100 parts by weight of tar, 40 parts of stone pitch, and 330 parts of sifted powdered coke or coal. In the middle of the moulded plate, across its whole thickness, is embedded a disc of coarse textile web or netting, not reaching quite to the circumference of the plate, and finished off all round with a rather thicker cord or strand than the remainder of the material. When so far completed, the plates are stacked in an iron vessel in a mixture of coal dust and sand all between and around them, and the whole is then exposed for about a fortnight to glowing heat in a furnace. After careful cooling the plates are removed and are found hard and porous, and are traversed in the interior by a number of small channels, communicating with a larger circular channel, where the textile material has been burned out. Two holes are then drilled into the edge of each plate at opposite diameters, reaching to the circular channel inside, whereby through communication is established. To construct a filter, a number of these plates are placed one above the other with spaces between, short tubes are inserted into the end holes, and all tubes on each side connected by a collecting pipe for the removal of the filtered liquor. The stack of plates is inserted in a cistern containing the liquor to be filtered, the purified water passing away through the collecting pipes. The deposit collects on the outer surface of the plates, whence it can be cleared off either by inverting the current of the pure liquid or by blowing steam through, or in any other approved manner. The specification describes fully the method of manufacturing the plates, the mode of, and apparatus for, testing them as to their soundness, and the construction of filters with the finished plates. There are four sheets of drawings and five claims.—B.

*Improvements in Distillation and in Apparatus therefor.* A. W. Ellis, London. Eng. Pat. 12,260, August 5, 1890. 11d.

IN his distilling apparatus the inventor makes use of a number of shallow vessels of rectangular section in plan. He places them one upon another, with suitable connexions and openings to allow of the liquid to descend and the vapour to rise through the system. The liquor in the vessels is heated by steam contained in coils laid just above the floor of each vessel, each coil consisting of a number of straight pipes slightly inclined in one direction, connected at their ends by cross-pipes, through one of which the steam enters whilst the condensed water leaves by the other and is conducted back to the boiler by gravity. Various modifications are indicated, as well as details and proportions of parts. There are three sheets of drawings and seven claims.—B.

*Improvements in Machinery for Effecting the Softening of Water and for the Clarification of the Softened Water.* C. E. Gittens, London. Eng. Pat. 12,533, August 11, 1890. 6d.

THE object of this invention is to effect, by automatic machinery, the softening of hard water as it is supplied to a tank or reservoir, in which the precipitate is allowed to subside. When space is an object, a subsidence filter of coke or other substance may be used to clarify the water from any precipitate in a more rapid manner. For this purpose the water, as it flows from the inlet, actuates a water-wheel which drives a pump (or an elevator with buckets) which can be adjusted to raise the proper proportion of the solution used for softening the water, and to deliver it into the tail-water running away from the wheel, so that it is intimately mixed with it.—E. S.

*Improvements in Apparatus for the Complete Evaporation of Solutions.* H. E. Hansen, Copenhagen, Denmark. Eng. Pat. 14,059, September 6, 1890. 6d.

THE object is to obtain an exceedingly rapid evaporation in vacuo of all kinds of solutions. The apparatus consists of a vacuum vessel inside which a double-walled funnel is caused to revolve slowly at some distance from the sides of the vessel. Both the inner and outer surfaces of the funnel are heated by steam admitted into the space between its walls. The rapidity with which evaporation takes place causes the product to be of such a light and porous nature that, e.g., in the case of malt extract, actual pulverisation is not needed, as merely grating it reduces it to a powder.—E. S.

*Improvements in Method and Apparatus for Producing Mineral Wool.* W. H. Kennedy, Etna, Penn., U.S.A. Eng. Pat. 14,846, September 19, 1890. 8d.

THE improvement consists in spreading out a regulated quantity of molten slag, which is contained in a cast-iron tank, preferably capable of holding about 15 gallons, in a fan-shaped stream, to the action of steam issuing from a series of round inclined perforations in the front of a steam chest, by which means the slag is reduced to "the greatest number and smallest possible fragments of fine fibre or wool, thereby increasing the quantity and improving the quality without increasing the power used." There are eight claims.—E. S.

## II.—FUEL, GAS, AND LIGHT.

*The Effect of Temperature on the Limits of Explosions of Combustible Mixtures of Gases.* J. Roszkowski. J. für Gasbeleuchtung, **33**, 491—497, 524—527, 535—537, and 553—558.

THIS is a lengthy account of experiments carried out under Dr. Bunte in the chemical laboratory of the technical school at Karlsruhe. It is prefaced by an account of the researches of Favre and Silbermann, Thomsen, and Berthelot on heats of combustion; of Bunsen and Deville on temperature of combustion and on dissociation; and of Bunsen, Berthelot, and Le Chatelier and Mallard on temperature of ignition and rapidity of explosion.

The author uses the term "limits of explosion" in the following sense: A combustible gas is capable of explosion not only when mixed with the exact quantity of oxygen required for its complete combustion, but also when the supply of oxygen is in excess of this. (This applies also to common air or mixtures of oxygen with other inert gases.) When the proportion of oxygen in the mixture is gradually increased, the explosion becomes weaker, and at last a point is reached at which the mixture can no longer be exploded. This is called the *lower limit of explosion*; it is that which corresponds to the smallest percentage of combustible gas. Similarly, if the proportion of oxygen present be gradually diminished, a point will be reached at which the mixture will be incapable, or only just capable, of explosion. The border-line separating the two is called the *upper limit of explosion*; it corresponds to the largest percentage of combustible gas. These limits have already been investigated by Bunsen (for electrolytic gas diluted with oxygen and carbon dioxide), Frankland (London coal gas), E. v. Meyer (hydrocarbons—upper limit only), and Lehmann and Wüllner. Wagner has shown that a mixture which cannot be exploded by a spark from a small induction coil may be exploded by a spark from a stronger coil. No experiments have hitherto been made on the effects of pressure and initial temperature. The author has investigated the latter point, which is of considerable

practical importance. In the cylinder of a gas engine, for example, there is a series of explosions occurring in which the combustible mixture is initially at a high temperature.

The experiments (over 1,000 in number) were made at the temperatures 15°, 100°, 200° and 300°. The combustible gases investigated were hydrogen, carbon monoxide, marsh gas and coal gas.

**I. Hydrogen.**—A. *Hydrogen mixed with oxygen.*—At the ordinary temperature (15°) the lower limit is a mixture containing 1 vol. of hydrogen to about 9·25 of oxygen: in other words, the mixture will still just explode when it contains 18·5 times as much oxygen as is required for the complete combustion of the hydrogen. The upper limit is 1 vol. of hydrogen to 0·1013 of oxygen at 15°, and 1 to 0·0654 at 300°. Thus rise of temperature extends the upper limit; it scarcely affects the lower limit. B. *Hydrogen and air.*—Here, again, rise of temperature causes no noticeable change in the lower limit (excess of air), whereas it extends the upper limit from 0·1147 at 15° to 0·0548 at 300°. (The limits are always expressed in terms of the volume of oxygen required per unit volume of the combustible gas, or else in percentage by volume of combustible gas in the mixture.) C. *Hydrogen and a mixture of oxygen and carbon dioxide.*—This mixture contained 21 parts of oxygen to 79 of carbon dioxide (always by volume). It might be regarded as common air in which the nitrogen was replaced by carbon dioxide. The experiments showed that generally the limits of explosion were even more restricted by dilution with carbon dioxide than by dilution with nitrogen. Up to 100°, however, the limits were somewhat extended (especially the upper one). Further rise of temperature restricted the range, so that a mixture which was explosive at 15° or 100°, would no longer explode at 200°, and one which would explode at 200° would not explode at 300°.

**II. Carbon Monoxide.**—A. *Carbon monoxide and oxygen.*—At 15° the maximum amount of oxygen that could be added was 5·41 vols. to 1 vol. of carbon monoxide, i.e., 10·8 times the theoretical amount. The minimum amount of oxygen necessary was 0·066 vol. These correspond to mixtures containing 15·6 and 93·8 per cent. of carbon monoxide respectively. The range increases from 0° to 100°, and from 100° to 200°, but from 200° to 300° it diminishes, the limiting values at the latter temperature being 3·140 and 0·086 vol. of oxygen, or 21·4 and 92·5 per cent. of carbon monoxide. B. *Carbon monoxide and air; and, C., carbon monoxide mixed with oxygen and carbon dioxide* (see I.—C.). The results in both cases were similar to those obtained when oxygen alone was used. The author repeatedly found that mixtures which could be exploded at the ordinary temperature would not explode if they had previously been heated to 300°. He attributes the restriction of the limits (especially the upper limit) at high temperatures to a gradual union of the combustible body with the oxygen at these temperatures. This is supported by the appearance of a bright blue aureole around the sparking wires, and also by actual analysis of mixtures before and after heating.

**III. Marsh Gas (Methane).**—A. *Marsh gas and oxygen.*—At 15° marsh gas requires at least 0·7517 of its volume to form an explosive mixture with oxygen. It can support an admixture of 14·7 vols. of oxygen, or about 7·3 times the amount required for its complete combustion according to the equation  $\text{CH}_4 + 2 \text{O}_2 = \text{CO}_2 + 2 \text{H}_2\text{O}$ . Rise of temperature scarcely affects the limits; this is true also for the next mixtures (B. and C.). B. *Marsh gas and air.*—The mixture must contain about twice as much oxygen (1·323 vol.) as when pure oxygen is used. It still explodes when it contains 3·29 vols. of oxygen or 15·65 vols. of air to 1 of marsh gas. C. *Marsh gas with the same mixture of carbon dioxide and oxygen.*—Admixture with carbon dioxide restricts the limits much more than admixture with nitrogen. They lie very near together, the extremes being mixtures containing 9·0 per cent. and 11·6 per cent. by volume of marsh gas. This nearly corresponds to the theoretical quantity required for combustion.

**IV. Coal-gas.**—The gas used was from the town supply of Karlsruhe.

The composition of the gas at the commencement and end of the experiments is shown in the following table:—

	Before Feb. 22, 1890.	After March 4, 1890.
Hydrogen.....	Vol. per Cent. 50·6	Vol. per Cent. 48·6
Marsh gas .....	31·8	33·6
Carbon monoxide .....	6·2	6·5
Hydrocarbons (ethylene, benzene, &c.) .....	5·3	5·3
Carbonic acid.....	1·9	2·1
Nitrogen (residue).....	1·2	2·8
	100·0	100·0*

\* In original.

A. *Coal-gas and oxygen.*—At 15° coal-gas requires at least 0·4358 of its volume of oxygen to form an explosive mixture. It can support admixture with 12·25 vols. of oxygen (the amount found by experiment to be necessary for complete combustion being almost exactly 1 vol.). These correspond to mixtures containing 69·6 and 7·5 per cent. respectively by volume. At 300° the upper limit is increased by 2·3 per cent., the lower only by 0·3 per cent. B. *Coal-gas and air.*—At 15° the lower limit is 13 vols. of air or 2·8 vols. of oxygen to 1 of coal-gas, the upper limit 0·71 vol. of oxygen. These correspond to mixtures containing 7·0 and 22·6 per cent. by volume of coal-gas. At 300° the upper limit is extended by 6 per cent.; the lower by 0·5 per cent. C. *Coal-gas with a mixture of oxygen and carbon dioxide.*—At 15° the limits are 7·9 and 25·1 per cent. by vol. of coal-gas. At 300° they are 9·4 and 18·0 respectively; so that rise of temperature narrows the range.

All the above experiments were made with moist gases. Similar results were obtained with dry gases, excepting that carbon monoxide (as already shown by Dixon and Clerk) did not in any case explode. The gases used were not always quite free from admixture with air, but in all cases they were as pure as could be expected, considering that they had to be stored in considerable quantity. The transition from explosive to non-explosive mixtures was not by any means abrupt; as the composition of the mixture was altered, the force of the explosion usually became less and less, until it passed into a slow combustion in the neighbourhood of the sparking-wires.

The original paper contains copious tables of results together with theoretical discussions and details of methods of preparation, analysis, and experiment.—D. E. J.

*The Petroleum Industry at the North-west German Exhibition, Bremen.* R. Kissling. Chem. Zeit. 14, 1117—1119.

THE chief industry carried on in the neighbourhood of Bremen is that of petroleum refining. The crude material is imported in tank-steamer chiefly from America, and is worked up by repeated distillations into three classes of products. Of the first, the most volatile portion is used for the preparation of "air-gas," the residue being employed for cleaning purposes, fat extraction, &c., and as a source of power in small engines. The second group consists of illuminating oils of various qualities, and the last of paraffin and machine greases.

Models of the plant employed, both in America for obtaining the crude product, and in Germany for refining it, together with samples of the spirits and oils in various stages of their manufacture, are exhibited, as are also different forms of apparatus for testing the products.—F. H. L.

*Egyptian Petroleum.* H. Kast and A. Kunkler. Dingl. Polyt. J. 278, 34—42.

PETROLEUM is found in Egypt at Gensah on the banks of the Red Sea, and has been described by Weil (Monit. Scient. 19, 295) and by Irvine (this Journal, 1887, 130 and 276).

The oil examined by the authors was dark brown, almost opaque, and when diluted with petroleum spirit had a green fluorescence. Its specific gravity at 17° was 0·9352. On cooling to -15° it became somewhat thicker, but no solid paraffin separated out. Its speed of flow measured in Engler's viscosimeter at 35° was 6 minutes 40 seconds. Treated with alcohol and water, the extracts had no action on litmus paper. The ash amounted to 0·12 per cent. and consisted of iron and lime. When distilled up to 280° the only gas evolved was sulphuretted hydrogen. The amount of hydrocarbons soluble in a mixture of concentrated and fuming sulphuric acid in the portion of the oil distilling up to 310° was found to be 24 per cent. The residue (76 per cent.), consisting of paraffins and naphthenes, was examined as to its refractive power, and gave figures closely agreeing with those obtained from Baku petroleum, which consists chiefly of the latter; the Egyptian oil, however, contained sulphur, even after the treatment with acid, hence the refractive indices cannot be taken as a proof that naphthenes form the largest part of the hydrocarbons.

From the following table of the boiling points, it will be seen that both mesitylene and pseudocumene are absent:—

—	Per Cent. by Volume.	Specific Gravity at 17°.
200	·0	·821
201—225	0·8	·817
225—250	13·7	·874
250—275	7·0	·892
275—300	29·6	·879
Residue	31·9	
	100·0	

During the distillation considerable decomposition occurred, which is probably the cause of the lower specific gravity of the last fraction.

The oil was examined as to its commercial value by distillation from a copper still, superheated steam being employed when the temperature reached 200°, as is done at Baku; it gave:—

—	Per Cent.	Specific Gravity at 17°.	Flash Point.	Consistency.
Burning oil, .....	11·3	·841	64	..
Intermediate ....	25·0	·880	95	..
Machine II. ....	16·7	·927	144	Liquid at -10°.
Machine I. ....	16·7	·949	195	Liquid at 0°.
Cylinder. ....	17·0	·955	173	Thick at 15°.
Coke and loss ....	13·3			
	100·0			

Tested for illuminating power in Elster's photometer, the burning oil gave a light of 9·8 German standard candles, the weight of oil burnt per hour being 31 grms. Exposed to air it rapidly developed an unpleasant smell.

The authors consider that Egyptian petroleum is not suitable for the manufacture of illuminating oils, but is an excellent material for the preparation of lubricating oils.

—F. H. L.

## PATENTS.

*Improvements in and connected with Furnaces for Burning Liquid Fuel.* C. A. Sahlström, London, J. C. Bromfield, Hove, and A. F. Hill, West Brighton. Eng. Pat. 14,338, September 11, 1889. 11d.

THE invention relates to various forms of furnaces which are heated by liquid fuel which is forced or blown in as fine spray by means of steam or air or both combined, and burnt with superheated steam and hot air so combined as to obtain the greatest possible heat. Drawings and descriptions are given of (1) a steam-boiler furnace; (2) of a cupola furnace; (3) a brass and copper smelting furnace; (4) a portable or movable retort furnace.—H. S. P.

*Improvements in Oil-Gas producing Apparatus.* J. Lawson, Bridlington Quay. Eng. Pat. 16,009, October 11, 1889. 6d.

THE oil is conveyed by gravity from a tank through a tube to the lower end of a spiral of hydraulic tubing placed in a furnace. Herein it is vaporised and conducted from the upper end of the coil to the condenser where the naphtha collects, and the gas passes onwards to the gasometer.—A. W.

*A New or Improved Artificial Fuel.* J. H. Wood, London. Eng. Pat. 17,480, November 4, 1889. 4d.

THE fuel is for use "in Portland cement works, and also in foundries for foundry work, in boilers and for domestic and other purposes." For Portland cement works the following mixture is said to be suitable, but may be varied:—90 per cent. of coke breeze or coke dust, 7·5 per cent. of pitch, 2 per cent. of "slurry, that is to say a plastic admixture of carbonate of lime, silica, and alumina," 0·375 per cent. of astatki or the semi-liquid residue of petroleum after it has been refined, 0·125 per cent. of soda, potash, or bicarbonate of soda. The coke dust and pitch are mixed separately and the slurry, astatki, and alkali also separately. The two mixtures are then fed into a chamber and heated by superheated steam, and stirred by suitable appliances. The combined mass is then cooled and pressed into blocks or bricks. For foundry purposes the slurry may be omitted, and the astatki increased and some slaked lime added. For domestic purposes a small proportion of coal-dust may be substituted for some of the coke-dust, and the proportion of pitch reduced.—H. S. P.

*Improvements in Apparatus for the Manufacture of Illuminating or Heating Gases.* W. Wood, Philadelphia, U.S.A. Eng. Pat. 19,572, December 5, 1889. 8d.

A CONTRIVANCE applicable, wholly or in part, to apparatus for generating water-gas, by which steam is superheated before being passed into the generator, and by which, if desired, oil may be introduced either into the generator or into the gases as they leave the generator. For details the specification and drawings must be consulted.—H. S. P.

*Improvements in Apparatus for Burning Hydrocarbon and other Oils.* G. Gerrard, London. Eng. Pat. 19,730, December 7, 1889. 8d.

THE lower end of a coil of tubing terminates in a jet which is turned upwards towards and is placed underneath the central axis of the coil, so that the latter is heated by the flame from the jet itself. The coil and jet are placed in a tube or casing which may be closed or have a regulated opening at the lower end, but which is open at the top. The oil is supplied to the upper end of the coil, either by gravity or by a pressure tank.—A. W.

*Improvements in Regenerative Gas Furnaces and Gas Producers.* F. Siemens, London. Eng. Pat. 20,983, December 13, 1889. 8d.

THIS is a claim for having a gas producer near the furnace chamber of a regenerative gas furnace, with means of supplying the producer with heated air from the regenerator and also with means of supplying it with the hot products of combustion from the furnace, or with cold air, in any desired proportions. The accompanying drawings illustrate

Fig. 1.

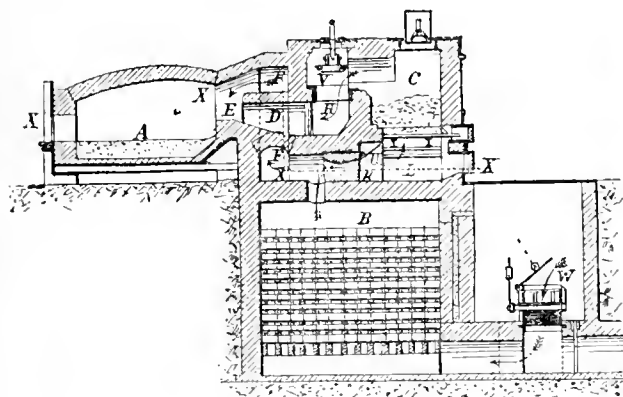
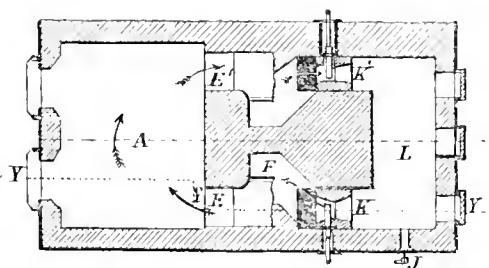


Fig. 2.



REGENERATIVE GAS FURNACE.

as determined by the opening of one or the other of two valves V. The hot air from the regenerator B flows partly by the passage F to the furnace port E, where it meets the gas from D, and the flame sweeps round from E to E'. Part of the hot air from B is caused by the steam jet K, to pass to the producer C at the one side, while at the other side a portion of the hot products of combustion is made to pass also into the producer by the steam jets K K'. By regulating these jets K K', any desired proportions of hot air and of the hot products of combustion can be supplied to the producer, or the passages thereto may be entirely closed by the valves U. Cold air, if required, is supplied by an auxiliary steam jet J. The reversing valve W is of the ordinary construction. A pipe leading from a blower is fixed to it, so that if desired an air blast may be employed, in which case it is not necessary to employ the steam jet K. Combinations such as the above may be made with regenerators of the reversible type or with those of the continuous current principle, and these are further illustrated in the specification.—A. W.

### III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

*On the Formation of Petroleum.* A. Veith. Chem. Zeit. 14, 1368.

ENGLER's researches have shown that petroleum is of animal origin. It is formed by the decomposition of marine animals, the nitrogenous organic matters being decomposed with evolution of ammonia, and the fats, under the influence of heat and pressure, into glycerol and fatty acids; the first is washed away and the latter are further decomposed with formation of hydrocarbons and water.

Engler considers the production of carbon dioxide and carbon monoxide, in the conversion of fish oil into petroleum, to be the result of a secondary reaction, as these bodies occur only in very small quantities in petroleum, and the less of these gases that are formed the more regularly is the reaction considered to proceed.

Engler's experiments can also be explained in another way. The conversion of fats into hydrocarbons may be brought about as follows:—The fat is first decomposed into glycerol and fatty acid. The glycerol is then converted under the influence of heat and pressure into acrolein, and by the elimination of water and condensation this is converted into benzene, while the fatty acids under the same conditions are converted into hydrocarbons and carbon dioxide. The carbon dioxide under the influence of dissociated hydrocarbons is reduced to carbon monoxide, and ultimately to volatile hydrocarbons.

The author thus considers that when gaseous hydrocarbons are found associated with petroleum, they are formed from carbon dioxide as above; when they are absent, he considers that the carbon dioxide has been absorbed by basic earths, with production of carbonates.—A. L. S.

*Improvements in and Relating to the Treatment of Wood, rendering the same highly Inflamable.* J. Gridley, Kingston. Eng. Pat. 13,767, September 2, 1890. 4d.

WOOD of any convenient shape and size is thoroughly dried by heat and placed in a boiling solution of resin in petroleum until it becomes impregnated with the same, after which it is taken out and subjected to a temperature of 400° F.—A. W.

*Action of Manures containing Thiocyanates (Sulphocyanides).* E. Mack and K. Silen. Tyroler landw. Blätter, 1890, 8, 198—200.

See under XV., page 1140.



## PATENTS.

*Improvements in Separating Tar and Ammonia from Gases, and in Apparatus therefor.* F. N. Mackay, Freshfield. Eng. Pat. 14,060, September 6, 1889. 8d.

THE object of the invention "is to economically cool blast-furnace and like gases, and remove tar and ammonia or other products therefrom."

The apparatus consists of a series of serpentine cooling pipes, of a tower where the gases are washed with water, a tower where they are washed with oil, and a "drying tower." The whole of this apparatus is jacketed, and the entering gases circulate through the jackets of each section of the apparatus before being brought into the washing and drying towers, and finally into the serpentine cooling pipe from whence the washed gases leave the apparatus. In this way the entering gases, while travelling through the jackets, are cooled by the returning gases. The gases, on entering, first pass through the jacketed space surrounding a series of cooling pipes, and thence into a main with branches leading to sets of three towers, each set consisting of a water-washing tower, an oil-washing tower, and a "drying tower." Following one of the branches, it is seen that the gases first pass through the jacket of the drying tower, then through the jacket of the oil-washing tower, and lastly through the jacket of the water-washing tower. From the jacket of this last tower the gases gain access at the bottom to the interior of the tower and pass up the tower, thence into the oil-washing tower, and next into the drying tower, and then into a main, from which the cooling pipes branch off.

The water-washing tower is divided into compartments by perforated plates alternately perforated in the centre and at the circumference, some of the compartments between the plates are filled with broken bricks, coke, or other suitable material, others are left empty, and are fitted with man-holes, whilst the bottom compartment is preferably filled with pieces of corrugated metal, bent wire, &c. A descending stream of water washes the gases as they ascend the tower, and the tar and ammonia-liquor are drawn off at the bottom. The gases enter the oil-washing tower at the top, and pass down the centre of the tower by a central pipe which reaches nearly to the bottom, and then ascend through an annular space between the central pipe and the jacket of the tower. This annular space is divided into compartments by perforated plates, on some of which are placed broken bricks or coke, and a stream of oil flows down the tower, through the plates, and over the material placed on them, and absorbs "various products still remaining in the gases." The gases then pass down the drying-tower, and any oil and water that is carried over drains off into the main, from whence the gases now pass to the cooling pipes.

Drawings of this apparatus and of a modified arrangement in which an ammonia refrigerator or other refrigerator is introduced, accompany the specification.—H. S. P.

*Improvements in Apparatus for Distilling Mineral Oils.* N. M. Henderson, Broxburn. Eng. Pat. 17,232, October 31, 1889. 8d.

THE "invention relates partly to the heavier mineral oils, such as those obtained in redistilling crude shale oil, and has for its object" the recovery from such oils of superior oils suitable for lubricating and other purposes.

The still used is a circular one, and is made in two parts—a hemispherical bottom of cast iron and an upper part of larger diameter of wrought iron or steel. The still is placed over a combustion chamber connected with a fire-grate. A coil of piping lies in the combustion chamber, and the heat from the fire, whilst acting directly on the bottom of the still, also superheats steam that is passed through the coil. The steam pipe is then led into the still and coiled within it, partially in the upper part and partially in the lower part, and then passes out of the still to a regulating valve or stop-cock, whence a pipe again enters the still, and proceeds centrally downwards nearly to the bottom, where the steam issues amongst the oil. A disc is fixed to the lower end of the pipe, to distribute the steam. "The steam is highly

heated when it first enters the still, but in passing through the coil in the still it yields part of its heat in an effective yet non-injurious manner through the pipes to the oil, and when it finally enters openly amongst the oil at the bottom pipe its temperature has become suitably reduced." Suitable valves are fitted, to permit of the steam being shut off from the coil and led directly into the oil, if desired. Drawings of apparatus accompany the specification.—H. S. P.

*Improvements in Treating Gases from Blast Furnaces, Gas Producers, and Coke Ovens, and in Apparatus therefor.* The Coltness Iron Company, Limited, Glasgow, and W. Young, Peebles. Eng. Pat. 17,673, November 6, 1889. 1s. 1d.

THE gases are first led into a dust chamber, where most of the dust is separated from them; the sooty tars are next separated by scrubbing with "watery tar," in which process the hot gases evaporate some of the water from the watery tars, and render them more suitable for distillation; and finally the ammonia and the more volatile oils still contained in the gases are separated by ordinary surface condensing apparatus, the last traces of ammonia being removed by washing and scrubbing the gases with water; or the ammonia may be recovered by scrubbing with sulphuric acid, or the tar and part of the ammonia recovered by cooling the gases to 100° F., after which the rest of the ammonia is separated by sulphuric acid. Drawings of suitable apparatus accompany the specification.

—H. S. P.

*Improvements in the Process of Producing Disinfectants.* A. Artmann, Braunsweig, and H. W. Kufeke, Hamburg, Germany. Eng. Pat. 20,231, December 16, 1889. 4d.

See under XVIII. C., page 1144.

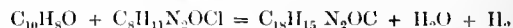
*Improvements in Coke Ovens.* W. L. Wise, London. From L. Semet, Brussels, Belgium. Eng. Pat. 20,368, December 18, 1889. 4d.

IN order to ensure regular heating of a series of coke ovens, it is necessary that each oven should be supplied with an equal quantity of gas. Advantage is taken of the equal flow of gas through orifices of equal dimensions under the same pressure. With this object the gas in the main supply pipe is kept at a constant pressure, and to the end of each pipe connected with the ovens is attached a perforated diaphragm with equal-size orifices of the cross sectional area necessary to pass the required volume of gas at the regulated pressure.—A. W.

## IV.—COLOURING MATTERS AND DYES.

*On the Action of Aromatic Bases on Meldola's Blue.* R. Hirsch and F. Kulekhoff. Ber. 23, 2992—2994.

"New" blue is formed by the action of nitrosodimethylaniline on  $\beta$ -naphthol according to the equation:—



the hydrogen which is evolved in the reaction reduces part of the nitroso base and it is necessary to use 3 mols. of the base and 2 mols. of the phenol to obtain quantitative results. Experiments proved that the amidodimethylaniline which is formed in the reaction acts on Meldola's blue and produces a greenish blue colouring matter, identical with the one described by Witt (this Journal, 1888, 559—560). Other bases act as well on the colouring matter, and the author has studied the action of aniline, toluidine, and

naphthylamine on Meldola's blue. They proceeded in the following way: 1 part of the base is added to a boiling solution of one part of the blue (free from zinc) in 10 parts of alcohol and the boiling of the mixture is continued. After a very short time the violet colour changes to green and after half an hour the reaction is finished. On addition of caustic soda, and after cooling, the base will crystallise and is recrystallised from toluene. The product obtained from aniline melts at 256° C., forms brown needles and dissolves in concentrated sulphuric acid with a violet-brown colour. On gradual addition of water the colour of the solution changes at first to orange and finally a blue-violet precipitate is formed. The product from *p*-toluidine forms brown plates melting at 250° C. and resembles the aniline product very much. Its solution in sulphuric acid is greenish grey, but changes on dilution to orange, and forms finally a blue-violet precipitate.

The colouring matter obtained from  $\alpha$ -naphthylamine melts at 260° C., and dissolves in concentrated sulphuric acid with a yellowish brown colour, which on dilution changes to a bluish green, and finally precipitates the violet sulphate.

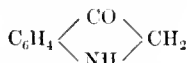
All colouring matters obtained from monamido bases are nearly insoluble in dilute mineral acids; the one prepared from *p*-amidodimethylaniline is, however, easily soluble, with a bluish-green colour. The yield of these new products is not more than 50 per cent. of the blue employed.

—A. L.

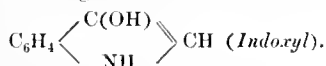
#### New Synthesis of Indigo and similar Colouring Matters.

K. Henmann. Ber. **23**, 3043—3045.

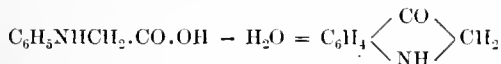
BAEYER observed that in some methods of preparing indigo a body must be formed as intermediate product, viz.:—



which he called *Pseudo-indoxyl*. All attempts to isolate it have however failed, it being at once converted by intramolecular change into—



The author intended to prepare Pseudo-indoxyl from phenylglycocine in the following way:—



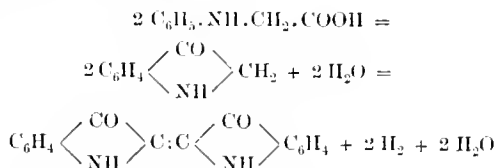
All experiments in which phenylglycocine is treated with dehydrating agents as zinc chloride, concentrated sulphuric acid, &c. failed however; fusion with caustic alkalis proved at last successful.

If 1 part of phenylglycocine be fused with 2 parts of caustic potash, the presence of air being as much as possible avoided, the mixture is coloured yellow at about 260° C., and finally deep orange-brown. If then a sample is mixed with water, *pure indigo* is precipitated instantly on the surface of the water. If this point has been reached, the heating must be discontinued at once as the substance yielding indigo is destroyed by prolonged heating. After cooling, the substance is dissolved in water and a current of air passed through, when an abundant voluminous precipitation of *pure indigo* takes place. If the fusion is effected, air being entirely excluded, a yellow coloured vat is obtained, which also precipitates indigo at once when in contact with air.

Instead of caustic potash caustic soda may be used; but the reaction then requires the assistance of a slightly higher temperature.—A. L.

#### A New Synthesis of Indigo. L. Lederer. J. prakt. Chem. 1890, **42**, 383.

On heating phenylglycocine with 4—5 times its weight of fused caustic soda a pale yellow melt is first obtained, and this on further heating becomes of a pure orange, the colour indicating the completion of the reaction. On treating the product with a large bulk of water, *pure indigo* is obtained. Its formation is indicated thus:—

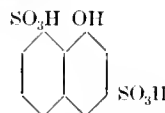


The intermediate product pseudindoxyl or indoxyl could not be isolated, and was probably oxidised directly to indigo-blue at the high temperature of the reaction.

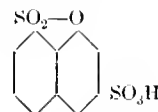
—A. K. M.

#### On Naphtholsulphonesulphonic Acids and $\alpha$ -Naphtholsulphamidosulphonic Acids. A. Bernthsen. Ber. **23**, 3088—3096.

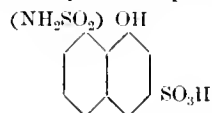
##### $\alpha$ -Naphtholdisulphonic acid—



forms an intramolecular anhydride of the Peri-series—

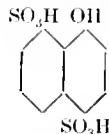


which on treatment with ammonia is converted into the sulphamide of the  $\alpha$ -sulphamidosulphonic acid  $\epsilon$ —



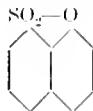
The anhydride, which the author calls  $\alpha$ -naphtholsultone-sulphonic acid, does not yield azo-colouring matters with diazo compounds, whilst the sulphamidosulphonic acid produces dyestuffs of remarkable properties when combined with diazo compounds.

##### The isomeric $\alpha$ -Naphtholdisulphonic acid $\delta$ —



can be converted into similar compounds; a sultone and a sulphamidosulphonic acid can be prepared from it. The  $\alpha$ -naphtholdisulphonic acid  $\delta$  is obtained in the following way: On nitration of naphthalene- $\alpha$ -sulphonic acid two nitro compounds (1, 5) and (1, 8) are formed, which on reduction produce two amido compounds. The 1, 8 acid forms a comparatively difficultly soluble sodium salt, by which it can be easily isolated. The amido compound is then either converted into the disulphonic acid, which is diazotised and gradually added to acidulated boiling water. After neutralising with lime, the calcium salt is converted in the usual way into the sodium salt of the  $\alpha$ -naphthol- $\delta$ -disulphonic acid. Or the 1, 8 amidosulphonic acid is

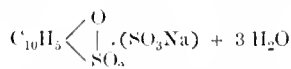
diazotised, the diazo compound boiled with water, acidulated by sulphuric acid. In this way the naphthol-sultone—



is obtained, which, on sulphonation, yields the sulphonic acid, and after treatment with calcium hydrate and sodium carbonate the sodium salt.

$\delta$ -Naphtholsultonesulphonic acid,  $C_{10}H_7\left\{ \begin{smallmatrix} O \\ SO_2 \end{smallmatrix} \right\} SO_3H$

is obtained by adding one part of naphtholsultone gradually to 8 parts of sulphuric acid containing 5 per cent. of sulphur trioxide. The acid requires to be cooled. After about an hour the mixture is poured into a mixture of 12 parts of ice and 16 parts of a saturated solution of sodium chloride, care being taken to stir well and to cool the containing vessel. The sodium salt of naphtholsultonesulphonic acid—



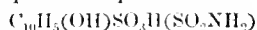
is precipitated and can be purified by recrystallisation. It forms thin plates and is much easier soluble in water than its  $\epsilon$ -isomeride.

The following table illustrates the differences between the sodium salts of the naphtholsultonesulphonic acid  $\delta$ , and the  $\alpha$ -naphthol- $\delta$ -disulphonic acid:—

Sodium Salt of.	$\delta$ -Naphtholsultonesulphonic Acid.	$\delta$ -Naphtholdisulphonic Acid.
Formula .....	$C_{10}H_7(OSO_2SO_3Na + 3 H_2O)$	$C_{10}H_7(OH)(SO_3Na)_2$
Solubility in $H_2O$	Comparatively small.	Very soluble in $H_2O$ .
Ferrie chloride produces.....	No colouration.	Blue colouration.
Solubility of barium salt in $H_2O$ .....	Very great.	Very slight.
Ammonia produces.....	Sulphamidisulphonic acid	No sulphamidisulphonic acid.
Diazo-compounds produce	No azo colour.	Azo colours.

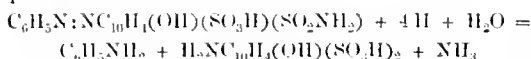
The sodium salt of naphtholsultonesulphonic acid can be easily converted by the action of caustic alkalis, carbonates, calcium hydrate, by boiling with water or by boiling with water containing 5 per cent. of sulphuric acid into salts of the naphtholdisulphonic acid, or into the free acid. On treatment of the disulphonic acid with concentrated sulphuric acid at  $100^\circ C$ . the sultonesulphonic acid is reformed.

$\delta$ -Naphtholsulphamidisulphonic acid—

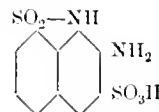


is obtained from the sodium salt of the before-described compound by mixing it with a concentrated solution of ammonia. An ammonia sodium salt is formed, and hydrochloric acid precipitates from its concentrated solution the sodium salt of  $\delta$ -naphtholsulphamidisulphonic acid.

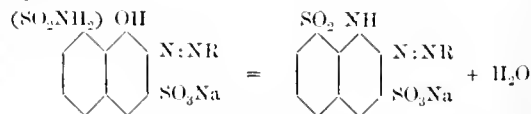
The azo colouring matters obtained from this compound are very much like those obtained from its  $\epsilon$ -isomeride, but totally different from the derivatives prepared from the corresponding disulphonic acids. They are much bluer, as a rule less soluble in water, and most of them crystallise well. Their formula is  $RN:NC_{10}H_7(OH)(SO_3H)(SO_2NH_2)$ . The combination with aniline forms a bluish-orange, with xylylene a blue-scarlet, with  $\alpha$ -naphthylamine a claret-red, and with benzidine a blue-violet. The reduction of the aniline derivative proceeds according to the following equations:—



*Action of Alkalis on the Colouring Matters obtained from the Sulphamidisulphonic Acid.*—On boiling for a short time the bluish-red solution of the colouring matter produced by the action of diazoxylene on naphtholsulphamidisulphonic acid with an alkali or an alkaline carbonate, the colour of the solution changes to a light reddish yellow. Other colouring matters obtained from these sulphamidisulphonic acids show a similar reaction. New products have been formed, which, however, still contain the nitrogen atom of the sulphamido group. They dye wool from an acid bath much bluer than the original colouring matters, and the combination with aniline (after treatment with boiling alkali) is a bluish-scarlet, with  $m$ -xylylene a cochineal, with  $\alpha$ -naphthylamine a reddish-violet, and with benzidine a steel-blue. On reduction of the xylylene colouring matter, xylylene and a compound,  $C_{10}H_6N_2S_2O_5 + H_2O$ , is formed. The latter, which forms small needles, is probably—



The intra-molecular anhydride of an  $\alpha$ -naphthylenediamine-disulphonic acid. It can be converted into  $\alpha$ -naphthylenediamine, which produces an azine with phenanthrenequinone. The action of alkalis on the colouring matters obtained from sulphamidisulphonic acid is probably expressed by the equation—



*Action of Concentrated Sulphuric Acid on Colouring Matters Obtained from  $\alpha$ -Naphtholsulphamidisulphonic Acids.*—Concentrated sulphuric acid dissolves these colouring matters with a certain colour which, however, soon changes. In some instances colouring matters are formed identical with the products obtained from the corresponding  $\alpha$ -naphtholdisulphonic acid; in other cases more complicated products are the result of this reaction.—A. L.

*On Diphenyl Bases.* E. Noelting and P. Werner. Ber. 23, 3252–3266.

ONLY diparadiamido derivatives such as diphenyl benzidine, ortho-tolidine, &c. are capable of yielding substantive cotton dyes when diazotised and combined with phenols, amines, or their derivatives. Diphenylene, the by-product in the preparation of benzidine, which has an unsymmetrical constitution, fails to give substantive dyes, and the authors have prepared a considerable number of derivatives of diphenylene and substances of analogous constitution to ascertain whether they all resemble diphenylene in not giving those dyes.

*Di-tolylene* is a by-product in the preparation of ordinary tolidine. It is contained in the mother-liquors of the hydrochloride of the latter base. The sulphate is readily soluble in water, and the salts of the new base also differ from those of orthotolidine in being coloured by bromine water first a dirty green, which changes to a red-violet. It gives no substantive dye.

*Bromo- and Iododiphenylene* are obtained by first preparing the parabromo- or iodo-azobenzene from amidoazobenzene by Sandmeyer's method and reducing the alcoholic solutions of the azo compounds with stannous chloride. These diphenylene derivatives give azo dyes, which hardly have any affinity for unmordanted cotton.

When meta-iodohydrazobenzene is dissolved in hot concentrated hydrochloric acid, iodine separates as well as considerable quantities of benzidine.

By reducing azobenzene-*para*-sulphonic acid with sulphurated hydrogen in ammoniacal solution, Griess obtained a benzidine sulphonic acid. This acid, however, is actually

hydrazobenzene sulphonic acid. It cannot exist in the free state, as on adding hydrochloric acid to its salts, benzidine sulphate separates out.

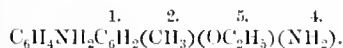
*Diphenyl bases from Hydroxyazo Derivatives.*—The ethylic ether of hydroxyazobenzene can be converted into an ethoxydiphenyl base by stannous chloride and acid. The base yields no substantive dyes, and probably has an analogous constitution to diphenylene.

The preparation of *orthotolyl-azophenol* and *orthotolyl-disazophenol* is described, the former by the action of one molecule, the latter of two molecules of diazo-orthotolyl-chloride on an alkaline solution of phenol. The former is converted into the ethyl ether, which on reduction with  $\text{SnCl}_2$  in alcoholic solution yields a diphenyl base, colours from which do not dye unmordanted cotton. The corresponding paratolyl-azophenol derivatives is similarly prepared, from which the hydrazo compound is readily obtained. This, however, instead of being converted by acids into a diphenyl derivative is split up into an azo compound and monamines. The following substances are also described:—

*Phenylazo-ortho cresetol*, its hydrazo derivative *orthotolyl-azo-ortho cresol*, *orthotolylhydrazo-ortho cresetol*, *orthotolyl-disazo-ortho cresol* and its ethyl ether, *paratolylazo-ortho cresol* and the ethyl ether, *paratolylhydrazo-ortho cresetol*, *paratolyl-disazo-ortho cresol* and its ethyl ether.

In the azo derivatives of paracresol the para position is free. By reducing the ethers of these bodies in acid solution true benzidine derivatives are therefore produced which were patented by Geigy in 1887.

*Phenylazo-paracresetol* is prepared from phenylazo-paracresol, ethyl bromide and sodium alcoholate by heating their alcoholic solutions. By reducing in ammoniacal and alcoholic solution with sulphuretted hydrogen, the hydrazo compound is produced which with acids undergoes a change into *methylthorbenzidine*—



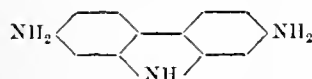
This base is capable of yielding substantive tetrazo dyes which have almost as perfect an affinity for unmordanted cotton as the usual benzidine derivatives. The shade in the ease of combination with  $\alpha$ -naphthol- $\alpha$ -sulphonic acid tends towards violet as compared with that obtained from the corresponding benzidine compound.

*Orthotolylazo-paracresol*, the cresetol derivative, and *orthotolylhydrazo-paracresol* are also described. From the latter *dimethylthorbenzidine* is readily prepared, which yields substantive cotton dyes.

*Paratolylhydrazo-paracresetol*, by treatment with mineral acids yields a diphenyl derivative which has an analogous constitution to diphenylene, as one para position is occupied in the azo compound. The dyes prepared from it consequently have hardly any affinity for cotton.—A. R.

#### Synthesis of a Diamidocarbazol from Benzidine. E. Täuber. Ber. 23, 3266—3269.

META-DIAMIDORENZIDINE, when heated for 10 hours with hydrochloric acid containing 18 per cent. of HCl from  $180^\circ$ — $190^\circ$  is converted into diamidocarbazol, ammonia being split off. This new compound, which has the composition—

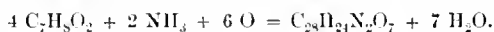


is best separated as its sulphate, which is almost insoluble even in boiling water. It yields strong substantive tetrazo dyes for cotton of shades similar to those derived from benzidine, and in this respect closely resembles benzidine sulphone and diamidodiphenyloxide, which only differ from diamidocarbazol in having an  $\text{SO}_2$  or O in place of the

imido group. This diamidocarbazol is not identical with the one obtained by the "Badische Company" by reduction of dinitrocarbazol. The author also refers to the nomenclature of substituted benzidines.—A. R.

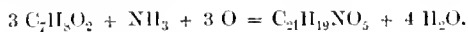
#### Orcein. K. Zulkowski and K. Peters. Monatsh. 11, 227—245.

THE authors have submitted pure orceinol to the action of ammonia and air, and have obtained three distinct dye-products, namely, *a*, orcein, which is the chief product; *b*, a yellow dye; and *c*, a dye resembling litmus. The two former were obtained crystalline, the last in an amorphous state. The same products are also obtained, and in a much shorter time, by the action of hydrogen peroxide and ammonia on orceinol. The first reaction is conducted as follows:—A solution containing 50 grms. of orceinol, 200 cc. of water, and 200 cc. of ordinary ammonia solution is put into a capacious flask, the mouth of which is then covered with a beaker. A granular precipitate is gradually formed, and in about two months the whole has become of a thick consistency. The greater part of the ammonia is then driven off by a gentle heat; the product is cooled, neutralised with hydrochloric acid until a distinct change of colour is produced, filtered, and washed with water. The precipitate contains all three dyes, and the red filtrate contains the yellow dye together with some orcein; the latter is concentrated and the dyes precipitated by the addition of salt. The separation of the crude product into its constituents is effected by extracting first with ether which dissolves the yellow dye, then with boiling alcohol which dissolves the orcein, the residue insoluble in spirit being the litmus-like substance mentioned. To obtain pure orcein the alcoholic solution is concentrated, diluted with an equal bulk of water, and set aside to crystallise; it is then filtered on the pump and dried in vacuo at the ordinary temperature. Pure crystallised orcein is insoluble in hot or cold water, in ether, benzene, chloroform, and carbon bisulphide, but is soluble in acetone, acetic acid, and alcohol; it dyes wool a beautiful amaranth red, its dyeing power being 150—200 times greater than that of orcein extract. Its composition agrees with the formula  $\text{C}_{23}\text{H}_{21}\text{N}_2\text{O}_7$ , and its formation may be expressed thus—



The yield amounted to about 50 per cent. of the weight of orceinol taken.

The yellow product is purified by crystallisation from a mixture of alcohol and water. It is sparingly soluble in cold, more readily in hot water, the best solvent, however, being alcohol. Its solution has an orange-yellow colour, which becomes deep violet on the addition of ammonia or caustic alkali. Its formation and composition are indicated by the equation—



The third substance being amorphous and insoluble has not been obtained pure. It exhibits a green metallic lustre, dissolves in alkalis to a deep blue solution resembling litmus, and, like the latter, the blue solution changes to red on the addition of acid.

The same three products appear to be formed when orceinol is oxidised by hydrogen peroxide in the place of atmospheric oxygen, but in this case the reaction is complete in three or four days. The proportions employed in this case were orceinol 100 parts, ammonia solution (22 per cent.) 200 parts, and hydrogen peroxide solution (3 per cent.) 1,200 parts.

An attempt to prepare a substance analogous to orcein by the action of ammonia and hydrogen peroxide on resorcinol yielded a negative result, the product in this case bearing more resemblance to litmus than to orcein. A mixture of resorcinol and orceinol in molecular proportions yielded on the other hand a substance  $\text{C}_{26}\text{H}_{20}\text{N}_2\text{O}_7$ , which the authors call *reso-orcein*, and which is analogous to

orcein. It dissolves readily even in dilute alcohol, the solution exhibiting a deep magenta colour though bluer than orcein.

A number of other dihydroxy- and trihydroxy-derivatives of benzene was likewise examined in the same direction, but no satisfactory results were obtained.—A. K. M.

*The Preparation of Ultramarine.* J. Wunder. Chem. Zeit. **14**, 1119—1123.

See under XIII., page 1137.

*Flavin and its Preparation.* V. H. Soxhlet. Chem. Zeit. **14**, 1345—1346.

See under VI., page 1125.

*On the Properties of the Natural Colouring Matters of Yellow Silk and their Analogy to those of Vegetable Carotene.* R. Dubois. Compt. Rend. **111**, 482—483.

THE following different dyes were extracted from silk naturally yellow. (1.) A golden yellow colouring matter soluble in potassium carbonate and reprecipitated by acetic acid. (2.) Crystals which appear yellowish-red by transmitted, and brown by reflected light. (3.) A lemon-coloured amorphous body. On evaporating its alcoholic solution granular masses separate. (4.) Yellow octohedral crystals resembling sulphur. (5.) A dark bluish-green pigment in minute quantities and probably crystallisable. Though these different substances were not obtained in a sufficiently pure state to draw conclusions as to their composition, the author observes that these dyes closely resemble those obtained from carotene.—A. R.

#### PATENTS.

*Improved Manufacture of Azoxyaniline and of Colouring Matters derived therefrom.* J. Inray, London. From "La Société Anonyme des Matières Colorantes et Produits Chimiques de St. Denis," Paris, France. Eng. Pat. 20,219, December 16, 1889. 4d.

IF *p*-nitro-acetanilide, melting at 207°, be reduced with zinc powder and salt in an aqueous solution, it is converted into acet-azoxy-aniline of melting point 275°. This, on treatment with hot hydrochloric acid, yields the hydrochloride of azoxyaniline readily soluble in hot water and nearly insoluble in hydrochloric acid. The base, obtained from the solution of the hydrochloride by the addition of an alkali, is a yellow precipitate insoluble in water and crystallises from dilute alcohol in small needles melting at 185°. Colouring matters are obtained by diazotising the base and combining the tetrazo-compound with  $\alpha$ -naphthol- $\alpha$ -sulphonic acid or  $\beta$ -naphthol disulphonic acid. The products dye unmooranted cotton violet-red and bluish-violet respectively from an alkaline bath, and are said to resist acids.—T. A. L.

*The Production of a Substituted Meta-Diamine and of Colouring Matters derived therefrom.* J. Inray, London. From "La Société Anonyme des Matières Colorantes et Produits Chimiques de St. Denis," Paris, France. Eng. Pat. 20,292, December 17, 1889. 4d.

BY heating together 10 kilos. of chloro-dinitro-benzene,  $C_6H_3Cl(NO_2)_2$  [ $Cl : NO_2 : NO_2 = 1 : 2 : 4$ ] with 7 kilos. of *m*-amidodimethylaniline on the water-bath, dinitro-*m*-amidodimethyldiphenylamine is obtained, melting at 136° and crystallising from alcohol in orange-coloured laminae. 15 kilos. of this product are then heated with 14 kilos. of nitrosodimethylaniline hydrochloride in an alcoholic or acetic acid solution. A colouring matter is soon formed and is precipitated by the addition of salt. It is purified by

redissolving in water slightly acidulated with hydrochloric acid and again adding salt, when it forms a crystalline brownish-red mass, which dyes wool, silk, and cotton mordanted with tannin, violet with a red fluorescence, visible in the case of cotton, in an artificial light. The dinitro condensation product may also be reduced with the ordinary reducing agents, yielding products which are also violet colouring matters.—T. A. L.

*Improvements in and relating to the Manufacture of Colouring Matters suitable for Dyeing and Printing.* J. Y. Johnson, London. From "The Badische Anilin und Soda Fabrik," Ludwigshafen, Germany. Eng. Pat. 20,668, December 23, 1889. 6d.

DIAMIDO-DIPHENYLENE ketone is combined with hydroxylamine forming diamido-diphenylene ketoxime. This substance, on treatment with nitrous acid, yields a tetrazo compound which combines with phenols, amines, their sulphonic and carboxylic acids, to form dyestuffs capable of dyeing unmooranted cotton orange, red, brown, purple or blue. The diamido-diphenylene ketone is obtained by mixing one kilo. of dinitro-diphenylene ketone with 10 kilos. of alcohol, and reducing with 10 kilos. of sodium sulphide ( $Na_2S + 9 H_2O$ ) at the boiling point of the liquid. The alcohol is then distilled off and the residue extracted with dilute hydrochloric acid, from which the base is precipitated by the addition of sodium carbonate. To produce the ketoxime, 21 kilos. of diamido-diphenylene ketone are dissolved hot in 1,600 litres of water and 35 kilos. of hydrochloric acid of sp. gr. 1.11. A concentrated aqueous solution of 10 kilos. of hydroxylamine hydrochloride is then added, and the whole allowed to cool down. About 63.5 kilos. of caustic soda lye of sp. gr. 1.38 are next added, and the temperature of the solution raised to about 70° and finally boiled. After filtering, the new base is carefully precipitated by adding about 44 to 48 kilos. of hydrochloric acid. The product is employed preferably as a paste, as unless carefully dried at a low temperature it is liable to undergo decomposition. In place of hydroxylamine hydrochloride its sulphonic acid may be employed, when the same product is produced. The conversion of the diamido body into its tetrazo compound is performed in the usual manner, and the latter may be combined with two molecules of the same or of different phenols or amines, &c.—T. A. L.

*Production of Compounds of the Diphenylmethane Group and the Rosaniline Series.* O. Inray, London. From "The Farbwerke vormals Meister, Lucius and Brüning," Höchst-on-the-Maine, Germany. Eng. Pat. 20,678, December 23, 1889. 6d.

WHEN formaldehyde is condensed with certain aromatic amines, there are formed derivatives of diphenyl-methane which on oxidation in presence of an aromatic amine yield colouring matters of the rosaniline series. By the action of formaldehyde on aniline, anhydroformalddehyde aniline  $C_6H_5N = CH_2$  is produced. 50 parts of this are heated with 70 parts of aniline hydrochloride and an excess of aniline on a water-bath with constant agitation when the mass becomes semi-liquid. After about 12 hours it is made alkaline, the excess of aniline distilled off with steam and the oil remaining solidifies to a crystalline mass of diamidodiphenylmethane which can be purified by crystallisation from benzene or water. It melts at 87°. Homologues of the compound are obtained by using homologues of aniline. All these compounds when oxidised together with an aromatic amine and hydrochloric acid yield colouring matters of the rosaniline series the oxidising agents being, nitro-benzene *o*- and *p*-nitro-toluene, nitro-xylene, salts of oxide of iron, arsenic acid, azobenzene and similar bodies. Formaldehyde also combines with secondary amines such as diphenylamine, and these substances on oxidation with an amine yield substituted rosanilines.—T. A. L.

*Improvements in the Manufacture of Raw Materials for Dyestuffs.* R. J. Friswell, London. Eng. Pat. 39, January 1, 1890. 4d.

A PROCESS for obtaining oxyazotoluidine from azoxytoluidine melting at 168° (obtained by the alkaline reduction of nitrotoluidine melting at 107°) by dissolving it in sulphuric acid of sp. gr. 1.84 and allowing the mixture to stand 24 to 140 hours at 15° to 60° C. By the addition of water the oxyazo compound is precipitated in the form of sulphate almost insoluble in water, and as an amido body can be used for the preparation of dyestuffs in the usual manner.

—T. A. L.

## V.—TEXTILES: COTTON, WOOL, SILK, Etc.

*On Silk Secretion by Bombyx Mori.* R. Dubois. Compt. Rend. 111, 206—207.

See under XXIII., page 1163.

*The Colouration of Silk by means of Coloured Food given to Silkworms.* L. Blanc. Compt. Rend. 111, 280—282.

VILLON and Blanchard have shown that by giving to silkworms coloured food—especially food coloured by indigo and madder—the silk of the resulting cocoons is coloured accordingly. The experiments of the author show that this colouration is only effected when the colour is used in the form of a powder, and is due to the colour being collected mechanically by the viscous material surrounding the threads of the silk. Soluble and readily diffusible colouring matters such as magenta, however, whilst they are capable of being absorbed by the intestinal epithelium of the silkworm and thereby colouring the secretive organs, do not colour the products of secretion—i.e. the resulting silk.—C. A. K.

*On the Properties of the Natural Colouring Matters of Yellow Silk and their Analogy to those of Vegetable Carotene.* R. Dubois. Compt. Rend. 111, 482—483.

See under IV., page 1124.

## VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

*Action of Aniline on Bleached Cotton.* S. Lipkowski. Chem. Zeit. 14, 1203.

KOECHLIN first, some years ago, suggested aniline as the cause of bleached cotton becoming tinted of a rose colour on lying for some time at the print works.

Aniline itself, however, is found not to bring about the phenomenon; only after a considerable time is bleached cotton tinted by aniline vapour, and then the colouration is brownish. But if, instead of aniline, a mixture, in certain proportions, of aniline salt, sodium chlorate, copper sulphide, or a hydrochloric acid solution of ammonium vanadate be employed, then after a period of from 18 to 24 hours a rose colouration is obtained on the goods. The active principle is thus not aniline itself, but its chlorate in the successive changes which this latter brings about under the action of hydrochloric acid, heat, light, &c.

Various sorts of material were experimented on, and it was found that the rose colour is especially developed on cotton from India; good American sorts give no colouration after 24 hours, and take only a faint yellowish tinge after 48 hours.

The bleaching operations are not without effect on the intensity of the colouration; excessive contact with chlorine water, for instance, and insufficient washing, leave the goods in a state conducive to the formation of the colour.—T. L. B.

*Flavin and its Preparation.* V. H. Soxhlet. Chem. Zeit. 14, 1345—1346.

FLAVIN is a light brownish yellow to deep orange yellow powder, chiefly used for obtaining yellow and orange shades on wool. It is also employed for dyeing vegetable fibres by using alumina mordants in conjunction with it. The dye is almost exclusively made in America by extracting quercitron bark with alkaline aqueous solutions under pressure. The solutions are rendered alkaline either by the addition of soda or borax or ammonia. After very careful filtration of the extracts, so much weak acid, preferably a mixture of hydrochloric and sulphuric acids, is added to produce complete saturation, and the mixture is then boiled. By this treatment it is assumed that the quercitrin is split up into quercetin and a sweet body, possibly isodulcite, the latter, along with the tannin, remaining in solution. The extract, after treating with acid, is filtered hot, and the press cakes dried and ground. The yield, according to the purity of the final product, varies from 4—10 per cent. The dye should not be entirely soluble in boiling water; on adding aluminium sulphate no precipitate should be produced, but the colour of the solution must alter to bright yellow. Aqueous solutions of the dye, if exposed to the air a few days, lose their tinctorial power entirely.

By the action of strong nitric acid flavin is converted into a much stronger dye, but of a different shade.—A. R.

*The Fixation of Insoluble Azo Dyes on Cotton Fibre in Calico Printing.* H. Schmid. Chem. Zeit. 14, 1406—1407.

It is well known that up to the present time it has been found impossible to render cotton fabrics dyed with commercial azo dyes, such as Ponceaux, Bordeaux, &c., fast towards soap. Most of these colours are dissociated from the fibre by water alone. A certain improvement was recently effected by Koehlin, who employs as mordant a mixture of several oxides, such as aluminium, magnesium, and zinc oxides. Still the dyes fixed by this means are attacked by soap. The use of a mordant rendering the azo dyes "soap fast" would certainly be preferable to any process of synthetically producing an insoluble non-sulphonated dye on the fibre itself. A number of methods embodying the latter principle have been patented during the last 10 years, but most of them are open to various drawbacks. One of the best processes which has stood the test of practice is to prepare the fabric with sodium  $\beta$ -naphtholate, the design being subsequently printed on the material by means of a thickened solution of a diazo salt (such as diazotised  $\alpha$ -naphthylamine). The chief objections to the method are:—1, the ease with which the diazo salt decomposes with evolution of nitrogen; 2, that the material prepared with sodium  $\beta$ -naphtholate may not be long preserved; and, 3, that the printed fabric must be at once finished in order to preserve the purity of the whites.

Nine years ago the author devised an improved process, the details of which have only now been published. It consists in preparing the fabric with a solution of sodium nitrite, the material being afterwards printed on by a mixture of a naphthol, an amine, and acid. For example, in order to produce Xylidine ponceau (Ponceau R.), a mixture of 25 parts of xylidine, 46 of hydrochloric acid of 20 B., 30 of  $\beta$ -naphthol, 80 of alcohol, 50 of water, and 250 of gum solution, is employed for printing, the fabric being previously treated with a solution of sodium nitrite containing 35 grms. per litre. After printing the colour is developed by an

ammoniacal steam bath; or the latter operation may be avoided by substituting for one-half of the hydrochloric acid in the recipe given below an equivalent amount of some volatile organic acid. Bright reddish-orange colours of considerable stability are obtained by the above process. If the xylidine be replaced by  $\alpha$ -naphthylamine, Garnet and Bordeaux red tones are obtained. By employing various mixtures of xylidine and  $\alpha$ -naphthylamine, a whole range of colours from orange red to dark red may be produced.

*Recipe for Bordeaux.*

- 84 grms. of  $\alpha$ -naphthylamine.
- 87 grms. of  $\beta$ -naphthol.
- 560 grms. of nitric acid at 36° B.
- 100 grms. of water.
- 560 grms. of alcohol of 90 per cent.
- 1,700 grms. of gum Senegal.

Preparation of the fabric: 40–50 grms. of sodium nitrite per litre.—H. T. P.

*Diamine Black R.* Oesterr. Woll- u. Leinen- Ind. 1890, 462.

This new dyestuff gives shades varying from grey to black, according to the amount used. It is applied on cotton in a boiling bath containing soda, sodium sulphate, or sodium phosphate. The shades produced are fast to light and soap. In common with other dyes of the benzidine class, Diamine black possesses the property of itself acting as a mordant for basic dyes, such as Safranine, Thioflavine, &c., and may, therefore, be usefully employed for producing compound shades with the latter class of dyes, as well as for darkening shades produced with other substantive colouring matters.

Silk is dyed with Diamine black under the addition of acetic acid to the dye-bath. The cotton portion of mixed goods composed of silk and cotton is alone dyed when an addition of soap is made to the dye-bath; the silk may be afterwards dyed another colour.—E. B.

*Diamond Black.* Chem. Zeit. Rep. 1890, 165.

*Diamond Black* may be dyed on wool in two ways: first, after mordanting with potassium bichromate and oxalic acid, when the dyestuff (1–2 per cent.) is applied along with 2 per cent. of acetic acid in the dye-bath; or second, in a single-bath containing 5 per cent. of nitrate of iron. The colour is developed on the temperature of the bath approaching boiling.

This dyestuff may be used in conjunction with other members of the alizarin group to produce compound shades. The shades it yields are extremely fast to acids, light, stoving, and milling. Silk and gloria-fabrics are dyed like wool.—E. B.

*Rubramine.* Chem. Zeit. Rep. 1890, 165.

This basic dye yields fine red shades on cotton mordanted with tannic acid, but also dyes unmordanted cotton if sodium acetate be added to the dye-bath. Wool and silk are dyed in a neutral or faintly acid bath.—E. B.

*The Production of Azo-Dyes on Cotton Piece-Goods.* H. Koechlin and G. Galland. Agenda du Chimiste, 1890.

The cloth is padded with a solution of 25 grms. of  $\beta$ -naphthol and 25 grms. of caustic soda at 38° B. per litre of water, then dried by hot air and passed for half-a-minute into one of the following developing baths:—

*For Pale Orange-Yellow.*—12.5 grms. of aniline, 25 grms. of hydrochloric acid, and 250 cc. of water are mixed, and then there is added a mixture of 9.5 grms. of sodium nitrite, 250 grms. of ice, and 250 cc. of water, and finally a solution of 100 grms. of sodium acetate in 250 cc. of water.

For other colours the aniline in the above recipe is replaced by other amido compounds, and the quantity of sodium nitrite is modified as below:—

*Orange.*—12.5 grms. of *p*-toluidine, 8.5 grms. of sodium nitrite.

*Orange-Red.*—10 grms. of *p*-nitraniline, 5.5 grms. of sodium nitrite.

*Scarlet.*—12.5 grms. of xylidine, 7 grms. of sodium nitrite.

*Red.*—10 grms. of  $\beta$ -naphthylamine, 6 grms. of sodium nitrite, and 50 grms. of sodium acetate.

*Deep Red.*—10 grms. of amidoazobenzene, 5 grms. of sodium nitrite.

*Dull Red.*—12 grms. of  $\alpha$ -naphthylamine, 7.5 grms. of sodium nitrite.

*Chocolate.*—10 grms. of benzidine, 5 grms. of sodium nitrite.

*Purple.*—7.5 grms. of dianisidine (sulphate), 3.25 grms. of sodium nitrite.

*Rose.*—10 grms. of naphthionic acid, 6 grms. of sodium nitrite, and 50 grms. of sodium acetate.

*Blue.*—1 litre of 10 per cent. solution of Safranine and 75 grms. of hydrochloric acid are mixed together, and there are successively added to the same a solution of 20 grms. of sodium nitrite in 250 cc. of water, mixed with 250 grms. of ice, and 250 grms. of sodium acetate dissolved in 2 litres of water.

A red is also produced by preparing the cloth with the following solution: 25 grms. of  $\alpha$ -naphthol and 30 grms. of caustic soda at 38° B. per litre of water, and, after drying, developing in a bath prepared with 10 grms. of  $\beta$ -naphthionic acid, 25 grms. of hydrochloric acid, and 250 cc. of water, to which are added in succession sodium nitrite (quantity not given in original) dissolved in 250 cc. of water and mixed with 250 grms. of ice, and 100 grms. of sodium acetate dissolved in 250 cc. of water.

The reds obtained with *p*-nitraniline and  $\beta$ -naphthylamine may be darkened by boiling with a solution of copper sulphate containing 2 grms. of the salt per litre.

In calico-printing the cloth is prepared with the alkaline naphthol solution, dried and printed with one or more of the diazo-derivatives prepared as above and suitably thickened, or a resist-colour containing 600–800 grms. of stannous chloride per litre of gum-solution may be first printed and the diazo-mixture padded over it.—E. B.

*Use of Nitroso-Compounds in Dyeing and Printing.* G. Ulrich. Mittheil. des k.k. Gew.-Museums, 1890, 47–59.

UNTIL quite recently the only nitroso-compounds finding employment for dyeing and printing were Naphthol green B (ferrous salt of nitroso- $\beta$ -naphtholmonosulphonic acid) and Resorcinol green (dinitrosoresorcinol). Later, however, several other nitroso-derivatives have been introduced, as, for example, the Gambines R and Y ( $\beta$ -nitroso- $\alpha$ -naphthol and  $\alpha$ -nitroso- $\beta$ -naphthol), nitrososalicylic acid, dinitroso-naphthoresorcinol, and *o*-naphthoquinonoxim; in fact, as Kostanecki shows, all ortho-quinonoxims and their substitution derivatives have colouring power.

*Naphthol green B.* comes into commerce as a dark green powder. Its aqueous solution gives the following reactions: Stannous chloride, acidulated with hydrochloric acid, decomposes it in the cold; oxidising agents, such as chromic acid, readily destroy it, even copper sulphate at the boil affecting it injuriously. It dissolves in strong sulphuric acid, with a yellow colour. On the fibre it shows, at a high temperature, little resistance to acids. In spite of this fact, most treatises on dyeing recommend the addition of acid to the dye-bath. The waste of colouring matter due to this cause is of little moment in the case of dark shades, but it is otherwise when pale shades are being dyed, with which the effect of a slight variation in the proportion of acid added to the bath is readily seen. In consequence of this, the author has sought to modify the method of dyeing. The following is a summary of the results of experiments made with this object:—

1. The colouring power of the dissolved dyestuff is quickly reduced, probably owing to oxidation, this action



being accelerated by the presence of acid and by heating. Experimenting with additions of various proportions of sulphuric acid, it was found that the quantity of this acid required for the decomposition of the dyestuff at a given temperature, varied inversely as the duration of action; thus, the addition of 1·166 grms. of sulphuric acid, diluted with water (10 grms. at 66° B. per litre) to a litre of dye solution containing 0·166 grm. of dyestuff, and maintained at the boil, effected its decolouration in three seconds, whilst 0·266 grm. of sulphuric acid (diluted at the same rate) added to another similar portion of the dye solution, had to be boiled 60 seconds with it to produce the same effect. The proportion of acid, however, required for decomposing the dyestuff at a given temperature is not constant, varying with the strength of solution of the latter. 0·33 grm. of sulphuric acid decomposed a litre of dye solution containing 0·016 grm. of dyestuff, and 0·366 grm. of the same acid sufficed to decompose a litre of dye solution 10 times as strong, the temperature in each case being 80°.

2. Under otherwise similar conditions, acetic acid acts injuriously at temperatures 20° higher than does sulphuric acid.

3. The addition of sodium sulphate in moderate amount raises the stability of the dyestuff at temperatures of 80° upwards.

4. Addition of ferrous sulphate tends to preserve the dye at temperatures below 80°. Ferric salts act injuriously.

5. Acetic acid in the presence of ferrous and sodium sulphates acts less destructively than sulphuric acid under the same conditions.

From further experiments it was ascertained that the best temperature for dyeing with this colouring matter was about 80°, and that the most suitable proportions of assistants for 0·5 part of dyestuff were 10 parts of Glauber's salt, 10 parts of ferrous sulphate, and two parts of sulphuric, or four parts of acetic acid.

Copper or tin dye-baths cannot be used, as these metals are attacked. In view of these objections and difficulties, the author considers it preferable to first mordant the woollen material, and then to rinse and dye it in a separate bath, to which sodium acetate is added.

**Nitroso-β-naphthol.**—The author recommends the following method of dyeing cotton with this colouring matter: the material is impregnated with an alkaline solution of the colouring matter, prepared by dissolving 5 parts of nitroso-β-naphthol and 5 parts of caustic soda (80 per cent.) in 90 parts of water; after "hanging," the material is worked in or passed through a fixing-bath corresponding to the shade desired, and composed of: for olive-green, a 5 per cent. solution of ferrous sulphate; for chocolate, a 2 per cent. solution of cobalt sulphate; for orange-yellow, a 2 per cent. solution of nickel sulphate. The goods may then be rinsed, dried, and finished.

The colour on the iron mordant is not fast to soap or dilute acid; those on the nickel and cobalt mordants are satisfactory in these respects.

The above method is applicable to printing; 5 parts of nitroso-β-naphthol and 5 parts of caustic soda (80 per cent.) are dissolved in 30 parts of water, thickened with 60 parts of dextrin solution (30 parts of dextrin in 30 parts of water), printed on, the cloth dried at a low temperature, and passed through one of the above-mentioned fixing-baths to develop the colour.

**Nitrosophenol** was experimented with, but no results of importance obtained.

**Dinitrosoresorcinol** (Syn. Fast myrtle, Resoreinol green, Alsaec green).—Excellent results are obtained with this colouring matter by the method described for nitroso-β-naphthol. For pale shades, the cotton material is prepared in a solution of 10 parts of the dyestuff in 10 parts of caustic soda (80 per cent.) and 980 parts of water. For dark shades a solution five times as strong is used.

The colours are produced by passing the material so prepared through one of the following solutions: for dark blue-green, 10 per cent. solution of ferrous sulphate; for chocolate, 5 per cent. solution of cobalt sulphate; for yellow-brown, 5 per cent. solution of nickel sulphate. The shades are similar to those yielded by nitroso-β-naphthol but for

equal amounts of dyestuff, much more intense. The cobalt and nickel colours are absolutely fast to soap; the iron colour is not perfectly fast, but faster than when produced by the customary method. The ordinary method of dyeing with this colouring-matter also labours under the disadvantage that some of the colouring matter is wasted, being decomposed at the high temperature of the bath.

For printing, 5 parts of dinitrosoresorcinol are dissolved, by the aid of an equal weight of caustic soda (80 per cent.) in 30 parts of water and mixed with a thickening composed of 30 parts of dextrin and 30 parts of water. The thickened mixture is printed and the colour-lakes produced as above described. This standard colour mixture, when reduced 1 : 15 to 1 : 63 and fixed by cobalt, yields delicate brownish tints, which may well replace the more expensive similar shades obtained with alizarin-orange.

**Nitrososalicylic Acid.**—Dye tests were made with a crude specimen of this compound prepared by adding sodium nitrite to an acidulated solution of sodium salicylate. No results of any value were obtained; ferric salts yield myrtle, and ferrous salts brown shades, by the above-described alkaline method, but only the latter are even moderately fast to soap. As the tests, however, were made with an impure product, they cannot be accepted as conclusive.

—E. B.

## VII.—ACIDS, ALKALIS, AND SALTS.

*A Double Salt of Dithionic Acid.* G. Bødländer. Chem. Zeit. **14**, 1140.

The author has succeeded in preparing a double salt of dithionic acid containing barium and rubidium according to the following formula:— $\text{BaRb}_4(\text{S}_2\text{O}_6)_3 \cdot 11\text{H}_2\text{O}$ . This compound crystallises in bunches of extremely small white needles, which polarise light; its solubility in water at 15° being 10·34 per cent.

The formation of this double salt is of interest, as Kolbe (J. prakt. Chem. [2], **19**, 485) has asserted that dithionic acid is monobasic, its true formula being  $\text{HSO}_3$ . Fock (Zeits. f. Krystallogr. **14**, 361) has described a dithionate of ammonium and aluminium having the formula—



but other attempts to prepare similar compounds appear to have been unsuccessful.—F. H. L.

*Hydrated Neutral Aluminium Sulphate; Analysis of a Natural Product.* P. Marguerite Delacharloumy. Compt. Rend. **111**, 229—231.

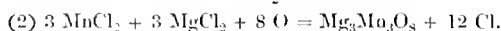
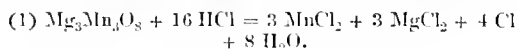
The author has shown that the formula of hydrated neutral aluminium sulphate is  $\text{Al}_2(\text{SO}_4)_3 \cdot 16 \text{H}_2\text{O}$ , and not  $\text{Al}_2(\text{SO}_4)_3 \cdot 18 \text{H}_2\text{O}$  as formerly supposed. Further he has examined two samples of a naturally occurring neutral aluminium sulphate, both of them beautifully crystalline, with the result that their composition also corresponds to the formula  $\text{Al}_2(\text{SO}_4)_3 \cdot 16 \text{H}_2\text{O}$ . This body, like that prepared in the laboratory, is characterised by a tendency to lose water. The naturally occurring product was embedded in a matrix of a basic sulphate of aluminium which had resulted from the decomposition of the neutral salt.—C. A. K.

*Equilibrium and Reciprocal Displacements of the Volatile Alkalies.* Berthelot. Compt. Rend. **111**, 289—296.

This paper is only of theoretical interest.—T. L. B.

*The Wilde and Reyhler Chlorine Process.* Industries, December 5, 1890, 556.

THE new process is based on the decomposition of hydrochloric acid in the following conditions:—When equivalent weights of crystalline magnesium sulphate ( $\text{MgSO}_4 \cdot 7 \text{H}_2\text{O}$ ), magnesium chloride ( $\text{MgCl}_2 \cdot 6 \text{H}_2\text{O}$ ), and manganese chloride ( $\text{MnCl}_2 \cdot 4 \text{H}_2\text{O}$ ) are heated together, they first dissolve in their water of crystallisation and then give off hydrochloric acid gas, leaving a pink hygroscopic mass consisting of a mixture of magnesium sulphate, manganese chloride, and magnesium oxychloride, entirely free from water. When heated to dull redness in a current of air in a muffle, this mixture evolves a further quantity of hydrochloric acid gas and chlorine, and leaves a black porous residue containing magnesium sulphate and a magnesium manganite of the composition  $\text{Mg}_3\text{Mn}_2\text{O}_8$ . The porous lumps formed in this way are introduced into an earthenware tube surrounded by a thin iron sheeting, and are heated to a temperature of about  $450^\circ \text{C}$ ., either directly or by means of a regenerative furnace. This tube is connected with two iron tubes, through one of which hydrochloric acid gas is led after it has been treated in a separate furnace, and through the other a current of air passes which has also been heated to the same temperature. The further end of the cylinder has also a tube attached to it which carries off the chlorine gas produced in the cylinder by the joint action of the air and hydrochloric acid passing over the heated magnesium manganite. The plant necessary for heating the gases introduced into the cylinder in which the reaction takes place has no special form; in fact, any plant at present in use for heating the air of a blast furnace can be used. The part played by the magnesium sulphate in the reaction, other than that of preventing the fusion of the mass, and thus maintaining its porous nature, is not known. Apparently the change that takes place consists in the alternate chlorination and oxidation of the magnesium manganite. These changes may be theoretically expressed by the following equations:—



In practice, however, the above reactions go on simultaneously, and the hydrochloric acid gas is not entirely decomposed, there always being a small percentage of this gas in the gases passing out of the cylinder in which the reaction takes place. The authors have investigated the conditions under which the maximum yield of chlorine is available, and have found that it not only varies with the temperature of the reacting compound, but depends also on the relative amounts of hydrochloric acid gas and air, and the rate at which the mixed gases pass through the cylinder.

It will be noticed that although the process is an alternating one, yet chlorine is evolved at both stages, and that therefore the method may be considered to be just as continuous as the Deacon process. The Brin's oxygen process, as now worked, offers a similar example of two alternate reactions taking place apparently at the same temperature. It may, however, be found more economical to employ two cylinders so heated that one shall be in the chlorinating stage while the other is in the oxidising stage, and thus secure a constant rate of production of the chlorine gas. The inventors point out that the cost of fuel and of labour is less than in the Deacon process, and the plant is much simpler than that used by the Péchiney method. The manganite is found not to require renewal for a long while, and then only a little is needed at a time, and the high percentage of chlorine in the gas which is evolved renders it possible to prepare directly therefrom bleaching powder of high chlorine content. The plant erected at MM. de Naeyer et Cie's works has now been at work some time, and has proved capable of continuously decomposing 76 per cent. of the hydrochloric acid used, and of the remainder four-fifths have been recovered and utilised in the process.

# PATENTS.

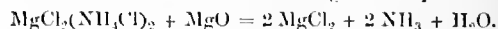
*Improvements in the Recovery of Chlorine from the Residual Liquors of the Ammonia-soda Process, from Solutions of Calcium Chloride, from certain Impure Solutions of Magnesium Chloride, and from some other Industrial Residues.* F. M. Lyte, London, and J. G. Tatters, Runcorn. Eng. Pat. 17,217, October 30, 1889. 6d.

"THE object of this invention is the production in the free state of the whole or nearly the whole of the chlorine contained in the calcic chloride resulting from the ammonia-soda process, in the magnesic chloride contained in the mother-liquors of salt works and the liquor from the treatment of carnallite, and in the hydrochloric acid or calcic chloride from the Leblanc or Weldon processes respectively."

The process depends upon the production of anhydrous magnesium oxychloride, of varying composition, from anhydrous ammonium-magnesium double chloride, the chlorine being evolved from the oxychloride in the ordinary way by passing over it air or oxygen while it is maintained at a suitable temperature.

In treating ammonia-soda waste liquors the two following methods of working are described, but the process admits of various other modifications. The liquor which remains after distilling off the ammonia with lime in the ammonia soda process is the liquor to be dealt with. It consists chiefly of calcium and sodium chlorides. In both the methods described this liquor is first freed from sodium chloride, and this is effected by concentrating it until "on cooling to ordinary temperatures, the calcium chloride it contains would form a saturated solution, or till the boiling point rises to  $110^\circ \text{C}$ . or thereabouts." During the evaporation most of the sodium chloride is deposited and the rest separates on allowing the liquor to cool and repose. This depends upon the insolubility of sodium chloride at ordinary temperatures in a concentrated solution of calcium chloride. Potassium chloride may be separated in the same way but not quite so completely. According to the first mode of working a sufficient quantity of soft burnt magnesia is added to this purified calcic chloride solution, and it is at the same time diluted to about 1.2445 sp. gr. at  $15^\circ \text{C}$ . By treatment with carbonic acid the lime is then precipitated and a solution of magnesium chloride obtained. To this is added sufficient ammonium chloride (obtained subsequently by sublimation from the anhydrous double ammonium magnesium chloride) to form ammonium magnesium chloride, and the solution of this salt is then evaporated to dryness and desiccated. On heating this salt, ammonium chloride volatilises and is used for making a fresh batch of the double salt, and anhydrous magnesium chloride remains. This is fused or ground and mixed with a sufficiency of dry magnesia (left from a previous generation of chlorine) and agglomerated by heating in a closed vessel, forming anhydrous magnesium oxychloride which may be broken up and used for generating chlorine. The patentees claim that whereas Péchiney recovers only 40.14 per cent. of the chlorine contained in his oxychloride, they on the other hand obtain about 60 per cent. of the chlorine in their anhydrous oxychloride, and that they avoid the cooling of the furnace caused by the evaporation of the water contained in Péchiney's oxychloride.

The patentees consider the second mode of working the most important. After obtaining the calcium chloride solution free from sodium chloride, it is saturated with ammonia and carbon dioxide, which produces a solution of ammonium chloride and precipitates the lime as carbonate. The solution of ammonium chloride is then mixed with sufficient magnesium chloride and evaporated to dryness. The dry ammonium magnesium chloride thus formed is then heated with magnesia, at a temperature not exceeding  $300^\circ \text{C}$ ., under which conditions magnesium chloride is formed and ammonia evolved according to the equation—



On subsequently raising the temperature to  $400^\circ \text{C}$ . or  $500^\circ \text{C}$ . a further reaction takes place between the magnesium chloride and the water formed at the lower temperature according to the equation—



Half the magnesium chloride being decomposed with the formation of magnesia and evolution of hydrochloric acid. "By this second heating, if the fusing point of magnesium chloride be attained, the magnesium chloride and magnesia formed will agglomerate entirely into an oxychloride, varying in composition according to the amount of magnesia present." This oxychloride may then be used in the ordinary way to generate chlorine. By this mode of working, therefore, besides the production of magnesium oxychloride, all the ammonia used in the process is recovered, and may be used for again saturating the calcium chloride liquor, and also the hydrochloric acid recovered may be passed into a milk of magnesia, forming magnesium chloride, to be again used for mixing with the ammonium chloride solution to produce the double chloride.

The heating may be done in a closed iron vessel as, provided all air is carefully excluded, no chlorine is evolved, and neither anhydrous hydrochloric acid nor ammonia has any action on hot iron. A small excess of magnesia and stirring the mixture assists the operation.

Both modes of working may be applied to recover chlorine from the hydrochloric acid of the Leblanc process, and from the calcium chloride from the Weldon process, but not so advantageously. In the case of hydrochloric acid, one half of the acid taken is neutralised with ammonia and the other half with magnesia or magnesite. When clear the two solutions are mixed, forming a solution of the double salt, from which ammonia, hydrochloric acid, and magnesium oxychloride can be obtained successively, as already described. The oxychloride yields chlorine and magnesia, the latter of which, with the ammonia, can be again used for saturating hydrochloric acid.

In treating the impure magnesium chloride contained in the mother-liquors of salt works and the liquor from the treatment of carnallite, the whole of the magnesium contained in the liquors as magnesia is precipitated by the addition of lime. The resulting solution of calcium chloride is then freed from sodium or potassium chloride by concentration, whilst the precipitated magnesia is washed free from sodium and potassium chlorides and returned to the purified solutions of calcium chloride, which is then diluted and treated with carbon dioxide. This throws down the lime and gives a pure solution of magnesium chloride, as described in the first method of working, or the pure calcium chloride solution may be worked according to the second method described (see following abstract).—H. S. P.

*Improvements in the Production of Hydrochloric Acid from certain Industrial Residues, together with the Recovery of Sulphur and Oxide of Magnesium.* F. M. Lyte, London, and J. G. Tatters, Runcorn. Eng. Pat. 17,218, October 30, 1889. 6d.

ANHYDROUS magnesium chloride or oxychloride is produced from the waste calcium chloride liquors of the ammonia-soda process, according to the method described in Eng. Pat. 17,217 (preceding abstract). Sulphuretted hydrogen is also produced from the soda waste of the Leblanc process as follows:—The waste "is to be mixed with water until it marks about 50° Tw., and then run into closed iron cylindrical vessels. Carbonic acid is then pumped through it, as in Chance's process." The sulphuretted hydrogen, together with just sufficient air, is then passed over magnesium chloride or oxychloride, heated to 600°–800° F., so that the decomposition of the chloride or oxychloride is effected without the formation of sulphurous acid or leaving any sulphuretted hydrogen unburnt. Gaseous hydrochloric acid and sulphur pass over. The sulphur is condensed in brick chambers, while the hydrochloric acid passes on, and may be either used in the gaseous state or may be condensed in the usual way.—H. S. P.

*Improvements in and Relating to Chemical Fire-extinguishing Compounds.* C. M. Martin, Washington, U.S.A. Eng. Pat. 17,517, November 4, 1889. 4d.

THE compound is made of 60 lb. of sodium chloride, 30 lb. of "sodium hyposulphate," 6 lb. of sal-ammoniac, 2½ lb. of magnesium sulphate, ½ lb. of magnesium chloride, ½ lb. of calcium oxide, and ½ lb. of iron oxide. The mixture is "dissolved in water" in the proportion of 3 lb. to a gallon of water. "When thrown on a fire it at once emits sulphurous acid gas in combination with free chlorine and ammoniacal gas, which instantly extinguishes the flames." —H. S. P.

*Improvements in the Manufacture of Alum.* F. M. Spence and D. D. Spence, Manchester. Eng. Pat. 17,888, November 9, 1889. 6d.

THE inventors use the following plan to form well crystallised soda alum, a body much harder to obtain in this condition than the corresponding ammonia and potash alums. A hot highly-concentrated solution of the alum, produced by dissolving in aluminio-ferrie sulphate the proper amount of salt cake, is run into a cold concentrated solution of the same salt in the proportion of two to one, a magna being produced upon cooling. This is thoroughly stirred and turned over, crystals of the alum being gradually formed which may now be readily separated by known means from the mother-liquors. These latter, after the removal of an amount containing iron equal to that introduced by the hot concentrated solution, constitute the cold concentrated solution already mentioned.—S. G. R.

*Improvements in the Treatment of Copper or Cupreous Compounds for the Production of Sulphate of Copper and the Extraction of the Precious Metals, and in Apparatus applicable therefor.* T. Haeghe, Briton Ferry, and "The Cape Copper Company, Limited." Eng. Pat. 18,297, November 15, 1889. 8d.

See under X., page 1134.

*Improvements in or connected with the Manufacture or Production of Ammoniacal Salts.* E. Bowen, Swansea. Eng. Pat. 18,356, November 16, 1889. 4d.

INSTEAD of passing steam into the mixture of lime and ammoniacal liquor, heated air is blown in. It is found that the resulting mixture of vapours will completely precipitate the iron (ferrous) in solution in acids such as sulphuric or hydrochloric, a clear solution of the ammoniacal salt being obtained, a result which is not attained by the usual method. The air may be heated by the waste heat derived from the ammonia still.—S. G. R.

*Improvements in the Manufacture of Hypophosphites.* J. A. Kendall, London. Eng. Pat. 20,392, December 18, 1889. 4d.

IN order to obtain the hypophosphites of various metals it has been found to be more advantageous to incorporate charcoal with the phosphorus before the addition of the latter to the milk of lime or other hydrate used. The charcoal or powdered coke is made into a thin cream with water and mixed by means of heat and agitation with the phosphorus. When cool the mixture is heated with the desired hydrate till the reaction is complete.—S. G. R.

*Improvements in obtaining Chlorine.* W. Donald, Salt-coats. Eng. Pat. 5488, April 11, 1890. 8d.

The chlorine is generated by passing hydrochloric acid gas into a mixture of nitric acid and sulphuric acid, cooled by cold water or by brine at, or a little below, the freezing point. The chlorine and nitric peroxide thus formed then pass into another vessel containing nitric acid, and a second stream of hydrochloric acid gas is also introduced into this vessel. The chlorine is finally purified by passing through a nitric acid scrubber and by washing with sulphuric acid. The nitrous acid formed may be oxidised in any well-known way, either in separate apparatus or by introducing air with the hydrochloric acid, or with the mixture of chlorine and nitric peroxide.

Drawings of a suitable apparatus accompany the specification. (See also this Journal, 1887, 822; and 1890, 389.) —H. S. P.

*Improvements in and relating to the Manufacture of Carbonates of Alkalis and Acetone.* F. W. A. Frerichs, St. Louis, U.S.A. Eng. Pat. 9793, June 24, 1890. 4d.

CALCIUM acetate is mixed with potassium sulphate in equivalent proportions and after filtration the potassium acetate formed is evaporated to dryness and destructively distilled with the evolution of acetone, potassium carbonate remaining behind in the retort. Barium and strontium acetates and sodium sulphate may also be employed.

—S. G. R.

*Process for obtaining Chlorine and Bromine by the Aid of Electricity.* G. Nahsen, Hanover, Germany. Eng. Pat. 11,699, July 26, 1890. 4d.

See under XI., page 1136.

## VIII.—GLASS, POTTERY, AND EARTHENWARE.

*The Cause of the Iridescence of Table Glass.* A. Jolles and F. Wallenstein. Sprechsaal, 23, 685.

THE iridescent film in glass is partially soluble in water, the remainder being unattacked by hydrochloric acid, but yielding easily to caustic soda. Both solutions contain sodium, sulphuric acid and carbonic acid. The portion insoluble in acid can only be silica, no lime being found even by the spectroscope. The film exists only on one side of the glass, and must be formed during the final heating, being probably caused by the sulphurous acid in the burning gases, which acts on the surface of the glass, forming sodium sulphate and silicate, the latter being subsequently decomposed into free silica, which separates out in the amorphous form.

—F. H. L.

*Note on the Production of Kaolin.* A. Simon. Zeits. f. angew. Chem. 1890, 357—358.

IN the southern part of the Department of Allier, France, granite, the felspar of which has been decomposed, to a depth of about 35 metres, by atmospheric influences, into silicic acid, alkaline silicates and aluminum silicate (kaolin), is so treated with running water that, by suitable regulation of the flow of the latter, fine quartz sand, mica, &c. are first separated, the water containing the suspended "kaolin" passing afterwards into vessels in which the pure clay is deposited. The water is drained off from the deposited kaolin, which is transferred in a moist state to another vessel, in which it is removed to the drying chamber. The dried kaolin is chiefly used in porcelain, crockery-ware,

ultramarine and paper factories. When it is required for paper manufacture, a little methylene blue, or the like, is added in aqueous solution to the water in the settling tanks. —E. G. C.

*On the Composition of Kaolin Porcelains.* G. Vogt. Bull. Soc. Chim. (3), 4, 343—350.

THE analysis of the Chinese porcelains yields results which have been assumed to prove that their composition is about as follows: Kaolin, 50 per cent.; felspar, 30 per cent.; and quartz, 20 per cent. The author, by treating with sulphuric acid some of the actual raw materials as used in China, has extracted a soluble portion which agrees in the amount of its components practically exactly with white mica, both containing about 45·5 per cent. of silica, 37·5 per cent. of alumina, and 6·0 per cent. of water of combination, together with potash and soda. In France it was also believed that pegmatite is analogous in composition to the Chinese minerals yeon-ko and petun; but in reality, as seen in the subjoined table, this is far from being the case:—

	Yeon-Ko.	Pegmatite.
	Per Cent.	Per Cent.
Quartz .....	52·9	23·9
White mica .....	31·3	..
Potash felspar .....	..	72·8
Soda felspar .....	13·4	
Calcium carbonate .....	2·0	..
Soluble silica .....	1·0	..
Soluble matter not estimated ....	..	3·3

In all the different materials used, conclusive proof has been found of the presence of mica. This is in a state of exceedingly fine division, but it can be recognised both by analysis and by microscopic examination with polarised light. A comparison of the imperial porcelain of China with that made at Sèvres reveals the great difference between the two manufactories:—

	China.	Sèvres.
	Per Cent.	Per Cent.
Kaolin (2 SiO <sub>2</sub> , Al <sub>2</sub> O <sub>3</sub> , 2 H <sub>2</sub> O) ....	23·4	35·6
Mica (6 SiO <sub>2</sub> , 3 Al <sub>2</sub> O <sub>3</sub> , K <sub>2</sub> O, 2 H <sub>2</sub> O) ..	23·4	..
Soda felspar .....	25·0	38·0 with potash felspar.
Quartz .....	28·2	26·4
	100·0	100·0

A mixture was made of—

	Parts.
Pure kaolin .....	25
Mica .....	25
Orthose .....	25
Quartz .....	25

which gave a fine, beautifully transparent porcelain. It will be noticed that these are substantially the proportions taken in the Oriental porcelain. It appears to be difficult to find in France materials sufficiently rich in pure mica for use on a large scale.—S. G. R.

*The Kaolin in Blandford, Massachusetts.* W. O. Crosby.  
Technology Quarterly, 3, 228—237.

THIS paper is a description of the kaolin beds of Blandford, which have only within the past year been sufficiently opened up to allow of an examination of the geological surroundings and the formation of an opinion as to the origin of the deposits. The geological situation is described in detail, and from a consideration of it the kaolin has apparently been derived from large veins of a coarse pegmatite or endogenous granite, containing little mica, and consisting chiefly of felspar. It is clearly the product of decomposition *in situ* in pre-glacial times—a genuine remnant of an ancient sedentary soil, which has escaped the erosive action of the great ice-sheet. The following is an analysis:—

	Per Cent.
Silica .....	52.03
Alumina .....	31.76
Water .....	15.55
Magnesia .....	0.54
Oxide of iron .....	Trace.
Lime .....	Trace.
Alkalis (K <sub>2</sub> O and Na <sub>2</sub> O) .....	Trace.
	99.88

The kaolin shows throughout the structural features of pegmatite, and contains undisturbed groups of crystals, half-decayed mica crystals, small grains and fragments of semi-decomposed felspar, and scattered crystals of tourmaline. The last-named are sometimes a foot in length, and although rotted to a coarse black earth, are still essentially intact in form. All these facts tend to disprove the possibility of the kaolin having been washed into its present position. The author then proceeds to give his reasons for considering this a pre-glacial sedentary soil, such soils being known to exist elsewhere in New England.—A. W.

#### PATENT.

*Improvements in the Manufacture of Fire-clay Pipes called "Swan-Necks."* A. Griffiths, Bonnybridge. Eng. Pat. 20,372, December 21, 1889. 6d.

LENGTHS of clay tube partially formed in an ordinary machine, and while still moist, are introduced into a horizontally disposed flask or box of circular section made in two superposed and detachable semi-cylindrical halves. A tubular die is advanced from each end of the flask by tubular screws to form the pipe ends, and a ram or plunger passed through the dies and screws forms the interior of the pipe. A plate fixed to the bottom of the flask with an attached handle serves to remove the completed pipe.

—S. B. A. A.

### X.—METALLURGY.

*The Dry Assay of Tin Ores.* Part II. H. O. Hofman.  
Technology Quarterly, 3, 261—280.

See under XXIII., page 1154.

*Aluminium Steel.* R. A. Hadfield, Sheffield. Meeting Iron and Steel Inst., U.S. America, October 3, 1890.

HAVING mentioned the principal methods of producing the metal itself, reference may be made to the already large employment of aluminium alloyed with copper, known as aluminium-bronzes, for quite a countless number of purposes. It may be interesting here to state, as one of the first public notices, that in the "Morning Star" of May 21, 1862, considerable reference was made to the probability of aluminium-bronze coming into extensive use. The many good qualities of these alloys will no doubt make the use of aluminium in

this direction alone of very wide importance. The following table gives the range of tensile strength and density of these forged aluminium-bronzes:—

#### TESTS OF ALUMINIUM BRONZES.

(By John H. J. Dagger, in a Paper read before the British Association, 1889.)

		Tensile Strength in Tons per Square Inch.	Elongation.	Density.
			Per Cent.	
Aluminium-bronze.	11 % Al. ...	49 to 45	8	7.23
	10 " ...	33 " 40	14	7.69
	7½ " ...	25 " 30	40	8.00
	5-5½ " ...	15 " 13	40	8.37
	2½ " ...	13 " 15	50	8.69
	1½ " ...	11 " 13	55	"

The brittleness of alloys above 11 per cent. prevents their use. Those containing 60 per cent. to 70 per cent. aluminium are very brittle and beautifully crystalline; with 50 per cent. the alloy is quite soft, but under 30 per cent. the hardness returns. The 20 per cent. bronze has a whitish-yellow tint, somewhat resembling bismuth, but is very brittle, and can be pulverised in a mortar. One of the most valuable properties of the alloys given in the above table is that of being forgeable and capable of being worked at a red heat. Table B, having been specially prepared by Professor Tetmayer, of the Polytechnic School, Zurich, for the Aluminium-Industrie Actien Gesellschaft, at Neuhausen, is given for comparison with the foregoing:—

	Aluminium.	Tensile Strength in Tons per Square Inch.	Elongation.
	Per Cent.		Per Cent.
Aluminium-bronze.	11.50	52	0.50
	11.00	44	1.00
	10.00	42	11.00
	9.50	49	19.00
	9.00	37	32.00
	8.50	33	52.50
Aluminium-brass ...	5.50	28	64.00
	4.00	45	6.50
	3.00	33	7.50
	2.50	34	20.00
	2.00	31	30.00
	1.50	29	39.00
	1.00	26	50.00

Professor Tetmayer has plotted his results, the curve obtained showing that with increasing aluminium content the tensile strength increases slowly at first, but then grows rapidly as the alloy is made richer in the lighter metal.

*Alloys of Cast Iron and Aluminium.*—As with other cast iron, aluminium cast iron, if it may be so termed, naturally comes under a different classification to that of the malleable compounds of aluminium and iron. Keep found that aluminium has, in a considerable degree, a similar influence to that of silicon upon cast iron, a fact strikingly confirmed in some special experiments of the writer relative to the action of aluminium upon combined carbon in spiegeleisen, and

described later on. It is, however, only proposed to deal here with aluminium in its use in the manufacture of steel or steady compounds. There appears to be much misconception as to whether its employment is productive of good, and the author confesses that he believes that whilst the price remains so high, and except in certain special cases, its application does not seem likely to become large. Aluminium appears to be of most service as an addition to baths of molten iron or steel unduly saturated with oxides, and this in properly regulated steel manufacture should not often occur. Speaking generally, its rôle appears to be similar to that of silicon, though acting more powerfully. It must, however, be remembered that these experiments have been made with practically pure aluminium. If pure silicon were also obtainable, its effect would probably be found to be almost the same. So long, therefore, as ferro-silicon varying in silicon from 8 to 20 per cent., can be obtained at from 3*l.* 10*s.* to 10*l.* per ton, as compared with aluminium or ferro-aluminium of like percentages costing 112*l.* to 250*l.* per ton, it will be seen that the probable field of usefulness for the latter must be much circumscribed.

#### CAST ALUMINIUM STEEL.

**Melting.**—The material obtained for these experiments was produced by melting in crucibles, in the ordinary manner, good wrought bar iron, and adding the aluminium (about 98 per cent. of aluminium), manufactured by the Pittsburgh Reduction Company's system, shortly before "teeming." Although a difficult one, the object was to obtain an alloy or compound consisting as nearly as possible of aluminium and iron alone. In the material made it will be found that the other elements present do not amount to more than 50 per cent., so that this intent may fairly claim to have been accomplished. The ingots, 2½ in. square, were reduced by forging in the ordinary manner to bars 1½ in. diameter. The consideration of the qualities of these alloys is divided into two heads: first, the material in the cast; second, the material in the forged state.

**Cast State.**—Nothing special was noted during the melting operations, but in all cases upon adding the aluminium "coruscation" was observed. This evolution of heat and light has also been observed when alloying aluminium with copper. Does not this tend to indicate that aluminium added to iron produces true compounds and not merely alloys, as it is well known in laboratory operations that the evolution of heat during the mixture of two substances is often a sign of chemical union? The curious properties of manganese steel seem especially to prove that such a material is a true compound, and no doubt aluminium steel may be classed under the same head.

**Fluidity.**—It is doubtful whether aluminium increases the fluidity of properly made steel, but if it does, the apparent increase or evolution of heat just referred to seems to the author far more likely to account for any increase of fluidity that may occur rather than the so-called lowering of the melting point. As the aluminium increases to large amounts over about 0.50 per cent., the metal becomes quite thick, "creamy," and sets quickly. No doubt this is partly caused by a considerable portion of the aluminium being oxidised to alumina and becoming entangled as slag, in the same way as occurs when silicon is oxidised to silica. Unless suitable flux is present, this excess of oxide cannot be carried off, the molten metal becomes less fluid, and causes the "thickness" noticed. There is a resemblance in this respect to the action of high percentages of silicon added to iron. When exceeding about 0.75 per cent. of aluminium the molten material so rapidly "creams" over on the surface, that the production of sand-moulded articles is only effected with difficulty—in fact, it was only by using considerable care that the cast test bars were obtained. (Keep, even with cast iron, found a decided decrease in fluidity by the addition of aluminium.) It must not be overlooked that the author is here referring to comparatively high percentages. So far as his own experiments have gone, aluminium, as regards lower percentages, also acts in a similar manner to silicon under the same circumstances.

So much has been said as to aluminium lowering the melting point of iron that the author wonders how and why this statement was originated, as it certainly seems to have no foundation in fact. This is confirmed by Osmond, who by means of the "Le Chatelier" pyrometer (this Journal, 1890, 326) kindly made for the author a special determination as to the melting point of an alloy containing 5 per cent. Al and about 94½ per cent. Fe. The sample in question did not show the slightest signs of fusion until a temperature of 1,475° C. was reached. As mild steel by the same pyrometer was shown to fuse at about 1,500° C., there could therefore have been little or no lowering of the fusion point; and if this is the case with an alloy containing 5 per cent., still less can it be so with one containing only 10 per cent., which is the quantity usually described as being present when the so-called lowering point is supposed to occur. It is especially satisfactory to have the testimony, not only of Osmond (this Journal, 1890, 517 and 865), but that of H. M. Howe (this Journal, 1890, 946), who both agree with the author on this point. In the author's opinion, if any increase of heat or fluidity takes place by the addition of small amounts of aluminium, it may be due to either of the following causes, or to both combined. First, there may be evolution of heat owing to oxidation of the aluminium, as the calorific value of this metal is very high—in fact, higher than silicon. According to Berthollet, the conversion of aluminium to  $Al_2O_3$  equals 7,900 cals., silicon to  $SiO_2$  is stated as 7,800. This oxidation is probable, as in the writer's experiments the addition of aluminium to iron was always accompanied by more or less, often considerable, loss of the former metal. This is also proved by the fact of its appearing in the oxidised state in a white powder upon the side of the ingot mould, as well as in the slag; also from the fact that when small amounts are added they are rarely found in the product when being analysed. In the 5 per cent. specimens previously referred to, the author found no less than 2 per cent. was wasted. Against the supposition that by oxidation increase of heat is obtained must, however be placed the fact that when the aluminium exceeds, say, about 75 per cent., the less fluid is the product and the more metal is wasted; but at the same time the explanation first offered might still hold good as regards the lower percentages, say under 50 per cent. Secondly, and more probably, the correct explanation. As proved by Galbraith, the fluidity of molten iron and steel depends not only upon the amount of heat imparted by external means or by oxidation, but often upon the quantity of intermingled slag or oxide of iron (this Journal, 1890, 864—865). He found that in certain cases in excessively over-oxidised steel, although the heat of the furnace was more than usual, the product was still lacking in fluidity. It would be interesting to have the point followed up and thoroughly tested by means of the "Le Chatelier" pyrometer. As supporting this, it may be observed that the well-known German metallurgist, Ledebur, believes the rôle of aluminium "to be no other than that of destroying the dissolved oxide of iron present." That such over-oxidised metal possesses different properties as compared with ordinary steel or iron is very clear, for the author has had brought under his notice certain cases of over-oxidised iron which continued to rise, give off gases, and prove unsound in the ingots, notwithstanding the addition of even considerable quantities of aluminium. They are, of course, exceptional, and to the author seem to be explained by the facts pointed out by Galbraith. The loss of the metal in melting is variable, but in nearly all cases amounts to a considerable proportion of the percentage added.

The experiments prove that more aluminium may be present in the cast material than silicon in cast silicon steel before brittleness sets in. After reaching the region of brittleness, and by adding still higher percentages of aluminium, there is no return of strength, on the contrary; so that this material in no way resembles the curious property noted in this respect with regard to manganese steel.

**Bending Tests.**—The annealed samples up to 85 per cent. bent double cold; those unannealed were not so good. The gradual increase of aluminium is very clearly shown to



reduce the toughness both of the unannealed and annealed specimens. They were sound with the exception of two samples, but not more so than cast silicon steel of corresponding percentages.

**Hardness.**—As might be expected from the soft nature of the metal aluminium, its addition to iron does not add materially to the hardness, again in this respect resembling the effect of silicon. Seven to 8 per cent. cast aluminium steel may be readily drilled and filed. Neither does its addition, unlike manganese, interfere with the magnetic susceptibility of the iron present.

**Appearance of Fracture.**—Aluminium in the cast specimens opens the grain, *i.e.*, increases the size and coarseness of the crystals, and as the higher percentages are reached it is somewhat difficult to distinguish the fractures from those of cast silicon steel. The crystals in the 5 per cent. and upwards specimens become very large and cleave somewhat after the nature of spiegelstein. This accounts for the increasing brittleness of the cast material, and neither the fracture nor the brittleness accompanying it in such samples can be changed by annealing.

#### FORGED ALUMINIUM STEEL.

**Malleability.**—As high as 5.60 per cent. aluminium may be present before malleability ceases. This is about the same limit as in the silicon steel described by the writer. It may be pointed out, as regards the specimens under discussion, that they are very low in manganese. Notwithstanding this, the malleability is satisfactory.

**Mechanical Properties, &c.**—Up to 2.24 per cent. aluminium the annealed samples bent double cold, but upon reaching 5 per cent. a great diminution of strength took place, and annealing practically produces no effect.

One difference clearly brought out is, that the tensile strength of iron is not increased so much by an addition of aluminium as of silicon. It has been stated that very small percentages of aluminium considerably raise the limit of elasticity. It will be seen from the table of tests, and specially from the two almost carbonless samples just quoted, that this increase is slight, if any, as compared with ordinary steel, and also that aluminium does not raise the elastic limit so much as silicon. Seeing that aluminium is an exceptionally soft and malleable metal, possessing a tensile strength of 8 to 10 tons, it is hardly to be expected that this should have been otherwise, and it is a pity that such incorrect statements as above mentioned are started, without foundation in fact. The elastic limit of samples should always be determined on annealed specimens, as by cold forging or rolling, the elastic limit of even mild steel can be readily raised several tons per sq. in.; and it would hardly be correct to call this the normal permanent set of the sample under examination. Aluminium and silicon, therefore, stiffen iron but little, whilst carbon, chromium, tungsten, manganese, and nickel clearly increase rigidity; and this, from their nature and hardness, might be expected.

**Hardness.**—As might be expected from the soft nature of the metal aluminium, its addition to iron does not confer appreciable hardness when in either the forged or cast state. In this respect also it resembles silicon steel. The forged material containing 5 per cent. is very soft and easily drilled or filed. The action of aluminium may therefore be classed along with that of silicon, sulphur, phosphorus, arsenic, and copper, as giving no increase of hardness to iron, in contra-distinction to carbon, manganese, chromium, tungsten, and nickel. Water quenching upon either forged or cast aluminium steel (in which carbon is practically absent) seems to produce no effect, the samples when dipped at even a welding heat being almost unchanged. It has been stated that the influence of aluminium on high carbon or tool steel is to destroy its hardening action. This is quite incorrect; the writer has prepared steel containing  $1\frac{1}{2}$  per cent. aluminium and 1 per cent. of carbon, which hardens sufficiently to scratch glass. Here again we have another addition to the lists of elements which, alloyed with iron, do not produce "water-quenched hardness," if the term may be allowed. In fact, notwithstanding that

manganese, chromium, tungsten, and nickel do impart a certain and considerable kind of hardness to iron, either in the cast or forged state, carbon retains its position as being the only element which confers upon iron the property of becoming hard by water-quenching. In steel made with the hard metals just mentioned, if the carbon present be low, no matter at what heat they are water-quenched, the characteristic hardness of carbon steel, such as scratching glass or forming the edge of a cutting tool, is wanting.

**Fracture.**—Aluminium causes the tough percentages, both in the cast and forged material, to break with dark fibrous fracture, similar to wrought iron of good quality, only much darker in colour. In several of the tested tensile specimens a rough and ribbed surface is noticed, proving looseness of texture.

**Weldability.**—Samples with .61 per cent. of Al, .72 per cent. of Al, and 2.20 per cent. of Al were tested for welding, but unsuccessfully. It would be interesting to try electric welding. The writer has always found that these special steels or alloys, such as silicon, manganese, or chromium steel, have slight, if any, welding properties.

**Application.**—If aluminium can eventually be brought to compete in price with existing alloys, there is a probability of something being done in introducing its use on a larger scale. Aluminium may find a use for certain special purposes, such as in higher carbon steel, where silicon does not seem to act so powerfully in producing soundness as in the case of milder steel with carbon .50 per cent. and under. Its special advantage seems to be that it combines in itself the advantages of both silicon and manganese; but so long as alloys containing these metals are so cheap, and aluminium dear, its extensive use seems hardly probable.

**Points of Resemblance between Aluminium and Silicon Steel.**—Osmond wrote to the author as follows:—"In 1833 I made some aluminium steel, and found its tensile strength and elongation were modified almost exactly as by the same proportions of silicon. Second, aluminium, like silicon, causes a precipitation of graphite in cast iron. Third, the cooling curve of the aluminium alloy is nearly the same as that of the 4.50 per cent. silicon alloy. Fourth, aluminium and silicon have almost the same atomic volume."

**Effect of Aluminium upon Manganese Steel and Spiegelstein.**—Believing that aluminium, like silicon, would cause a precipitation of graphite, the writer added between 3 and 4 per cent. to ordinary spiegel (12 and 25 per cent. of manganese). The result in both cases was a complete change from the well-known spiegel fracture to that of ordinary close No. 3 grey pig iron. Although there has been this great attraction in the form of the carbon, no change in the non-magnetic properties of the iron was apparent. This experiment, therefore, clearly proves that the neutralisation of the special magnetic qualities ordinarily possessed by iron is in manganese compounds quite independent of the particular state or form in which the carbon present exists.

**On the Removal of Silver by Means of Zinc.** H. Rössler and B. Edelmann. *Berg. und Hütten. Zeit.* 49, 245.

LEAD and zinc are increasingly soluble in one another as the temperature rises, as the following table shows:—

Temperature.	Zinc contained in the Lead.	Lead contained in the Zinc.
	Per Cent.	Per Cent.
400° (melting point of zinc) .....	0.6–0.8	1.7
500° .....	0.9–1.3	2.3
600° (melting point of aluminium) .....	1.5–2.3	3.5
700° (bright red heat) .....	Over 3.0	5.6

When a considerable excess of zinc is present, it rises to the top, carrying with it the silver from the lead. In working



on the large scale a large amount of zinc is undesirable, but under these conditions it is difficult to avoid loss by oxidation, and by the formation of a scum containing more lead than zinc. To prevent this loss it is advisable to cover the mass with a layer of melted sodium potassium chloride, which also assists the separation of the metals. By this method alloys can be produced containing 1 part of silver to 3 of zinc.

Further experiments, on removing the zinc from the lead by the action of indifferent gases, such as carbonic acid, either alone or mixed with nitrogen, or carbonic oxide with nitrogen and hydrogen, proved completely successful, especially if the gases be employed hot, and the temperature of the metal be kept as high as 700°. (See also this Journal, 1890, 77.)—F. H. L.

#### PATENTS.

*Improvements in the Treatment of Copper or Cupreous Compounds for the Production of Sulphate of Copper and the Extraction of the Precious Metals, and in Apparatus applicable therefor.* T. Haeghe, Briton Ferry, and "The Cape Copper Company, Limited," London. Eng. Pat. 18,297, November 15, 1889. 8d.

COPPER in leaf or granular form, or in shots, is charged into a lead-lined tub provided with a perforated lead false bottom supported on bricks or other material which can resist the action of hot sulphuric acid. The tub is covered with a perforated lead tray on which is poured a continuous stream of hot dilute sulphuric acid which gradually percolates through the metal. Air or steam is injected by a blast pipe placed immediately under the false bottom, and the acid liquors are drawn off by an exit pipe lower down. The action of the descending acid and ascending steam and hot air effects the rapid dissolution of the copper; the hot concentrated solution of copper sulphate which flows out from the lower pipe is crystallised in troughs, and the mother-liquor strengthened with acid and used again.

Any gold or silver contained in the copper is left in the metallic condition at the bottom of the tubs and troughs.

—S. B. A. A.

*Improvements in the Manufacture of Metallic Alloys and Compounds and in Apparatus therefor.* J. C. Bull, Belvedere. Eng. Pat. 19,148, November 28, 1889. 4d.

THE author prepares copper and tin phosphides "without loss of phosphorus" by passing phosphorus vapour into the molten metals. The phosphorus is volatilised in a cast-iron retort similar to that used for the distillation of mercury, but fitted with a valved iron tube at the further end of which there is a bend and a vertical end-piece of fireclay or carbon, and with a spring safety valve and tube for escaping vapours. The retort is at first filled with carbonic anhydride, and is heated steadily to a red heat. When a saturated alloy of a metal with phosphorus is required, the vapours are led into the metallic bath until they begin to burn on the surface. To prepare other compounds the retort and tube are counterpoised before starting, and the diminution of the weight observed from time to time, the retort being prevented from moving more than a fraction of an inch. Other volatile substances, as sodium, potassium and mercury, may be treated in the same manner.—S. B. A. A.

*Improvements in the Method of and Converters or Machinery for the Manufacture of Iron or Steel.* W. Sellar, Sunderland. Eng. Pat. 19,333, December 2, 1889. 8d.

THE molten metal is run into a square or cylindrical converter mounted on hollow trunnions and provided with a convex hearth, to which an oscillating motion is imparted by suitable mechanism. Air or a mixture of air and gas is injected through the hollow trunnions into the converter above the level of the metal, and at the close of the reaction the metal is made perfectly fluid by increasing the supply of gas and air in proper proportions. The oscillation of the converter is continued for some time after the stoppage of

the gas supply, in order to release the gases absorbed by the metal. The metal is then either run out through a tap-hole in the malleable condition or it is converted into steel by the addition of carbon. It is claimed that the passage of the metal in a thin layer over the convex surface of the hearth greatly facilitates the oxidation of the impurities and the expulsion of absorbed gases.—S. B. A. A.

*Improvements in and connected with the Extraction or Reduction of Zinc from Ores or Compounds.* C. Rabache, Morechain, France. Eng. Pat. 19,501, December 4, 1889. 1s. 1d.

THE distinguishing features of this process are the preliminary "releasing of the metallic part of the ore from its gangue by the vaporisation of the water of combination," and the smelting and reduction of the ore by pure carbon monoxide in the absence of atmospheric air. The ore is sorted, crushed to a granular powder 1 to 3 mm. in diameter, and heated for a short time to a temperature of 200°–400° C., by the combustion of carbon monoxide, with continuous shaking. The latter operation effects "the expulsion of the water of crystallisation, sulphur, and other volatile impurities, and the separation of the metallic particles from the gangue;" the ore is then concentrated with or without previous sizing, and the pure zinc oxide powder obtained in this way introduced in a continuous stream into a furnace of special construction (see Eng. Pat. 11,851 of 1890, next page) previously heated to a minimum temperature of 2,730° F. by the combustion of carbon monoxide in air. The air supply is then stopped and carbon monoxide alone introduced when "the falling ore is enveloped by the gas and a flameless combustion of the carbon monoxide gas in the oxygen of the ore ensues, resulting in the reduction of the metal, which falls in a molten state on the floor of the smelting furnace, and is removed by suitable means."

—S. B. A. A.

*Improvements in Cleaning Sheets of Wrought Iron, Iron Wire, and Articles of Wrought Iron, and in utilising the Waste Products obtained, and in Apparatus to be employed in conducting the said Cleaning and Utilising Processes.* E. P. Peyton, Birmingham. Eng. Pat. 19,865, December 10, 1889. 8d.

WROUGHT iron plates are pickled in hot dilute sulphuric acid in the ordinary way, commercial oil of vitriol diluted with 10 vols. of water being preferably used at a temperature of 200° F. The improvement consists in allowing the contents of the pickling tank when sufficiently concentrated to flow slowly through a long cooling channel and there deposit the greater part of the dissolved sulphate of iron, the mother-liquor passing into a vessel from which it is continually pumped back into the pickling tank, vitriol and water being added till it is of the proper strength. The process is continuous after being fairly started. Cylindrical pickling tanks are used for cleaning wire.—S. B. A. A.

*Improvements in the Manufacture of Steel or Ingot Iron.* J. H. Darby, Brymbo. Eng. Pat. 20,586, December 21, 1889. 6d.

THE method previously described in Eng. Pat. 418 of 1888 (see also this Journal, 1889, 546 and 549), which consists in running metal from the ladle through a vessel containing carbon into a supplementary casting ladle has the disadvantage of requiring an arrangement of the converter and appliances for casting, absent in many works. The following is an alternative process. The coke or other carbonising material is finely ground and placed in a hopper carried on the ladle crane and communicating with a worm or spiral or with the cavities of a wheel the movement of which discharges a regulated quantity of the carbon down a spout into a small vessel placed between the ladle and the ingot mould. In this vessel the current of metal from the

ladle meets and thoroughly mixes with the carbon, afterwards passing through an opening in the bottom into the ingot mould. The steel produced by this method will stand quiet in the moulds so that the addition of ferro-silicon is unnecessary, whilst the amount of carbon in the same may be arrived at with great certainty. Spiegel or ferro-manganese may be first added to the metal if desired.—S. B. A. A.

*Improvements in Apparatus for Treating Finely-divided Metalliferous Material for the Purposes of Separation and Amalgamation.* W. L. Wise, London. From J. B. Walker, Corvallis, Oregon, U.S.A. Eng. Pat. 20,677, December 23, 1889. 11d.

THE author proposes "to disintegrate the pulp so as to separate each particle of precious metal from the sand and to introduce the pulp particle by particle into the lower part of a mass of mercury." This is effected by means of an apparatus consisting essentially of a drum or pan containing (1) a central shaft; (2) a low pedestal continued into a tube enclosing a portion of the shaft; (3) a disintegrating feed-wheel consisting of a tubular portion secured to the upper free portion of the shaft and continued into a disc overlying the pedestal and containing peripherally inclined and covered flutings or passage ways with centrally raised walls and inwardly facing cutting edges; (4) an annular water reservoir with perforated outer wall surmounting the feed-wheel, the space between its inner tubular wall and the tubular part of the feed-wheel forming a feed chamber; (5) the mercury chamber, consisting of the annular space between the pan casing and the reservoir and pedestal; (6) an annular discharge trough attached to the exterior of the pan; (7) an agitator consisting of three series of concentric floats, one set being fixed to the wall of the reservoir, another set oscillating over the surface of the mercury, and the third set moving in the trough.

The pulp falling from the feed chamber is thoroughly disintegrated by the sharp edges of the walls of the flutes in the discoid part of the feed-wheel, and the particles are separately forced (by the centrifugal force due to the rotating feed-wheel) through the flutes past suitable valves into the mercury surrounding the pedestal by which the gold and silver are retained. The sand rises to the surface of the mercury, where it is met by the flow of water from the reservoir, and is swept away by the agitators into the trough. For details of the valves, feed motion, &c., the drawings in the specification must be consulted.—S. B. A. A.

*Improvements in Apparatus for the Extraction or Reduction of Zinc and other Metals from Ores or Compounds.* C. Rabache, Morchain, France. Eng. Pat. 11,851, July 29, 1890. 11d.

THE apparatus are adapted to the method described in Eng. Pat. 19,501 of 1889 (previous page), and consist of:—(1) a "rotatable" drum fitted with a sand-luted cover and with slanting internal ledges for shaking up the contents, and provided with suitable openings for charging and discharging the ore, and with perforated pipes for introducing carbon monoxide; (2) a small horizontal hermetically-closed smelting furnace terminating in a vertical gorge or gullet and having in the centre a syphon serving as a free and constantly opened outlet for the fused metal.

Claims are also made for a new sizing machine composed of concentric revolving cylindrical screens; for two forms of concentrators or "densimetric separators;" for an apparatus for producing carbonic anhydride from limestone; for an apparatus consisting of a series of enclosed upright retorts for converting the carbonic anhydride into carbon monoxide; and, lastly, for a floating compensating valveless gas holder for storing the carbon monoxide, and for a gas exhaustor and regulator. Four sheets of drawings are appended.

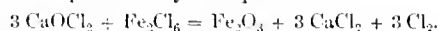
—S. B. A. A.

*Improved Process of and Means for the Separation of Tin from other Metals, more particularly from Scrap Tinplate and used Preserve Tins.* A. A. F. Bang and M. C. A. Ruffin, Paris, France. Eng. Pat. 12,553, August 11, 1890. 8d.

IN Eng. Pat. 70 of 1890 (this Journal, 1890, 950) it was proposed to strip tinplate by means of soda lye and nitrate of soda or other oxidising agent. According to the present specification atmospheric air is used in lieu of nitrate of soda. The scrap may either be placed in a deep tank and treated with soda or potash lye of 15° to 20° B. at a temperature of about 60° C., air being injected at the bottom of the tank, or the use of compressed air may be avoided by loosely piling the scrap in a deep cylindrical vessel carried on lateral trunnions and closed on both sides by movable gratings. A pipe connected by a pump with the lye tank terminates over the cylinder in a wide rose which distributes the heated lye over the metal, while the liquor after percolating through the cylinder falls into a trough placed underneath, and is thence returned to the lye tank. The heat of the metal and lye produce a strong upward air current through the cylinder, and the intimate contact of the air, lye and metal promotes rapid oxidation and solution of the tin. The cylinder is occasionally reversed to ensure a uniform action. Large quantities of scrap may be treated in a stationary cylinder provided with a central longitudinal perforated tube closed at one end and connected with a fan at the other. The stanniferous lyes are preferably decomposed with carbonate of lime, the lyes being passed over until the deposit consists almost entirely of calcium stannate.—S. B. A. A.

*An Improved Process and Means for Producing the Generation of Chlorine Gas for the Chlorination of Gold or other Ores.* C. T. J. Vautin, London. Eng. Pat. 12,641, August 12, 1890. 4d.

THE chlorine is generated from a mixture of about equal weights of bleaching powder and perchloride of iron or manganese, together with about 5 per cent. of water. The reaction is represented by the equation—



The materials are charged into a vessel containing the ore to be acted upon, and the vessel is preferably closed during the generation of the gas. One of the advantages is the use of materials which may be transported with perfect safety.

—H. S. P.

*Improvements in Apparatus for the Extraction of Gold.* C. T. J. Vautin, London. Eng. Pat. 13,259, August 22, 1890. 6d.

THE author employs an air-tight combined dissolving and leaching barrel, carried on hollow trunnions and fitted in the interior with oblique baffles or part diaphragms for mixing the ore and reagents, and at one end with a filter bed so secured that the filtering medium is not displaced on rotating the barrel. The gold-bearing solution is either drawn through the filter bed by a diaphragm pump, or it is forced out by admitting water under pressure at the top of the leaching-barrel. The water supply and solution exit pipes pass through the trunnions. Drawings of the new barrel and pump and of a complete gold-extraction plant with a novel arrangement of depositing vessels, are attached to the specification.—S. B. A. A.

*The Manufacture and Production of a Dissolving and Reducing Agent, and the Employment of the same in Coating Metals with Metals or Combinations with Metalloids, Extracting Metals from their Ores or Combinations and in other Arts and Manufactures in which Dissolving or Reducing Action is required.* C. E. Bernard, Beaumont, France. Eng. Pat. 14,837, September 19, 1890. 6d.

THE "dissolving and reducing agent" is prepared by saturating glycerol with bromine at a temperature not

exceeding 80° C. and preferably about 60° C. It may be used for obtaining a metallic deposit upon a more readily oxidisable metal. Thus, for example, zinc may be coated with aluminium by first saturating the above compound at 100° C. with aluminium oxide added in small quantities at a time. The clear well-settled solution is then painted upon the perfectly clean zinc article or the latter may be immersed in the solution, when a brilliant white deposit is at once produced, which can be rubbed and polished with a leather. Or the article to be coated can be plunged into an electroplating vat containing the new compound with the further addition of a little sodium bisulphite and caustic soda when, upon passing an electric current, an immediate deposition of the metal is effected. Thus lithium, cobalt, "carbon," &c. can be thrown down. The compound is said to be also capable of "dissolving or reducing silica, spar, asbestos, tale, arsenic, phosphorus, cotton, wool, silk, cellulose, &c." —S. G. R.

*Improvements in Method and Apparatus for Producing Mineral Wool.* W. H. Kennedy, Etna, Penn., U.S.A. Eng. Pat. 14,846, September 19, 1890. 8d.

See under 1., page 1116.

## XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

### PATENTS.

*Improvements in the Manufacture of Electrodes or Plates for Electrical Batteries.* P. Schoop. Eng. Pat. 15,963, October 10, 1889. 6d.

THE plates are formed by the simultaneous electrolytic deposition of spongy lead, containing hydrogen, on one plate, and oxide of lead, or oxidised lead in combination with an alkali, on the other. The electrolyte used may be a lead salt with a suitable alkaline salt—e.g., 1 part of lead sulphate, 1 part of nitrate of ammonium, 10 parts of water—or an organic lead salt combined with an inorganic one, with or without an alkali. The organic salt may be the tartrate, sulphonate, &c., or that formed by adding glycerin to concentrated sulphuric acid, diluting and adding a lead base.—E. T.

*Improved Process of Purifying the Anhydrous Double Chloride Compounds of Aluminium.* H. Y. Castner, London. Eng. Pat. 18,062, November 12, 1889. 8d.

THE anhydrous compound, containing ferric and ferrous chlorides as impurities, is melted and caused to flow at a suitable rate through a series of baths containing electrodes. A considerable current is passed between the electrodes in the first bath, less in the second, and so on, the current being proportioned to the quantity of impurity still left. By this plan the iron alone is deposited on the cathodes without waste of aluminium.—E. T.

*Process for obtaining Chlorine and Bromine by the Aid of Electricity.* G. Nalmsen, Hanover, Germany. Eng. Pat. 11,699, July 26, 1890. 4d.

WHEN chlorine or bromine are obtained by electrolysis waste usually goes on by the reaction of these bodies on the water of the electrolyte. To prevent this the patentee cools the solutions below 0° C. by the aid of refrigerating machinery, taking precautions that the solutions shall be kept highly concentrated, current not below 0.25 amp. per sq.

decimetre, and the voltage 25 to 30 per cent. above that theoretically calculated. Chlorine so obtained is quite dry and free from hydrochloric acid.—E. T.

*Improvements in Galvanic Batteries.* H. J. Allison, London. Eng. Pat. 13,344, August 25, 1890. 8d.

A CHLORIDE of silver cell is made up, in which the case is of zinc, and forms one electrode. The chloride of silver is cast, with a corrugated or indented surface, round a silver wire to which are attached pieces of silver wire, foil, or gauze to make more intimate contact with the chloride. The electrolyte is rendered viscous by the addition of a suitable albuminous or gelatinous material. The silver wire projecting above the chloride of silver is covered with close-fitting india-rubber tubing. The cell is closed by Portland cement or other material which is porous to gas, but will not allow the viscous electrolyte to pass. Such cells are made very small and may even be put into a push or ordinary fitting.—E. T.

## XII.—FATS, OILS, AND SOAP MANUFACTURE.

*Tar Soaps and "Lysol," a New Disinfectant.* C. Engler. Dingl. Polyt. J. 278, 26—34 and 78—83; Pharm. Centralhalle, 1890, 449.

DISINFECTANTS which are prepared from soap and coal-tar oils may be divided into two classes: 1st, those that are solutions of soap in hydrocarbons; and 2nd, those that are solutions of hydrocarbons or phenols in soap. When petroleum oil (b.p. 150°–250°) is heated with a mixture of oleic acid and the oxides or hydrates of various metals, considerable quantities of soap are found to be dissolved in the oil, the maximum occurring in the case of lead, 15.5 per cent. of PbO being found in the liquid. To this first category belong most of the so-called "solidified oils," which are generally aluminium, magnesium, or other soaps dissolved in petroleum, and also the disinfectants known as "kreolin," "soluble phenyle," &c. All these solutions have the disadvantage of separating into two layers on the addition of water, only the soap going into the aqueous solution.

To avoid this loss the author has tried to prepare mixtures falling within the second class; the carbolic oils employed consisting (I. and II.) of about 82 per cent. of phenols, and (III.) of about 13 per cent. The alcohol was added to assist the saponification but was found to be unnecessary:—

	A.	B.	C.	D.	E.	F.
Linseed oil.....	30.8	30.8	30.8	..	..	..
Train oil.....	..	..	..	30.8	..	..
Rape oil.....	..	..	..	..	30.8	..
Rosin.....	..	..	..	..	..	33.1
Caustic potash*.....	18.4	18.4	18.4	18.4	18.4	30.5
Alcohol.....	20.0	20.0	20.0	20.0	20.0	23.2
Carbolic oil I.....	30.8	..	..	..	..	..
Carbolic oil II.....	..	30.8	..	30.8	30.8	13.2
Carbolic oil III.....	..	..	30.8	..	..	..

\* One part solid dissolved in two of water.

The solutions obtained were of a brown colour, and on dilution with distilled water gave perfectly clear liquids, C being slightly clouded owing to the impurity of the carbolic

oil employed. On distillation solutions A—E gave off an amount of oil practically identical with the quantity originally added, proving that the phenols exist in the free state. Solution F gave on distillation 17 per cent. of oil, the excess of 3·8 per cent. being probably due to decomposition of the rosin. To prove conclusively that the phenols were not combined, being liberated only on heating, cold solutions of potassium "carbolate" and "cresylate" were treated with oleic acid, when considerable heat was evolved, showing that chemical reaction was taking place.

"Lysol" is a new preparation of Schülke and Mayr's (Hamburg), which evidently is a similar substance. It exists in three forms known as No. II., No. III., and "Pure Lysol," and is a brown, transparent, syrupy liquid, which turns litmus blue, but contains no free alkali. The ash, which was found to be potassium carbonate, amounts to from 5·9 to 6·5 per cent. On distillation up to 225°, 47 to 51 per cent. of oil was obtained, containing about 95 per cent. of higher phenols (carbolic acid itself being absent).

Each of its preparations is miscible with water in all proportions, and hence forms a disinfectant which can be made of any desired strength, without the loss accruing from the insolubility of the active principle. It has also the advantage over phenols rendered soluble by the addition of strong sulphuric acid, in that it exists in a neutral solution, and hence might possibly be of great service against phylloxera, &c.

Schottelius (Münchener Medicinische Wochenschrift, 1890, No. 20) has examined the power that lysol possesses of destroying pathogenic organisms, the bacilli employed being those of cholera, typhus, and swine fever. In the case of the first two, the addition of ·33 per cent. of either lysol II. or III. destroyed in 20 minutes all bacilli, the power of the material over swine-fever being only slightly less. Lysol was next compared with ordinary phenol, and Pearson's kreolin (Jeyes') as to its power of instantly destroying all living germs. For this purpose *Staphylococcus py. aureus* and *Typhus bacillus* were chosen as having very different degrees of vitality, the minimum quantity being shown in the table:—

	Lysol.	Kreolin.	Phenol.
	Per Cent.	Per Cent.	Per Cent.
<i>Staphyloc. p. aureus</i> .....	·3	1·2	2·5
<i>Typhus</i> .....	1·2	..	..

—F. H. L.

*A New Fatty Acid.* E. Gérard. Compt. Rend. **111**, 305—307.

THE oil from the seeds of *Datura stramonium* (thorn apple) contains, according to the present article, a fatty acid which has hitherto remained undiscovered. The seed oil is extracted with ether; the extract is taken to dryness, and purified by dissolving in petroleum ether, filtering and distilling. A greenish-yellow oil is thus obtained. This oil, on saponification with litharge, yields lead soaps, from which the salts of oleic and linoleic acids are extracted by ether; the residual salts are decomposed with hydrochloric acid, and the resulting fatty acids are dried and dissolved in alcohol, from which, by cooling, a mass of crystals may be obtained. The crystals melt at 56°. Of these crystals a cold-saturated alcoholic solution is made and fractionally precipitated with barium acetate. The earlier products yield an acid melting at 55°, the later portions an acid melting at 62° (palmitic acid). The acid melting at 55° is an acid whose composition is stated to agree with the formula  $C_{17}H_{33}O_2$ . Further fractionation with barium acetate gives constant results and no further splitting up of the substance could be recognised. The author proposes the name "daturic acid."

Compounds claiming to have this formula have already previously been described; one by Becker in 1857, melting at 52°; another by Hantz soon afterwards, whose melting

point was put at 60°; and thirdly, one by Krafft in 1879, melting point 60°.

The formula claimed for the new acid places it between stearic and palmitic acids, yet its melting point is considerably lower than that of either of these acids.—T. L. B.

*Report on Soaps.* Report of the Central Laboratory of the Italian Customs Department, 1886—1889. 356—364.

THE tariff distinguishes between common, perfumed, and glycerin soaps, subject to duties of 8, 40 and 60 lire per quintal. In the first class are included every kind of hard soap, and soap containing silicious and earthy matters, as also pasty mixtures of fat, oil, soda and potash, together with cart-grease composed of oils of resin and fatty substances mixed with alkalis. In the second class are included soaps in tablets, &c. and soft soaps, which are not of the nature of so-called silk-soaps, while transparent or semi-transparent soaps, even though not perfumed, are relegated to the third class. Lastly, medicinal soaps such as those of opodeldoo, naphthalene, eucalyptus, iodine, &c., and soaps with lead oxide as their base, are classified as compounded drugs not specified. The Department determined the proportions of water, total alkali, free alkali, and fatty acid saponified, resin, glycerol, alcohol, medicinal substance, and essence or essential oil. Samples of common soap appear to have been declared as calcium chloride, various fats and vegetable fat.—V. H. V.

## PATENTS.

*Improvements in Extracting Oil or Grease.* W. H. Lever, J. D. Lever, and E. G. Scott, Port Sunlight. Eng. Pat. 18,988, November 26, 1889. 4d.

IN the ordinary process of extracting oil or grease from seeds or other materials by means of bisulphide of carbon, the inventors substitute for that solvent the tetrachloride of carbon (this Journal, 1890, 1063), which is said to be less dangerous to use and to leave the extracted oil in a purer condition.—G. H. B.

*Improvements in Soap Powders.* G. P. Clarke, London. Eng. Pat. 13,985, September 5, 1890. 4d.

A NEUTRAL salt of ammonium is incorporated with ordinary soap powder. In order to preserve the salt, preferably the sulphate of ammonium, from being decomposed by the sodium carbonate of the soap powder, the crystals are coated with finely-powdered sugar, and also, if necessary, with some fatty substance. It is only on dissolving the mixture in water that the neutral ammonium salt is decomposed, carbonate of ammonium being formed.

—G. H. B.

## XIII.—PAINTS, PIGMENTS, VARNISHES, AND RESINS.

*The Preparation of Ultramarine.* J. Wunder. Chem. Zeit. **14**, 1119—1123.

IN 1840 the manufacture of ultramarine was confined to three works, two in Germany and one in France. Since that date the number of colours has largely increased, including at present light and dark blue, green, red, violet, and white. For the production of ultramarine green and blue (from sodium sulphate), a mixture of kaolin, sodium sulphate, and coal is heated in crucibles to a bright red heat: sodium sulphide is produced which forms with the components of the kaolin a green mass. On treating this with sulphurous acid gas at a temperature of over 300° C. a blue of moderate depth results. Air-tight crucibles cause

the formation of Ritter's ultramarine white, a pale grey substance which on moderate heating is converted into a fine green. To produce a "pure" blue, a mixture of kaolin, rich in alumina, sodium carbonate and sulphur, in the proportion of equal equivalents of the latter two, is heated with rosin or asphaltum; the lower portion of the contents of the crucible become dull green, the upper being blue, the colour however improving by moderate heating. To obtain a uniformly dark blue mass, silica is mixed with the kaolin, and a larger proportion of sulphur employed than in the last case. This may be burnt either in crucibles or in bulk, the latter method giving a product richer in colour, and one that better withstands the action of alum; but which owing to its containing free sulphur is unavailable for some purposes such as calico printing, the hot rollers employed being liable to injury. This free sulphur can be removed neither by heat nor by boiling with caustic alkalis without injury to the colour; but by boiling the ultramarine in a solution of sodium sulphide by the aid of steam in zinc vessels, the author has succeeded, the colour being often considerably improved by the process.

Chemically pure sodium carbonate and "ammonia soda" make the mass difficult to burn, but on the other hand, a slight addition of caustic alkali or of sodium sulphide is of assistance in the process. The richer the mixture is in silica the more difficult is the burning, but the deeper is the colour of the ultramarine, and the greater its power of resisting the action of alum and weak acids. Oxygen must be carefully excluded during the combustion, as otherwise silica is reformed, which yields with the sodium and aluminium present a hard slag. To avoid this many manufacturers run their furnaces with an insufficient supply of air; this, however, considerably increases the amount of fuel used, beside producing large quantities of smoke. By burning the coal in a separate chamber, and allowing only the flame and hot air to circulate round and through the furnace in which the crucibles or substance in bulk is placed, the author has been able to work with considerably less coal, without producing more than a trace of smoke, and without any deleterious action on the mixture. A further economy is gained by allowing the gases to circulate through another furnace before escaping, which can thereby be raised to a dull red heat. Samples should be taken at intervals during the burning, by means of an iron rod with a spoon-shaped end; these must be kept covered till cold, and when they are found to be dark green the operation is complete.

Ultramarine containing much silica is usually of a reddish-blue colour. To obtain a pure blue, the author heats the mixture in covered crucibles. The green product is powdered and heated in a limited supply of air to a temperature of  $160^{\circ}$ – $180^{\circ}$  and a small quantity of steam; colours varying from greenish-blue to pure blue being obtainable at pleasure. By this method a good blue was formed from a mixture containing 69.32 parts of silica to 50.67 of kaolin, the highest proportion previously available being 66.7 of silica to 33.3 of kaolin.

The crude ultramarine blue contains from 20 to 24 per cent. of free sodium sulphate, which is usually removed after powdering by repeated agitation with hot water. The author recommends that the blue should be mixed with the water to a paste, boiled with steam to dissolve the sulphate, and the mass thrown on to a filter on Bunsen's principle, where the solution is removed under diminished pressure. The filter recommended has a diameter of 1 m. and a depth of 5 m.; 8 cm. from the bottom a perforated tray is fixed, on which a filter cloth is laid, and a manometer to show the pressure. Over the ultramarine a thin sheet of metal provided with small holes is laid, to avoid disturbing the substance during washing. If it be desired to work up the sulphate solution, a small quantity of carbonate should be added to precipitate the dissolved iron.

The final grinding takes place with water under edge-runners, the weight of the runners being supported on a footstep bearing which is adjustable by levers and a counter-balance, so that the pressure on the bed can be altered to suit the fineness of the material. It is then washed and levigated, the finest particles being precipitated by the addition of some salt which has no action on it, e.g., ammonium salts, calcium sulphate, or even hard water.

**Ultramarine violet.**—The first samples of this colour were made under the direction of Leykauf by the ultramarine works at Nuremberg in the year 1859. The process employed was to allow moist calcium chloride to act on ultramarine blue in a warm place. In the year 1872 the author treated ultramarine with chlorine gas at a temperature of  $300^{\circ}$  C.; the mass assumed a reddish-brown colour, which on treatment with water became violet. This colour contained water in combination, and at a high temperature was decomposed into the original blue. A similar brown substance is produced by the action of sulphuric anhydride or sulphur chloride on heated ultramarine. When the chlorine compound is treated with absolute alcohol a reddish-violet substance is obtained containing some organic matter in combination, a smell of ethyl chloride being produced. By leading ammonia gas over the heated chlorine compound, it is absorbed, and a violet colour produced, aniline acting in a similar manner. The violet is also formed by passing a mixture of chlorine and steam over ordinary ultramarine heated to  $160^{\circ}$ – $200^{\circ}$  C.

Mahla heated a mixture of ammonium nitrate and chloride (which decompose in the following manner,  $\text{NH}_4\text{Cl} + 2 \text{NH}_4\text{NO}_3 = 6 \text{H}_2\text{O} + 5 \text{N} + \text{Cl}$ ) and ultramarine to  $200^{\circ}$  C. and directly obtained a fine violet. This compound contains ammonia in combination. In the course of manufacture it has been found that by working in porous crucibles and exposing the mass to a long-continued heat, the addition of nitrate to the mixture is unnecessary. A mixture of hydrochloric acid gas and air has been substituted for the chlorine with success, care being taken to keep the temperature between  $180^{\circ}$  and  $230^{\circ}$ .

The violet produced by the action of moist chlorine differs from the original blue, by having lost one-sixth of its sodium and having gained water and oxygen; the approximate formula for the blue being,  $\text{Na}_6\text{Al}_7\text{Si}_6\text{S}_4\text{O}_{21}$ , and for the violet,  $\text{Na}_5\text{HAl}_7\text{Si}_6\text{S}_4\text{O}_{21} + \text{H}_2\text{O}$ . It also contains much thiosulphate. When treated with iodine solution in excess, decomposed by hydrochloric acid, and the sulphates removed by barium chloride, the tetrathionic acid which is formed by the action of the iodine on the thiosulphate may be oxidised by nitric acid and precipitated as barium sulphate, the yield from violet (calculated as free sulphur) being 2.3 per cent., the blue giving only 0.29 per cent.

For the manufacture of the violet by the action of chlorine and steam, the blue is first spread out in thin layers on earthenware trays, heated in a furnace to  $280^{\circ}$  C., and steam passed over it to remove the sulphur. It is then allowed to cool to  $160^{\circ}$  when a mixture of chlorine and steam is led over it for three hours. Iron, not being attacked by chlorine between  $150^{\circ}$  and  $250^{\circ}$ , may be used for the material of the furnace. Hydrochloric acid may be substituted for the chlorine, the temperature employed being  $220^{\circ}$ – $230^{\circ}$ ; the operation lasts about seven hours, and by allowing the heat gradually to fall to  $160^{\circ}$  the colour of the product becomes very brilliant. For the production of the violet by means of sal-ammoniac with or without the addition of nitrates, the blue is heated with 5 per cent. of ammonium chloride for 14 days in a chamber built over the blue furnaces; when sodium nitrate is added the operation is completed in less time.

Ultramarine violet heated to  $280^{\circ}$  in a stream of hydrogen is converted into a new colouring matter, a light blue. On analysis this substance gives figures closely agreeing with the formula  $\text{Na}_5\text{H}_3\text{Al}_7\text{Si}_6\text{S}_4\text{O}_{25}$ .

Heated to from  $135^{\circ}$ – $145^{\circ}$  in a stream of nitric or hydrochloric acid gas, the violet is converted into ultramarine red, which substance is apparently an acid, whose sodium salt is the violet. Analysis of the red gives  $\text{Na}_3\text{H}_5\text{Al}_7\text{Si}_6\text{S}_4\text{O}_{25}$ .

—F. H. L.

#### ERRATUM.

This Journal, November number, 1890, p. 1046, col. 2, last two paragraphs, for "Oil of Flax," read "Linseed Oil." Also p. 1045, *ibid*, for "Flax" read "Linseed."

## XIV.—TANNING, LEATHER, GLUE, AND SIZE.

### PATENTS.

*Improvements in the Method of and Apparatus for Extracting the Tannin from Bark or other Materials used for Tanning Purposes.* J. Hutchings and W. N. Hutchings, Warrington. Eng. Pat. 20,403, December 18, 1889. *Sd.*

In the centre of an ordinary pit or leach of any convenient size is fixed a square wooden trunk having openings at the bottom. Enclosed by this is a plunger which is a strong wooden box fitting the trunk and about three feet deep. This plunger can be weighted in any convenient way, such as by partially filling with stones, &c., and has a simple pump clack at the bottom. The plunger is connected by a rope with the end of a rocking-beam, by the oscillation of which it is alternately raised and lowered. The mode of working is as follows:—Baskets, half filled with bark, valonia, or other tanning material, are placed in the leach surrounding the trunk till the leach is filled, and water or liquor is then run into the leach. By the movement of the plunger the liquor is kept in constant movement and the soluble constituents of the tanning material are rapidly exhausted. Drawings are given.—B. H.

*Improvements in Machines for Colouring Leather, Skins and like Materials.* C. Knabe, Osterwieck-am-Ilarz, Germany. Eng. Pat. 15,167, September 25, 1890. *6d.*

"The invention consists in the combination of a rotary table on which the material to be coloured is extended, with a colouring drum which is moved to and fro over the table whereby it is at the same time set in rotary motion alternately in the one and in the other direction." Details and drawings of the machine are given in the specification.

—B. H.

## XV.—MANURES, Etc.

*Field Experiments with Swedes.* E. Kinch. Report of the Royal Agricultural College Club, Cirencester, 1889.

In these experiments the same seed, Sutton's "Crimson King Swede," and the same manures were employed during the same season on various soils:—1. Stiff brown clay, on trias, Peplow, Shropshire. 2. Light "hungry" sand on New Red Sandstone, Cheswardine, Staffordshire. 3. Sandy soil, Elstree, Herts. 4. Light soil, St. Alban's, Herts. 5. Rather stoney marl, on Forest Marble, Siddington, Gloucestershire. 6. Red loam on Carboniferous Limestone, Bristol, Somerset. 7. Loam on greensand and gravel, Burecote, Oxon. 8. Calcareous clay, on Great Oolite, Royal Agricultural College, Cirencester. The manures consisted of superphosphate with 26.81 per cent. soluble phosphate, nitrate of soda containing 98.41 per cent. of the pure salt, and Thomas' slag with 17.8 per cent. phosphoric acid, and 85 per cent. passing through a 100-mesh, and over 70 per cent. through a 150-mesh sieve. The following weights of roots in tons and cwt. per acre were obtained under the conditions specified:—

### WITHOUT OTHER MANURE.

Soil.	Thomas' Slag.		Super-phosphate 3 Cwt.	No Phosphate.
	1 Cwt.	6 Cwt.		
	Tons Cwt.	Tons Cwt.	Tons Cwt.	Tons Cwt.
1. Stiff clay .....	11 7	8 17½	9 14½	8 14
2. Light sand .....	19 19	21 8½	18 6½	20 1½
3. Sand .....	15 16½	14 18½	15 7½	11 18½
4. Light soil .....	11 17½	13 4½	13 6½	8 8½
5. Marl .....	14 8½	14 11½	14 3½	13 6
6. Red loam .....	1 16½	15 4½	15 11	6 9½
7. Loam .....	20 7	16 8	20 1½	19 5½
8. Calcareous clay..	12 6½	19 8½	13 4½	11 8½

### WITH FARMYARD MANURE.

1. (15 tons) .....	11 18	14 18	15 8	13 1½
2. (15 tons) .....	33 7½	29 19½	33 7½	20 18
3. (18 tons) .....	15 19½	16 19½	21 8½	17 2
4. (12 tons) .....	14 17½	15 19½	17 17½	17 1½
5. (12 tons) .....	18 1½	17 12½	20 0	17 8
6. (18 tons) .....	22 18	19 11½	22 19	20 15½
7. (12 tons) .....	23 14½	20 15½	23 16½	21 5
8. (12 tons) .....	15 10½	14 12	15 19½	14 0½

### WITH NITRATE OF SODA, 1 CWT. PER ACRE.

1. ....	8 11½	8 13½	16 12½	7 18½
2. ....	22 0	2 5½	21 11½	21 1
3. ....	16 1½	16 1½	17 3½	15 17½
4. ....	11 7½	Diseased.	14 1½	Diseased.
5. ....	17 5½	16 19½	18 5½	16 12
6. ....	14 2½	13 8	13 11½	6 9½
7. ....	21 3½	21 10½	19 5½	21 15
8. ....	16 17½	16 17½	19 11	17 15½

From which it will be seen that the superphosphate alone gave an increase in nearly all cases, and generally acted well in conjunction with farmyard manure and in fewer cases with nitrate. Slag alone also gave a good increase in most instances, and in some cases equal to the superphosphate, but it is more uncertain in action than the latter, especially when used with nitrate or farmyard manure. The farmyard manure exhibited a good effect in all cases and the nitrate in most. Soils 2 and 7 were in too good condition to benefit much by the manures.—D. A. L.

*Value of Superphosphate as compared with a Mixture of Ferrons Sulphate and Mineral Phosphate.* P. M. De-lacharlonny. Jour. de l'agricult. 1890, 1, 825—829.

Plots were first dressed with farmyard manure and then with different manurial mixtures:—superphosphate and sodium nitrate with and without potassium sulphate and iron nitrate; superphosphate, potassium sulphate, and iron sulphate; mineral phosphate and iron and potassium sulphates with and without sodium nitrate and potassium nitrate. Vine was cultivated and the wine harvest estimated. The results indicated that not alone could the superphosphate be profitably replaced but even surpassed by the use of the ferrons sulphate and mineral phosphate together.

—D. A. L.



*Behaviour of Sandy Soils towards Superphosphate.* A. Thomson. *Bied. Centr.* **19**, 585—588.

PERCOLATION experiments, made to ascertain the factors influencing the absorption and retention of phosphoric acid by sandy soils, have led to the following conclusions. Phosphoric acid is absorbed and retained most energetically when ferrie and aluminium hydroxides and calcium carbonate are present simultaneously, but separately the hydroxides are more active as absorbents than the calcium carbonate; whilst sand and orthoclase neither absorb nor retain phosphoric acid. Sodium chloride solutions, as compared with distilled water, dissolve less phosphoric acid from superphosphate. They diminish the absorption of phosphoric acid by calcium carbonate (a 1 per cent. solution more than a 2 per cent.), whilst the absorptive power of the hydroxides is augmented by a 1 per cent. common salt solution and is depressed by a 2 per cent. solution. The absorption by the combined absorbents is reduced by both salt solutions to about the same extent. Potassium nitrate behaves in a similar manner to the sodium chloride, but more energetically, and the 2 per cent. solution is in most cases more active than the 1 per cent. solution. Distribution exerts an important influence on the absorption of phosphoric acid, but no simple relationship was noticed between the quantity of phosphoric acid dissolved and the duration of the contact of the solvent with the material. Agriculturally considered superphosphate can only be expected to act as a fertiliser on sandy soil when a large quantity of calcium carbonate or a smaller quantity of both the calcium carbonate and the hydroxides are present, and when there is not too much nitrate either applied to or formed in the soil.—D. A. L.

*Action of Manures containing Thiocyanates (Sulphocyanides).* E. Maek and K. Silen. *Tyroler landw. Blätter*, 1890, **8**, 198—200.

DIMINISHED yields of maize, manured with ammonium sulphate containing ammonium thiocyanate (sulphocyanide), and cut and weighed green, indicate a very great sensitiveness in these plants towards thiocyanates; which became evident even from the generally deteriorated appearance of the plants when the quantity of thiocyanate amounted to 9.0 kilos. per hectare.—D. A. L.

*The Exhaustion of Unmanured Soil.* P. P. Dehérain. *Compt. Rend.* **111**, 253—250.

IN a previous paper (*Compt. Rend.* **109**, 781) an account has been given by the author of experiments made upon land that has not been manured since 1875. He finds that this unmanured soil can yield a good crop of oats and a fair crop of wheat, but that the culture both of beet and of clover is impossible. This ground differs little from well-manured ground in respect to the quantities of nitrogen, of phosphoric acid, and of potash it contains, but it has only half of the amount of carbon derived from organic matter, or 7 grms. per kilo. of soil. There is, further, little difference in the amount of moisture retained by the manured and unmanured soil. To throw further light on the subject the author has examined the drainage water from numerous samples of soils, for it appears from the above that it is organic matter that renders the soil suitable for the growth of beet and clover.

The method of observation adopted was to place the soil in large pots and to collect the drain water by a suitable pot placed below. Manured, unmanured, and meadow soil were examined in this way. There was found to be little variation in the amount of drain water obtained in the three cases, nor do the different quantities of nitric acid found in the drain waters appear sufficient to account for the sterility of the unmanured soil. During the year 1889—90 the soils experimented upon lost 100 grms. of nitric acid, corresponding to 600 grms. of sodium nitrate per hectare, in the drain water. This loss had taken place on soil without any crop on it in the case of the unmanured land, whilst with the manured land the author's experiments go to show that it occurs mainly during the periods between the growth of

crops. To avoid this loss of nitrates it is recommended to sow some rapidly-growing plant, such as rape, immediately after gathering the crop of beet, clover, &c., so that something shall be in the soil which is capable of transforming the nitrates into organic matter. When these plants are ploughed into the soil at the end of autumn or the beginning of spring they restore to the soil the whole of the nitrogen which would otherwise have been lost, and in addition a powerful organic manure which is particularly efficacious for certain kinds of crops.—C. A. K.

*The Treatment of Black-Rot.* A. de l'Écluse. *Compt. Rend.* **111**, 284—285.

IF the leaves of the vine are covered with a composition containing a soluble copper salt or oxide of copper, the black-rot loses the power of germinating and the vine is preserved. This treatment should be constantly applied to all the green parts of the plant at least 12 days before the first attacks of the black-rot are to be expected.

If, however, the leaves are already covered with the pest, the grapes may be still preserved, as long as the peduncle has not been affected, if the composition be applied 15 days before the appearance of any contaminated fruit. In order to ensure complete success by this means the whole of the green surfaces of the vine must be completely covered by the salts of copper during the periods mentioned. It is not sufficient to coat only the upper surfaces of the leaves.—C. A. K.

#### PATENTS.

*Improvements in the Treatment of Sewage and Sewage Matters, and in the Production of Fertilising Products therefrom.* W. B. Kemshead, Mitcham. Eng. Pat. 18,677, November 21, 1889. *4d.*

*See under XVIII. B., page 1144.*

*Improvements in the Treatment of Organic Matters for the Production of Manures and in Apparatus therefor.* J. Guillaume, Paris, France. Eng. Pat. 5419, April 9, 1890. *11d.*

*See under XVIII. B., page 1144.*

### XVI.—SUGAR, STARCH, GUM, Etc.

*Studies on Starch.* C. Scheibler and H. Mittelmeier. *Ber.* **23**, 3060—3075.

THIS paper is divided into three parts, in the first and second of which the chemistry of starch is discussed from the historical and theoretical standpoints. For the considerations here involved the original communication must be consulted. In the third portion of their paper the authors describe the results of their investigations on the nature of dextrans. Pure commercial dextrin was used in the experiments. It contained appreciable quantities of glucose indicated by the phenylhydrazine reaction. It was found that the dextrin could be readily purified by repeated precipitation with alcohol, or by osmosis. The sugar-free dextrin thus obtained consisted no doubt of a mixture of several dextrans. The aldehydic character of the dextrin was shown by its behaviour towards various reagents. Its solution was browned by alkalis and distinctly reduced Fehling's solution. The solid dextrin was slowly dissolved in the cold by phenylhydrazine, and this solution yielded on precipitation with alcohol a brilliant white powder, consisting of the *hydrate of dextrin*. This substance is hydrolysed by diastase. It is very soluble in water and



contains 1.02 per cent. of nitrogen. This number gives one a rough idea of the molecular weight of dextrin, as will be seen by comparing it with the percentages of nitrogen contained in different dextrin phenylhydrazones:—

Formula.	Per Cent. N.
$C_{64}H_{112}O_{70}N_2HC_6H_5$ .....	1.18
$C_{96}H_{162}O_{40}N_2HC_6H_5$ .....	1.04
$C_{108}H_{182}O_{90}N_2HC_6H_5$ .....	0.93
$C_{120}H_{202}O_{100}N_2HC_6H_5$ .....	0.84

An osazone was also prepared containing 1.63 per cent. of nitrogen. It forms a yellow powder and is slightly soluble in water.

A cold solution of dextrin treated with sodium amalgam loses its property of reducing Fehling's solution, and is no longer browned by alkalis. The reduction product may be precipitated by means of alcohol, and is obtained as a white powder, which does not dissolve in phenylhydrazine even on warming. The authors propose to name this new body "*dextrite*." Dextrite is saccharified by diastase and by strong acids. By the gradual oxidation of dextrin with bromine, the corresponding carboxylic acid is obtained. It forms a white powder, the solution of which reddens litmus paper and decomposes calcium carbonate on boiling. The solid substance is soluble in phenylhydrazine. The solution of the acid is not precipitated by basic acetate of lead, or by lime water. It does not reduce Fehling's solution, but after boiling with acids, or after treatment with diastase, it acquires a strong reducing action. The comparative ease with which this acid is produced is a further proof of the aldehydic nature of dextrin. Dextrin has been treated with oxidising agents by other observers, but only with the object of destroying sugars, to an admixture with which the reducing power of dextrin was ascribed, the assumption being made that the dextrin itself would not be affected by the reagent employed, such as, for instance, mercuric cyanide. According to the present research it appears that the "non-reducing" dextrin obtained by such a process consists in all probability of the carboxylic acid of dextrin.

The question, whether or not pure dextrin reduces Fehling's solution, is thus answered in the affirmative.

—H. T. P.

*On Sugars richer in Carbon from Rhamnose.* E. Fischer and O. Piloty. Ber. 23, 3102—3110.

RHAMNOSE is a methyl pentose, the formula of the anhydrous sugar being  $CH_3(CH.OH)_4.CO.H$ , and when treated in the way which has been previously described (this Journal, 1890, 638), it yields sugars containing one or several atoms of carbon more. These sugars the authors propose to call rhamno-hexose, rhamno-heptose, rhamno-octose, &c.

*Rhammitol*,  $CH_3(CH.OH)_4.CH_2OH$ , is the pentahydric alcohol obtained by the reduction of rhamnose by sodium amalgam. In order to obtain a crystallisable product, a 10 per cent. solution of rhamnose is shaken with  $2\frac{1}{2}$  per cent. sodium amalgam, and kept cool. During the first half of the reaction the solution is kept just acid with sulphuric acid, and during the latter half just alkaline.

The reduced alkaline solution is filtered, neutralised with sulphuric acid, evaporated on the water-bath, and the syrup taken up with hot absolute alcohol. On evaporation, the alcoholic solution yields a syrup, which crystallises on cooling. The yield is about 60 per cent. on the sugar taken. The recrystallised rhammitol tastes sweet, melting point  $121^\circ C.$ , and distils partly undecomposed. It is easily soluble in water and alcohol, with difficulty in chloroform and acetone, and is almost insoluble in ether. A concentrated solution in acetone deposits fine globules, which crystallise, and grow to fairly large triclinic prisms. A solution of rhammitol is optically active  $[\alpha]_D = +10.7^\circ$ ; it does not reduce Fehling's solution.

*Rhamno-hexonic Acid*,  $CH_3(CH.OH)_5.CO.OH$ .—This has previously been described as iso-dulcitol-carboxylic acid (this Journal, 1888, 636). An aqueous solution is dextro-rotatory,  $[\alpha]_D = +83.8$ .

*Rhamno-hexose*,  $CH_3(CH.OH)_5.CO.H$ , is obtained by reducing a solution of the lactone of rhamno-hexonic acid by  $2\frac{1}{2}$  per cent. sodium amalgam. The solution must be kept cool and acid by the addition of dilute sulphuric acid as the reaction proceeds. The reduced solution is filtered, and sufficient soda added to give an alkaline reaction after standing a quarter of an hour. The solution is then just neutralised with sulphuric acid, evaporated to a syrup, and the chief portion of sodium sulphate removed by crystallisation, and the mother-liquor taken up by boiling absolute alcohol. The sodium salt is precipitated, and the sugar remains in solution, which crystallises from the syrup obtained by evaporating the alcoholic solution. The yield is 60—65 per cent. of the lactose taken. It crystallises in well-formed short columns on thick tables, melting point  $180^\circ$ — $181^\circ$ , without decomposition. It differs from rhamnose in crystallising in the anhydrous state. It is difficultly soluble in absolute alcohol, but more easily in hot methyl alcohol. Its aqueous solution tastes sweet, and gives all the ordinary sugar reactions, and does not ferment with beer yeast. It is levo-rotatory, having  $[\alpha]_D = -61.1$ .

*Rhamno-hexosazone*,  $C_7H_{13}O_7(N_2H.C_6H_5)_2$ , is obtained in the usual way, in fine yellow needles, melting point  $200^\circ$  with decomposition. It resembles rhamnosazone in being easily soluble in hot alcohol, but almost insoluble in water.

*Rhamno-hexitol*,  $CH_3(CH.OH)_5.CH_2OH$ , is prepared from rhamno-hexose, just as rhammitol is from rhamnose. It crystallises from hot alcohol in small colourless prisms, melting point  $173^\circ$ ; it does not reduce Fehling's solution, and is dextro-rotatory,  $[\alpha]_D = +11.6$ .

*Rhamno-heptonic Acid*,  $CH_3(CH.OH)_6.CO.OH$ . By acting on rhamno-hexose with hydrocyanic acid in aqueous solution, the amide of rhamno-heptonic acid separates in two days. The mixture is then heated on the water-bath until all the hydrocyanic acid is expelled, and then a solution of baryta added and evaporated on the water-bath until all smell of ammonia disappears. All baryta is then removed by sulphuric acid, and the solution evaporated to a syrup, from which the lactone crystallises, and which is purified by recrystallisation from alcohol. The yield is 65 per cent. on the sugar taken. The lactone melts  $160^\circ$ ; it is very easily soluble in water, fairly soluble in ethyl and methyl alcohol, and insoluble in ether. Its aqueous solutions are dextro-rotatory,  $[\alpha]_D = +55.6$ .

*Rhamno-heptonic Acid hydrazide*,  $C_8H_{15}O_7.N_2H_2.C_6H_5$ , forms fine white needles; it becomes coloured when quickly heated to  $215^\circ$ , and melts a few degrees higher. It is fairly easily soluble in hot water, and almost insoluble in cold water and alcohol.

*Rhamno-heptose*,  $CH_3(CH.OH)_6.CO.H$ , is obtained by reducing the lactone of rhamno-heptonic acid. It forms a colourless syrup, easily soluble in alcohol and water, but not in ether. It has not been crystallised. To obtain it free from ash, it is converted into the hydrazone, which is with difficulty soluble in water, and may be easily purified and the sugar re-obtained from it. Aqueous solutions of the sugar are dextro-rotatory,  $[\alpha]_D = +8.4$ . The hydrazone has a composition corresponding to the formula  $C_8H_{16}O_6.N_2H.C_6H_5$ , melting point  $200^\circ$ , with decomposition. The sugar also yields an osazone,  $C_8H_{14}O_5.(N_2H.C_6H_5)_2$ , which crystallises in fine yellow needles, melting point  $200^\circ$  with decomposition. It is only slightly soluble in hot alcohol.

*Rhamnose-octonic Acid*,  $CH_3(CH.OH)_7.CO.OH$ , is produced by the action of hydrocyanic acid on rhamno-heptose in a similar manner to the production of rhamno-heptonic acid from rhamno-hexose. The lactone is purified by recrystallisation from acetone. It melts at  $171^\circ$ — $172^\circ$ , and is easily soluble in water and alcohol. Its aqueous solutions are levo-rotatory  $[\alpha]_D = -51.2$ . It yields a phenylhydrazide  $CH_3(CH.OH)_7.CO.N_2H_2.C_6H_5$ , with difficulty soluble in hot water, melting point  $220^\circ$  with decomposition. It crystallises in fine white needles.

A sugar is obtained by the reduction of rhamnose-oetonic acid which is doubtless rhamno-oetose. The authors had not sufficient material to properly determine its characteristics, but they prepared its osazone, melting point  $216^{\circ}$ , and found that the sugar reduced Fehling's solution.

—A. L. S.

*Improvements in Sugar Refining.* E. O. Lippmann. Chem. Zeit. 14, 1405—1406 and 1425—1426.

LAST year the author reported on Steffen's and Seyferth's sugar-refining processes (see this Journal, 1889, 993), and at the time expressed his opinion that their practical merits would most likely be decided during the following season. As regards Steffen's method it has shown itself to be of great value, and the adoption of a considerable number of deep troughs, permitting the washing of thicker layers of sugar, has been found practicable. In the case of raw sugars it is of importance to choose uniform, well-grained, and not too moist samples, since such specimens are most readily washed. A period of 8—10 hours was at first supposed to be sufficient for washing; but later experience shows that 12—15 or even 16 hours are necessary. All kinds of irregularly-grained, moist, and pasty samples containing invert-sugar are unsuitable for purification by this process. The washing of charges ("Füllmassen") presents no difficulties, provided they are properly boiled and sufficient time be given. Steffen's process is particularly adapted to the direct production of white sugar; the raw sugar treated must, however, be of fairly good quality. As regards the yield of sugar obtainable by Steffen's process, it is stated that a raw sugar containing 88—92 per cent. of cane sugar gives about the same absolute quantity of crystal sugar polarising  $99.5-99.9$ . As regards the quantity of pure syrup required to wash a raw sugar, it depends on the amount of molasses adhering to the latter. The pure syrup finally left in contact with the washed mass may be removed by centrifuging, and used over again. This fact has led some observers to state that there is no consumption of pure syrup at all; in other words, that an unlimited amount of raw sugar may be washed with a given quantity of syrup. In practice, however, it is impossible to prevent the pure syrup from mixing to some extent with the molasses it is intended to displace. The consumption of pure syrup is variously estimated at from 2 to 5 per cent. The cost of working Steffen's process is slight. The washing of 2—3,000 centners of sugar daily requires an engine of 55—60 H.P.; whilst the wages to be paid amount to about 10 pf. per centner. On the other hand the cost of plant, and the royalty to be paid to the inventor, are extraordinarily high.

Seyferth's paraffin method is in every respect a complete failure. It has, however, given rise to the Waghäuser refining process, which appears to be a combined centrifuging and washing method. Ten centrifugal machines, taking a charge of 150 kilos. each, are said to refine 40,000 kilos. of raw sugar in 20 hours, six workmen being required.

Foelsche employs a battery of centrifugal machines, fitted with non-perforated drums, the syrup being skimmed off as in milk centrifugals. The raw sugar is treated in the centrifugals with syrup or steam or both. The results obtained by this process are said to be excellent. Drost and Schulz's method aims at the direct production of crystallised cane from beet-sugar mass. The beet-sugar mass freed from green syrup by centrifuging is washed with 8—10 per cent. of filtered thick beet-juice at  $66^{\circ}-67^{\circ}$  B. Finally, steam is allowed to act on the crystals for several minutes. 2—3 per cent. of the solid sugar are dissolved by this means, and the syrup produced, as well as the thick juice used for washing, are boiled down for sugar. The operation requires 30 minutes for its completion. A raw sugar containing 86.1 per cent. of cane (by polarisation) and 7 per cent. of water was treated with thick juice having a purity coefficient of 91.5; 63—64 per cent. of crystallised sugar, polarising  $99.7$ , and green syrup of 71 purity coefficient were obtained finally.

—H. T. P.

*Gum from Beetroot.* Deutsche Zuckerind. 15, 1128.

EICHELMANN has succeeded by heating together at a temperature of  $120^{\circ}-200^{\circ}$  dried shreds of beet with 20 per cent. of caustic lime, and removing the excess of lime from the aqueous solution by carbonic acid, and the products of the decomposition of the albuminoids by alcohol, in producing a gum, which, in its adhesive properties, is hardly inferior to gum arabic.—F. H. L.

## XVII.—BREWING, WINES, SPIRITS, Etc.

*Recent Progress in Brewing.* Dingl. Polyt. J. 277, 133—135.

CRISMER (Weinlaube, 1889, 210) recommends saffranine as a reagent for dextrose, used as follows:—2 to 3 cc. of saffranine solution of 1 per cent. strength are heated with 2 to 3 cc. of caustic soda solution of 10 per cent. The colouration disappears at  $60^{\circ}$  or  $65^{\circ}$  C., if some drops of dextrose solution be added. After some time, red lines appear on the surface of the liquid, where the saffranine reappears by oxidation.

J. Geyer confirms (Pharm. Zeit. 1889, 683) the experiment made by Thierfelder, viz., that potassium glucuronate ( $C_6H_7KO_9$ ) forms a compound with phenylhydrazine very similar to the one sugar producees. Therefore this reaction is not always reliable.

E. Preuss (Zeits. f. Rübenzuckerind. 1890, 18) recommends the following preparation of Soldani's reagent for the determination of invert sugar:—15.9 grms. of copper sulphate are added to a hot solution of 594 grms. of potassium bicarbonate, and, when cold, diluted to 2,000 cc. This solution, which does not easily decompose, has a specific gravity of 1.1789. The test is made as follows:—150 cc. of this reagent are heated to boiling, and after the invert sugar solution is added, the boiling is continued for 10 minutes. The following quantities of copper and invert sugar are equivalent:—

Copper.	Invert Sugar.	Copper.	Invert Sugar.
Mgrms.	Mgrms.	Mgrms.	Mgrms.
18.8	5	142.4	45
34.9	10	157.0	50
50.9	15	171.3	55
66.6	20	185.5	60
82.2	25	200.4	65
97.6	30	213.1	70
112.7	35	226.6	75
127.8	40	240.6	80

(Compare this Journal, 1890, 825.)

HERZFELD (Zeits. f. Zuckerind. 1890, 52), in criticising the general application of Soldani's reagent, points out that, compared with Fehling's reagent, it contains only the fifth part of copper, and may therefore be less sensitive; also that its power of dissolving lime is less; that it precipitates cupric oxide when highly diluted, and more cuprous oxide when strongly boiled.

*Application of Electrolysis in Sugar Determination.*—As caustic potash has sometimes an injurious effect upon asbestos, Formanek (Böhm Zeits. f. Zuckerind. 1890, 178) recal dextrins. The aldehyde & the cuprous oxide on pal shown by its behaviour toward tartaric acid, &c. This has been done long ago by Märcker, but has been left as inaccurate. Asbestos, which shows the above-named deficiency, should first be boiled with caustic potash and nitric acid.—H. S.

*On the Furfural-yielding Bodies from Wheat and Rye Chaff.* E. Steiger and E. Schulze. Ber. **23**, 3110—3113.

It has been known for some time that furfural is produced by distilling wheat and rye chaff with sulphuric acid. It is also known that when arabinose or xylose, or those bodies which yield one of these sugars on hydrolysis, are distilled with sulphuric acid in the same way, they also yield furfural. Although Stone and Tollens (Ann. **249**, 239) obtained an osazone from the products obtained by digesting wheat or rye chaff with sulphuric acid, which osazone possessed all the properties of arabinosazone, yet they were unable to isolate arabinose from these solutions.

The authors have re-investigated these products, and have succeeded in crystallising arabinose from them, which they have identified by its crystalline form, optical activity, &c. They consider that the body which yields arabinose on hydrolysis is a constituent of the cell membrane. Owing to the difficulty of separating it from the cellulose, it is impossible to determine its properties with any degree of completeness. It is insoluble in water, in very dilute alkali, in very dilute mineral acids, in solutions of diastase and digestive ferments.

The mother-liquors left after the crystallisation of arabinose have a considerably lower optical activity than arabinose; on account of this the authors suspect the presence of xylose.—A. L. S.

#### *The Analysis of Malt.* Chem. Zeit. **14**, 1368.

THE agricultural section of the International Agriculture and Forestry Congress at Vienna discussed a uniform system for the analysis of malt. The following points were agreed upon:—

1. *Sampling.*—The sample must represent a true average sample. This is best done by well mixing the malt-heap by a shovel, taking samples from different parts of the heap, well mixing these, and from this taking the sample for analysis. If this cannot be done, a sampling stick may be used. Under no circumstances should the sample be taken from the upper part of the malt-heap. The samples must be stored in tightly closed bottles or tin canisters. When sending it for analysis, the reason for doing so should be stated, the temperature at which it was dried, and the origin of the barley.

2. *Determination of the Moisture.*—About 5 grms. of malt are weighed between two watch glasses, ground in a mill, and the ground malt dried either in an air-bath or, preferably, in a water-oven. If the air-bath be used, the drying should not exceed four hours, if the water-bath, the heating should be continued until the malt ceases to lose weight. The difference between two experiments should not exceed 0.25 per cent. The temperature should be 97°–105° C. If the malt is very damp, the drying must commence at a lower temperature.

3. *Determination of the Extract.*—50 grms. of malt are ground and digested with 200 cc. of water at 45° C. for half an hour, the temperature is then allowed to rise to 70° C. at the rate of 1° C. rise per minute; this temperature will then be reached in 25 minutes, the mash is kept at 70° C. for one hour, it is then quickly cooled to 20° C., and the weight of the mash made up to 450 grms. by the addition of distilled water. The iodine colouration is to be taken 10 minutes after reaching 70° and every five minutes after. The mash is filtered through a large folded filter, the first 100 cc. being returned to the filter. The funnel is kept covered, and the filtrate received in a dry stoppered bottle. It is unnecessary to wait until the whole of the wort has been filtered. The odour and the clearness of the wort are to be noted. The colour is determined by comparison with solutions of iodine. The specific gravity by the pycnometer at 15° or 17.5° C., and the determinations converted into the amount of solid matter by Balling's tables.

The analysis of roasted malt is to be made with 25 grms., to which are added 25 grms. of malt of which the extract yield is known.

4. *The Determination of Maltose.*—25 cc. of wort are diluted to 200 cc., and 25 cc. of the dilute solution taken and boiled with 50 cc. of Fehling's solution for four minutes, the determination being made according to Soxhlet's directions.—A. L. S.

#### *Quinine Wine.* Pharm. Zeit. 1890, **35**, 371.

FIFTY grms. of powdered Peruvian bark are macerated with 50 grms. alcohol and 450 grms. of water for two days. After pressing, the bark is again treated in the same way. The mixed extract is evaporated to 100 grms., allowed to stand two days, filtered and evaporated to a thin syrup. This is dissolved in 50 grms. glycerol and 150 grms. of sugar syrup; 1 gm. of citric acid, 40 grms. of cognac and 760 grms. of sherry added, allowed to stand 14 days in a cool place and filtered. This wine is efficient, stable, and remains clear at ordinary temperatures.—A. L. S.

*The Presence of Furfuraldehyde (Furfural) in Commercial Alcohols.* L. Lindet. Compt. Rend. **111**, 236–238.

See under XXIII., page 1155.

#### PATENTS.

*Means for Treating Spent Grains or other Analogous Material for the Separation of Liquid therefrom.* G. Thornley and T. Buxton, Burton-on-Trent. Eng. Pat. 16,891, October 25, 1889. 4d.

See under I., page 1115.

*New or Improved Means and Apparatus for Indicating the Fermenting State or Condition of "Sponge" or Dough, preparatory to Baking or Cooking the same; applicable also to Indicating the Fermentation of other Substances and Fluids.* A. F. Garrod, Leytonstone. Eng. Pat. 6341, April 25, 1890. 8d.

See under XVII., page 1144.

*Improvements in Apparatus for the Complete Evaporation of Solutions.* H. E. Hansen, Copenhagen Denmark. Eng. Pat. 14,059, September 6, 1890. 6d.

See under I., page 1116.

#### ERRATUM.

This Journal, November number, 1890, 1054, columns 1 and 2, throughout the abstract on "The Influence of Various Fruit Ferments," &c., for "Enzyme" read "Yeast or Ferment."

## XVIII.—CHEMISTRY OF FOODS, SANITARY CHEMISTRY, AND DISINFECTANTS.

### (A.)—CHEMISTRY OF FOOD.

*Freezing Meat by means of Cold Liquids.* T. Schloesing. Compt. Rend. **111**, 85–89.

A COMMISSION instituted by the French Minister of War, under the presidency of Berthelot, to inquire into the best method of freezing meat rapidly and of preserving it at a low temperature, has adopted the following arrangement as the most suitable. The cooling is effected by means of air, which latter is itself cooled by passing down a coke tower, up which a current of cold liquid is forced. This

tower is fixed in the centre of the cylindrical space in which the meat is placed and has a ventilator on the top which serves to draw in the air to be cooled, which then passes over the coke and down into the chamber containing the meat; as this air becomes heated, it rises and is then again drawn through the coke tower and this process repeated. The tower is made of wood and rests on wooden blocks. The freezing liquid is conveyed from the freezing machine by an iron tube to the axis of the tower, and from this tube a number of lead tubes furnished with numerous orifices branch off horizontally. The liquid as it runs down is collected in a leaden vessel and taken back to the freezing machine. To prevent the calcium chloride added to the freezing liquid in order to keep it liquid, coming in contact with the meat, the tower is surrounded by a jacket made of planks of wood. Details of the arrangements for hanging up the meat to be frozen and of the precautions taken to prevent the access of heat from the outside are given. The diameter of the tower employed was 2 metres, but its size must be regulated by that of the room in which the meat is frozen.—C. A. K.

#### PATENT.

*New or Improved Means and Apparatus for Indicating the Fermenting State or Condition of "Sponge" or Dough preparatory to Baking or Cooking the same; applicable also to Indicating the Fermentation of other Substances and Fluids.* A. F. Garrood, Leytonstone. Eng. Pat. 6341, April 25, 1890. 8d.

THE invention has for its object the indication of the condition of "sponge" or dough during the process of fermentation by giving an alarm signal by means of an electric bell or otherwise, actuated by the movement of the dough, &c. The invention may be adapted for brewing and other analogous processes of fermentation.—V. H. V.

#### (B.)—SANITARY CHEMISTRY.

##### PATENTS.

*Improvements in the Treatment of Sewage and Sewage Matters and in the Production of Fertilising Products therefrom.* U. B. Kemshead, Mitcham. Eng. Pat. 18,677, November 21, 1889. 4d.

THE sewage is received in a circular tank and there is disinfected together with the admixed water, by the addition of a disinfecting powder known commercially under the name of "Smith's Disinfecting Cleaning Powder." Other powders may be used provided they contain neither lime, chlorine, nor carbolic acid. The powder, in the proportion of about 1 lb. to about 3 to 4 gallons of sewage, is well mixed by means of an agitator and the whole allowed to stand for two or three hours. The surplus water is separated by filtration and might at once be run into a stream, but as it contains a certain quantity of ammoniacal salts in solution it is preferably used for irrigating a sewage farm. The sludge when mixed with potassium and ammonium sulphate (the refuse from gasworks) may be used as a fertiliser for root-crops and ordinary purposes; the proportion adopted is 1 lb. of each of the salts to about 4 gallons of sludge. If used for cereal crops, 1 lb. of soluble potassium silicate is added. These admixtures are then mixed with loam or peat earth or road sweepings and the whole dried like bricks. The cakes of material can be reduced to powder and are then ready for packing in tins.—V. H. V.

*Improvements in the Treatment of Organic Matters for the Production of Manures, and in Apparatus therefor.* J. Guillaumie, Paris, France. Eng. Pat. 5419, April 9, 1890. 11d.

THE refuse matter, raw or solid, containing about 87 per cent. of water, is diverted into a receiving basin, in which air is forced under a pressure of 25 cms. of water. The raw material is herein acidified with sulphuric acid, an

operation which has the double advantage of partially disinfecting it and of fixing the ammoniacal products. The material is then carried automatically by an elevator into a reservoir, whence it is distributed into a desiccator; in this the materials come in contact with air supplied from a furnace by a ventilator at the rate of 24,000 cubic metres per hour at a temperature of 350°–400°. Bucket wheels on a horizontal shaft are situated in the centre of the desiccator by means of which the matter is lifted up and allowed to fall again in a shower on the hot current of air. By this means the mass loses the greater part of the water, and the air proceeding from the apparatus, and therefore saturated with aqueous vapour and mixed with the noxious gas, is conducted by a tunnel into the drying room. This consists of a large chamber, the raised floor of which is constructed of cast-iron plates, on which the substances containing about 40 per cent. of moisture are placed. The hot air circulating under the plates completes the desiccation and the matter is then ready for agricultural purposes. The air leaving the drying room is conveyed into a refrigerator in which it comes in contact with a fine spray of water, whereby the vapour is condensed; it then passes into the drying room formed of several circulating chambers filled with fagots to retain the moisture. The comparatively dry air is then passed through a tunnel and carried into the furnace where it is burnt, thereby saving fuel. The whole of the work is effected in closed vessels and without contact with the outside air except that drawn in by the ventilator to compensate for the loss of oxygen resulting from the combustion. The specification is accompanied by illustrative plans.—V. H. V.

#### (C.)—DISINFECTANTS.

*Tar Soaps and "Lysol," a New Disinfectant.* C. Engler. Dingl. Polyt. J. 278, 26–34 and 78–83; Pharm. Centrallhalle, 1890, 449.

See under XII., page 1136.

##### PATENTS.

*Improvements in Disinfectants and Deodorisers.* J. Price, London. Eng. Pat. 18,512, November 19, 1889. 6d.

THE material consists of one part of sodium manganate,  $1\frac{1}{2}$  parts of chloride of lime, and  $17\frac{1}{2}$  parts of salt cake. This mixture is prepared in a dry state, and when exposed gives off chlorine for a week or more without appreciable deliquescence; after exposure it is almost as efficacious for treating foul water or putting down drains. On account of its solubility it presents advantages over similar mixtures made up with lime, chalk, or sawdust, which choke up the drains and sewers. It is proposed to call the above mixture "sal-ozone."—V. H. V.

*Improvements in the Process of Producing Disinfectants.* A. Artmann, Braunschweig, and H. W. Kufcke, Hamburg, Germany. Eng. Pat. 20,231, December 16, 1889. 4d.

JEYES' disinfectant, or "creolin," consists of a mixture of tar oils containing carbolic acid of high boiling point, with small quantities of a pyridine base and caustic soda, resin, soap, and water. This disinfectant is easily soluble in water, but loses its emulsive power when hydrochloric acid, or sulphuric acid, or soda lye, &c. is added to it. As its disinfecting quality depends upon the presence of carbolic acid, its employment for many purposes is dangerous.

According to this invention, coal-tar oil, freed from carbolic acid, and of boiling point 180°–220° C., is shaken up with concentrated sulphuric acid in small quantities at a time (about 4 per cent. of the tar) at ordinary temperature. This process is continued until a sample, when shaken up with water, has a milky appearance. The tar oil thus heated with sulphuric acid, and partly converted into sulphonic acids and sulphones, is shaken up with water, and the milky liquid which contains the sulphonated hydrocarbon is separated from the undissolved tar oils,

which rise to the surface. The milky liquid is then heated with hydrochloric acid or common salt, when it separates in two layers, the upper one of which contains the sulpho-nated compound. This is separated off, neutralised with soda, then heated with spirit or other suitable solvent, and then submitted to distillation to remove the solvent; the remaining portion is mixed with a neutral coal-tar hydrocarbon. It is proposed to call the product formed "Artmann's creolin;" it possesses disinfecting qualities, and gives a durable emulsion with water, but it is not poisonous. A certain proportion of carbolic acid may be added; the mixture is then called "Artmann's basic creolin."

—V. H. V.

## XIX.—PAPER, PASTEBOARD, Etc.

*Distinction between Genuine and Imitation Parchment Paper.* E. Muth. Dingl. Polyt. J. **277**, 360—361.

See under XXIII., page 1156.

*Some New Processes in Paper Manufacture.* A. Haussner. Dingl. Polyt. J. **277**, 118—121.

INSTEAD of boiling rags in paper manufacture with lime, it has been recommended that a substance called "ammonine" should be used, a grey powder consisting, according to Dennewitz, of carbonate and silicate of soda, to which a considerable quantity of carbon bisulphide has been added. Its chief value is that it cleanses animal as well as vegetable fibres thoroughly without injuring them. Instead of subjecting the rags to boiling heat, a temperature of only 60° C. is applied, 5 kilos. of "ammonine" and 300 litres of water being necessary for every 100 kilos. of rags. The time of treatment is from half an hour to one hour. Coloured rags have to be treated with soda as well.

It has further been recommended of late to wash the rags in specially constructed washing machines, and not in the beaters, as is often done. But it seems to be difficult to construct a washing machine which is not injurious to the fibres, and it seems best to use modifications of the beaters, fitted with rolls without knives.

For the bleaching of rags, the so-called "oil-bleaching process" has been recommended lately. It was accidentally discovered that certain oils obtained from bituminous slate have a purifying influence upon rags, if added to them during the boiling process, a great saving of other bleaching materials being hereby effected. The quantity of oil to be used varies according to the nature of the rags, as follows:—

	Cc. of Oil.	
1. For linen rags (coarse sacking).....	700—900	For every 100 kilos.
2. For dyed cotton rags.....	700	
3. For bleached rags.....	500	
4. For esparto.....	1,400—1,800	

This process has also been used, with good results, for lignified substances and for straw. Easily inflammable oils have to be avoided as dangerous. A satisfactory explanation of this method has hitherto not been given, but its influence seems to be more mechanical than chemical. It is of special advantage if applied in connexion with the lime treatment of rags. Frank is of opinion that these oils may be used with advantage also in the treatment of sulphite cellulose, in order to obviate the difficulties which are sometimes caused by resinous secretions in the raw materials.—H. S.

*Percentage of Cellulose in Wood.* Papier Zeit.; and Chem. Trade J. 1890, **7**, 335.

See under XXIII., page 1156.

*A Quantitative Reaction for Lignin.* R. Benedikt and M. Bamberger. Monatsh. **11**, 260—267.

See under XXIII., page 1156.

## PATENTS.

*Improvements in Apparatus for Boiling Esparto Grass and other Materials.* G. W. Hawksley, Sheffield. Eng. Pat. 4976, March 31, 1890. 6d.

IN a cylindrical or other shaped vessel an upright cylinder is fixed, containing a series of vertical tubes. Steam admitted to the cylinder heats the liquor contained in the vertical tubes, causing it to rise and overflow, thus producing a rapid circulation.—E. J. B.

*Improvements in the Manufacture of Absorbent Sheets, Rolls, or Pads of Paper for Blotting, Filtering, and other Similar Purposes.* E. G. Wrigley, Preston. Eng. Pat. 5500, April 11, 1890. 4d.

THE inventor claims the combination of wood cellulose with cotton rags and vegetable fibres of a grassy nature, paper so made being more absorbent than one made entirely of cotton rags or wood pulp. Instead of passing the web of paper through press rolls it is pressed between two felts, the fibres thereby being kept more open.—E. J. B.

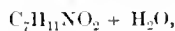
## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

*Indian Geranium Oil.* F. W. Semmler. Ber. **23**, 2965—2968.

IN a previous paper (this Journal, 1890, 889) the author has shown that the substance geraniol,  $C_{10}H_{18}O$ , obtained from geranium oil, is probably an open chain compound. An examination of bodies of the same formula, obtained from coriander oil, bergamot oil, and other ethereal oils, seems to show that these latter are also similarly constituted. A difficulty in the previous attempts made to prove the presence of the alcohol group in geraniol was introduced by all the derivatives prepared being non-crystalline. With the hope of obtaining a crystalline product geraniol was oxidised, when a body,  $C_{10}H_{16}O$ , resulted, which formed a crystalline acid sulphite. The oxidation was effected by means of chromic acid mixture, the product of the reaction being made slightly alkaline, and then distilled with steam, when the oxidation product passed over as an oil possessing a lemon-like smell. Several substances appeared to be formed in the oxidation—including terpenes—but the chief product was the body  $C_{10}H_{16}O$ , which was separated by shaking with acid sodium sulphite and decomposing the resulting crystalline compound, after washing with ether to remove adhering oily substances, with sodium carbonate. A colourless oil was obtained, whose analysis and a vapour density determination corresponded to the above formula. This substance, which contains two atoms of hydrogen less than geraniol, must either be a ketone or an aldehyde; from some of its properties the latter appears more probable. Details are reserved for a future paper.—C. A. K.

*The Alkaloids of the Areca Nut.* E. Jahns. Ber. **23**, 2972—2978.

THE areca nut (from *Areca catechu*) contains several alkaloids, including arecoline,  $C_8H_{13}NO_2$ , arecaine—



and a third basic substance which only occurs in small quantities (this Journal, 1889, 210). This last has proved to be nothing more nor less than choline, which was identified by means of its platinum double chloride. This salt separates in monoclinic crystals from aqueous solution; from alcoholic solution yellow octahedra, containing 1 mol. of water of crystallisation, are formed. It melts at 225°; the gold double chloride of choline at 244°—245°.

*Arecoline.*—When heated with hydrochloric acid in a closed tube to 150°—160° arecoline is decomposed with liberation of methyl chloride and formation of a body, arecaine,  $C_7H_{11}NO_2$ , isomeric with arecaine. This decomposition is effected with hydriodic acid by simple boiling, whilst caustic potash and baryta water decompose the arecoline still more readily—partial decomposition taking place in the cold on prolonged standing. Owing to this action of alkalis on arecoline the former should not be employed in extracting the latter from areca nuts, but the method described by the author (v. ante) in which dilute sulphuric acid is used should be substituted.

*Arecaine*,  $C_7H_{11}NO_2 + H_2O$ .—The product of the above reaction when crystallised from 60—70 per cent. alcohol forms colourless tabular crystals, containing 1 mol. of water of crystallisation, which they lose at 100°. It is soluble in water and in dilute alcohol; almost insoluble in absolute alcohol and insoluble in ether, chloroform and benzene. The dehydrated substance melts at 222°—223°, and carbonises on further heating. Like arecaine, arecaine has no poisonous action. It forms salts with acids which dissolve readily in water, with difficulty in alcohol, and which have an acid reaction. The platinum double chloride forms yellow octahedra which melt at 208°—209°; the gold double chloride crystallises in prisms, melting at 197°—198°. By passing hydrochloric acid gas into arecaine suspended in methyl alcohol, arecoline is formed, so that the latter appears as the methyl derivative of the former, a fact which is evident from the method of preparation of arecaine.

*Homarecoline*,  $C_9H_{15}NO_2$ .—This is the name given to the ethyl compound derived from arecaine by treatment with gaseous hydrochloric acid in presence of ethyl alcohol. It closely resembles arecoline, forming a strongly alkaline liquid, miscible with water, alcohol and ether in all proportions, distilling without decomposition and readily volatile with steam. It is poisonous, like its lower homologue arecoline. Of its salts the hydrochloride and gold and platinum double chlorides are described. The author regards arecaine as a weak monobasic acid, a fact borne out by its forming compounds with basic oxides and by its decomposing carbonates. It does not form an acetyl compound, and therefore it is not likely that the hydroxyl group is present, so that arecoline may be looked upon as the methyl ester of an acid,  $C_6H_{10}N.CO.OH$ . The nature of the nitrogenous nucleus  $C_6H_{10}N$  is under investigation.

—C. A. K.

*Poisonous Character of Diamide.* O. Loew. Ber. **23**, 3203—3206.

DIAMIDE, like hydroxylamine, is a strong poison in the case of the most different organisms; it is a poison of universal character. Both bodies distinguish themselves in their chemical relations by their energetic action on aldehydes and ketones. The author has already shown (Ber. Ges. Morph. und Physiolog. in München, 1888, 126) that the poisonous nature of hydroxylamine cannot depend on any conversion it may undergo into nitrous acid.

*Strychnine.* C. Stöckh. J. prakt. Chem. **42**, 399—415.

STRYCHNINE hydrochloride crystallises with  $1\frac{1}{2}$  molecules of water which are driven off at 150° or on prolonged standing over sulphuric acid; the hydrobromide contains 1 molecule of water of crystallisation, and both salts are very hygroscopic in the dehydrated state. The hydriodide is readily precipitated on the addition of potassium iodide to either of the foregoing, being almost insoluble in cold water; it also crystallises with 1 molecule of water. Analyses of these salts, as well as of the platinum double chloride, point to the generally accepted formula,  $C_{21}H_{22}N_2O_5$ , for strychnine. The melting point of the free base was found to be 269° C. The products of distillation of strychnine and lime have been previously examined by the author. The gaseous products were found to be hydrogen, ethylene and ammonia, whilst the liquid distillate consisted of an aqueous and an oily layer, in which ammonia and  $\beta$ -picoline were found. Ethylamine was also found in the aqueous portion of the distillate, whilst the oily portion contains skatole,  $\beta$ -picoline and perhaps  $\beta$ -ethylpyridine. The  $\beta$ -picoline boiled at 142°—143°, and the author does not consider that the differences in the boiling points of the  $\beta$ -picoline thus obtained, and that prepared by other processes (coal-tar  $\beta$ -picoline boils at 144°—148°, and that prepared by various synthetical methods between the extreme limits of 140°—146°) justify one in the assumption that there are two  $\beta$ -picolines (vide Ladenburg, Ber. **23**, 2688), but is of opinion that all the  $\beta$ -picolines hitherto prepared are identical. The author has shown in a previous paper that the study of the action of hydrochloric acid on strychnine led to no positive conclusions as to the manner in which the oxygen atoms are combined in the strychnine molecule, and he has therefore investigated the action of phosphorus pentachloride on the alkaloid with the same point in view. The two substances were heated together in chloroform solution on the water-bath until hydrochloric acid was no longer evolved, the product of the reaction poured into water, made alkaline with ammonia, and extracted with ether. The chief product of the reaction is a trichloro-strychnine,  $C_{21}H_{19}Cl_3N_2O_5$ , which somewhat resembles the substance described by Richet and Bouchardat's (Compt. Rend. **91**, 990) as monochlorostrychnine. It is insoluble in water, soluble in alcohol with a red colour, and separates from this solution in microscopic crystals. It possesses the bitter taste of strychnine, though this property is not quite so marked, and gives similar colour reactions. On heating it decomposes without melting. It is basic and forms a characteristic hydrochloride, platinum double chloride and sulphate; an analysis of the first of these is given. A secondary product of the action of phosphorus pentachloride is a chlorinated body, possessing no basic properties, and probably a mixture of tetra- and pentachlorostrychnine. It melts at 224°, darkening somewhat. The yield of this substance is very meagre.

This result leaves no doubt as to the absence of a hydroxyl group in strychnine, but the author hopes to be able to gain more insight into the constitution of the alkaloid by a study of the oxidation products with potassium permanganate, on which he is now engaged.—C. A. K.

*Brucine.* L. Berend and C. Stöckh. J. prakt. Chem. **42**, 415—420.

BRUCINE, when distilled with lime, evolves hydrogen, ethylene and ammonia as gaseous products, whilst the liquid distillate contains a little skatole, methylamine, and a methyl pyridine identical in its properties and boiling point (142°—143°) with the  $\beta$ -picoline obtained by the treatment of strychnine under similar conditions. (See previous abstract.)

The analysis of the free base and of its platinum chloride establish the formula  $C_{21}H_{22}N_2$ , and there is no doubt of the complete identity of the two picolines obtained from strychnine and brucine respectively. The  $\beta$ -picoline has a sp. gr. of 0.9756 at 0°; Zanoni found this value 0.9771. A lutidine, perhaps  $\beta$ -ethyl pyridine, is also found in the distillate, which is, however, free from quinoline bases.

—C. A. K.



*The Alkaloids of Atropa Belladonna.* E. Schmidt.

Apoth. Zeit. 5, 511.

THE author has examined the roots of *Atropa belladonna* in order to test the correctness of the assertion made by the authorities of Schering's Factory, that good belladonna roots contain only hyoscyamine and no atropine at all. The roots examined were of one, two, eight, and more years' growth. They were collected in spring; in summer, when the plants were flowering; and in autumn, when the berries were ripe. It was found that the young roots contained only hyoscyamine; whilst the old roots contained besides much hyoscyamine a little atropine. The leaves of the ordinary wild-growing belladonna contained much hyoscyamine and a little atropine. The ripe berries contained only atropine.

—H. T. P.

*Investigations of Crude Acetone Boiling between the Temperatures of 75° and 135°.* L. Wolfes. Chem. Zeit. 14, 1141—1142.

THE material on which the following experiments were conducted was obtained from the works of Hofmann and Schötensack, Ludwigshafen. It was distilled, and the distillate collected in three fractions. The first portion contained a substance boiling at 80°, which gave a crystalline compound on being treated with sodium bisulphite; from repeated analyses, however, figures could not be obtained agreeing with the formula of methyl ethyl ketone. To purify it, it was treated with phenylhydrazine, and was found to give off a gas smelling strongly of ammonia. A second portion was combined with hydroxylamine, forming an oil boiling between 149° and 150°, which on analysis appeared to be the oxime of methyl ethyl ketone.

Second fraction.—By repeated distillation, a substance boiling at 102°—103° was next obtained, which possessed, approximately, the formula of methyl propyl ketone; this also gave with sodium bisulphite a white crystalline body, whose sulphur and sodium agreed closely with the ketone compound. With hydroxylamine it gave an oxime boiling at 167°. For its further identification, a fresh portion was oxidised with potassium permanganate, and the salts of the resulting acids converted into silver compounds; and owing to the different degree of solubility of silver acetate and propionate, it was found possible to purify each separately and identify them. That no diethyl ketone was present in this fraction was proved by the fact that the filtrate from the bisulphite precipitate contained no more oil.

Third fraction.—This portion was found to boil between 125° and 131°, and by repeated fractionation was split up into several fractions of about the same amount. Of these, the one boiling at 128°—129° gave on analysis figures midway between those of methyl butyl ketone and diallyl ketone. This fraction, partially combined with sodium bisulphite, and the oil, freed from its combination by means of sodium carbonate, gave an oxime boiling at 182°, which proved to be that of methyl butyl ketone. Another part oxidised with potassium permanganate, the potassium salts converted into free acids by means of sulphuric acid and extracted with ether, the butyric acid was found chiefly in the ethereal layer, the acetic acid being only dissolved in traces. From the ethereal layer, on the addition of silver nitrate, silver butyrate crystallised out, and from the aqueous, the acetate was obtained, thus proving distinctly that the substance in question was methyl butyl ketone.

The oils which gave no crystalline compound with sodium bisulphite were distilled with a strong solution of caustic soda, the distillate forming an oil boiling at 128°—130°, which, however, did not appear to be any definite compound. This was oxidised with nitric acid, evaporated to dryness, the residue dissolved in water, and neutralised with caustic baryta; the precipitate was filtered off, suspended in water, and decomposed by sulphuric acid. The barium sulphate being removed, the solution was extracted with ether, which on evaporation left small crystals of succinic acid. The filtrate from the caustic baryta precipitate was treated with sulphuric acid to remove the barium and the filtrate extracted with ether; the residue was then treated with zinc carbonate in the cold, and after filtration evaporated

to dryness. The dried residue was dissolved in water and the solution heated, whereupon zinc pyrotartrate separated out.

This formation of pyrotartaric acid from that portion of the oil which gives no crystalline precipitate with sodium bisulphite proves that there is a ketone present containing a closed chain, which is probably identical with the one described by Kane (Ber. 8, 1256) as dumasine.—F. H. L.

*Terpenes and Etheral Oils.* O. Wallach. Annalen, 259, 309—324. (Compare this Journal, 1889, 635 and 959.)

PINOL must be regarded as the first product formed by the oxidation of oil of turpentine with oxides of nitrogen; it is, therefore, always formed in the preparation of pinenitroschloride. It is an unsaturated compound and combines with bromine, yielding a readily crystalline dibromide of the composition  $C_{10}H_{16}OBr_2$ , which melts at 94°.

*Pinol acetate*,  $C_{10}H_{16}O(O.CO.CH_3)_2$ , is obtained, together with pinol and pinol glycol when the dibromide is warmed with sodium acetate, or better still, with silver acetate, in glacial acetic acid solution; it is purified by distillation under reduced pressure. It crystallises well from water, melts at 97°—98°, and boils at 127° under a pressure of 13 mm.

*Pinol glycol*,  $C_{10}H_{16}O(OH)_2$ , is best prepared by boiling the acetate with dilute sulphuric acid for several hours; the glycol is then partially extracted by shaking with chloroform, and the remainder is obtained in crystals by evaporating the solution. It separates from water in compact crystals, melts at 125°, and is converted into the acetate when it is boiled with acetic anhydride.

*Pinol hydrate*,  $C_{10}H_{16}O < \begin{smallmatrix} H \\ OH \end{smallmatrix}$ , can be obtained by dissolving crude pinol (loc. cit.) in its own weight of glacial acetic acid, saturating the well-cooled solution with hydrogen bromide, and then shaking it with excess of well-cooled, very dilute caustic soda; the mixture is then submitted to distillation with steam to free it from cymol, and the residual solution extracted with ether. It crystallises in plates or needles and melts at 131°. When an aqueous solution of pinol hydrate is warmed with dilute sulphuric acid it quickly becomes turbid, owing to the separation of an oil; this oil is chemically pure pinol.

Pinol hydrate is identical with the crystalline compound which is formed, as was first observed by Sobrero, when oil of turpentine is left in contact with oxygen in presence of direct sunlight.

It has been previously stated that pinol gives terebinic acid on oxidation with potassium permanganate; further experiments with pure pinol have confirmed this statement. Pinol hydrate, on the other hand, yields terpenylic acid on oxidation with potassium permanganate under the conditions previously described; pinol glycol seems to behave in a similar manner.

Pure pinol is easily obtained from pinol hydrate as stated above, but the method of preparation of the hydrate must be greatly improved before it can be conveniently employed for the preparation of large quantities of pinol. Pure pinol can also be obtained by heating a solution of the pure dibromide in dry benzene with the theoretical quantity of very fine sodium wire until the whole of the metal is converted into bromide; the filtered solution is evaporated and the residual pinol purified by fractional distillation.

*Pinol tribromide*,  $C_{10}H_{15}OBr_3$ , is obtained as a by-product in the preparation of the dibromide from crude pinol; it crystallises from ethyl acetate in needles, and melts at 160° with decomposition.—F. S. K.

*Fenchol, a New Substance Isomeric with Camphor.*

O. Wallach and F. Bartmann. Annalen, 259, 324—331.

WHEN oil of fennel is distilled a portion passes over between 190° and 193°, which has a strong odour of camphor and a specific gravity of 0.934 at 23°; this liquid



has the composition  $C_{10}H_{16}O$ , and, judging from its behaviour, the authors consider it to be a position-isomeride of camphor, to which they give the name *fenchol*.

Fenchol combines with bromine when treated therewith in well-cooled light petroleum solution, yielding a bright red crystalline powder which deliquesces on exposure to the air, and is reconverted into fenchol by caustic soda.

*Fenchol oxime*,  $C_{10}H_{15}N.OH$ , is formed when fenchol is treated with hydroxylamine in alcoholic solution; it crystallises from alcohol in needles, melts at  $148^{\circ}$ – $149^{\circ}$ , and combines with hydrogen chloride, yielding a crystalline salt, melting at  $118^{\circ}$ – $119^{\circ}$ . The *anhydride*,  $C_{10}H_{15}N$ , is obtained as an oil when the oxime is warmed with dilute sulphuric acid; it boils at  $217^{\circ}$ – $219^{\circ}$ , and in physical properties resembles the anhydride obtained from camphor oxime. The two compounds can, however, be distinguished by their behaviour with hydrobromic acid. When the anhydride of camphor oxime is shaken with a well-cooled glacial acetic acid solution of hydrogen bromide, and the mixture then poured into ice-cold water, there is precipitated an oil which does not solidify on cooling. The anhydride of fenchol oxime gives, under the same conditions, an oil which readily crystallises in the cold, the crystalline substance being the unstable hydrobromide,  $C_{10}H_{15}N.HBr$ .

*Isafenchol oxime*,  $C_{10}H_{15}NO$ , is obtained when fenchol oxime is boiled with alcoholic potash for 5–6 hours; it melts at  $113^{\circ}$ – $114^{\circ}$ , and is isomeric with the compound which is formed from camphor oxime anhydride in a similar manner. When the anhydride of fenchol oxime is boiled with alcoholic potash for four days it is converted into *fencholinic acid*,  $C_{10}H_{16}O_2$ , a compound which corresponds to campholinic acid,  $C_{10}H_{16}O_2$ ; it is a colourless liquid and boils at  $257^{\circ}$ – $260^{\circ}$ .

In physical properties fenchol and its derivatives differ to a not inconsiderable extent from camphor and its derivatives, as is seen from the following table:—

	Fenchol Series.	Camphor Series.
$C_{10}H_{16}O$ .....	Liquid; boiling point, $136^{\circ}$ – $135^{\circ}$ .	Solid; boiling point, $204^{\circ}$ .
Oxime, $C_{10}H_{15}N.OH$ .....	Melting point, $148^{\circ}$ – $149^{\circ}$ .	Melting point, $118^{\circ}$ .
Oxime hydrochloride.....	Melting point, $118^{\circ}$ – $119^{\circ}$ .	Melting point, $153^{\circ}$ – $154^{\circ}$ .
Oxime anhydride, $C_{10}H_{15}N$ ...	Boiling point, $217^{\circ}$ – $219^{\circ}$ .	Boiling point, $216^{\circ}$ – $217^{\circ}$ .
Iso-oxime, $C_{10}H_{15}NO$ .....	Melting point, $113^{\circ}$ – $114^{\circ}$ .	Melting point, $124^{\circ}$ – $125^{\circ}$ .
Acid, $C_{10}H_{16}O_2$ .....	Boiling point, $257^{\circ}$ – $260^{\circ}$ .	Boiling point, $254^{\circ}$ – $255^{\circ}$ .

In chemical behaviour, however, the two series of compounds show a very striking similarity, a fact which seems to show that fenchol and camphor differ from one another only in the relative position of the oxygen atom in the molecule.—F. S. K.

#### Oxidation of Potassium Cyanide with Permanganate.

J. Volhard. *Annalen*, **259**, 377–380.

When potassium cyanide is oxidised with potassium permanganate in the cold a large quantity of potassium cyanate is produced; this reaction, if carried out as described below, may be conveniently employed for the preparation of urea.

To a solution of 39 grms. of potassium cyanide and 10 grms. of potash in 100 cc. of water, a solution of 63 grms. of potassium permanganate in 1 litre of water is gradually added by means of a dropping funnel, the temperature being kept below  $17^{\circ}$ ; when ice is employed for cooling the mixture, the whole of the permanganate can be added in about half an hour, but it is more convenient to cool with running water, a current of air being at the same time passed through the cyanide solution. As soon

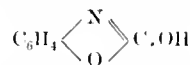
as the whole of the permanganate has been added the mixture is placed in cold water for about 7–8 hours until the green colouration has disappeared, then mixed with a concentrated solution of ammonium sulphate (70 grms.), boiled and filtered: the filtrate is evaporated and the urea extracted from the residue with boiling 95 per cent. alcohol. The average yield is 24 grms., or 68 per cent. of the theoretical quantity, but the urea obtained in this way still contains small quantities of chloride and sulphate of ammonium; it can be purified by treating its aqueous solution with freshly prepared barium carbonate, evaporating the filtrate to dryness, extracting the residue with absolute alcohol, and then recrystallising the product from alcohol.—F. S. K.

#### The Constitution of Carbonylorthamidophenol. O. Gressly and M. Nencki. *Monatsh.* **11**, 253–259.

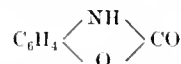
The authors allude to the methods of obtaining this substance and its two isomeric ethyl derivatives, and describe their own experiments conducted with the view to decide between the two formulae—



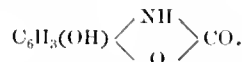
Their method consists in examining the changes which carbonylorthamidophenol undergoes in the animal system as compared with other aromatic compounds. It is known, for instance, that acetanilide administered to the rabbit becomes oxidised to paramidophenol, the acetyl group being split off. In the dog the main product is the anhydride of hydroxyphenylcarbanic acid, and in the human system acetanilide is partly converted into acetyl-paramidophenol. In each case therefore a hydrogen atom in the benzene nucleus is oxidised to hydroxyl. Aromatic compounds containing a carboxyl group are on the other hand not oxidised in the animal system, whilst the homologues of benzene, the aromatic alcohols, aldehydes, &c. are oxidised to carboxylic acids. Hydroxy compounds, such as the phenols, suffer no oxidation. In the case of substances containing neither a carboxyl nor a hydroxyl group, and where no oxidation of a side-chain takes place as in benzene, indole and camphor, a hydrogen atom in the nucleus again becomes oxidised to hydroxyl, the products being phenol, indoxyl and camphorol. Arguing from these facts the authors conclude that if carbonylorthamidophenol contains an hydroxyl group as indicated by the formula—



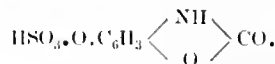
it should not suffer oxidation in passing through the system, whilst on the other hand if its constitution be—



they would expect a hydrogen atom in the nucleus to become oxidised to hydroxyl, and the product discharged by the animal should be—



Their experiments made both on rabbits and dogs show the latter view to be the correct one, the carbonylorthoxyamidophenol being discharged in the form of sulphate—



—A. K. M.

*Combinations of Camphor with Phenols and their Derivatives.* E. Leger. *Compt. Rend.* **111**, 109—111.

CAMPHOR forms a series of real compounds with the phenols, which, however, owing to their instability and the ease with which the components are regenerated by the action of heat, alkalis or solvents, have been regarded as simple mixtures. Some of these can be obtained crystalline, others only in the liquid state; the latter are regarded as definite compounds, because: (1) they result by mixing molecular proportions of their constituents; (2) when crystallised by cold the successive batches of crystals obtained all have the same composition; and (3) the introduction of phenols into an alcoholic solution of camphor diminishes the rotatory power of the latter by almost one-half.

The following compounds, prepared by simply bringing together molecular proportions of camphor and the phenol have been prepared, and were analysed polarimetrically:—

Phenol forms two compounds, both liquids,  $C_6H_6O \cdot C_{10}H_{16}O$  and  $2 C_6H_6O \cdot C_{10}H_{16}O$ , the latter of which combines with another molecule of camphor to form the former. Two compounds were also obtained from resorcinol,  $C_6H_6O_2 \cdot C_{10}H_{16}O$  and  $C_6H_6O_2 \cdot 2 C_{10}H_{16}O$ ; the first of these is a solid, melting about  $23^\circ$ , the second a liquid.  $\alpha$ -naphthol forms the compound  $C_{10}H_7O \cdot C_{10}H_{16}O$ , and  $\beta$ -naphthol,  $3 C_{10}H_7O \cdot 5 C_{10}H_{16}O$ ; both of these are liquid. With salicylic acid, a body,  $C_7H_5O_3 \cdot 2 C_{10}H_{16}O$  results, which is crystalline and only slightly decomposed by water; it melts towards  $60^\circ$ . Salol also appears to form a compound with camphor. The rotatory power of these substances are given in the paper.—C. A. K.

*The Rotatory Power of Camphor in Solution in Various Oils.* P. Chabot. *Compt. Rend.* **111**, 231—233.

THE author has examined the rotatory power of camphor in solutions of olive oil (sp. gr. 0.9125 at  $19^\circ C.$ ), almond oil (sp. gr. 0.9173, at  $19^\circ C.$ ), and seed oil (sp. gr. 0.9233 at  $19^\circ C.$ ). The rotatory power of these solutions is nearly proportional to the quantity of camphor dissolved, so that if the percentage of this body in solution be represented by  $p$ , the rotation of the plane of polarisation (using a sodium flame),  $\alpha$ , in a 20 cm. tube, is for—

Olive oil.....	$\alpha = 10' + p \cdot 1' 1''$
Almond oil.....	$\alpha = p$
Seed oil.....	$\alpha = 36' + p$

From these values the proportions of camphor contained in saturated solutions of the three oils can be calculated. The following results were obtained in this manner:—

	Camphor.
	Per Cent.
Saturated solution of olive oil at $19^\circ C.$ .....	26.983
„ „ almond oil „.....	28.53
„ „ seed oil „.....	28.89

Taking account of the slight rotatory power of the oil, the molecular rotatory power for camphor in each of the three oils  $[\alpha]_D$  has been found to be:—

(1.) *Olive Oil*—

For a 3 per cent. solution...	$[\alpha]_D = 55^\circ 12'$
„ 20 „ „.....	$[\alpha]_D = 55^\circ 12'$

(2.) *Almond Oil*—

For a 3 per cent. solution....	$[\alpha]_D = 56^\circ 47'$
„ 20 „ „.....	$[\alpha]_D = 54^\circ 19'$

(3.) *Seed Oil*—

For a 3 per cent. solution....	$[\alpha]_D = 54^\circ 24'$
„ 20 „ „.....	$[\alpha]_D = 54^\circ 7'$

It appears therefore that the value  $[\alpha]_D$  varies little with the degree of dilution, but increases with the latter, whilst with other solutions of camphor the reverse is the case.

—C. A. K.

*The Localisation of the Principles which furnish the Sulphur Essences of the Cruciferae.* L. Guignard. *Compt. Rend.* **111**, 249—251.

THE author describes a series of investigations having for their object the tracing of the localities in cruciferous plants in which sulphur containing essences, such as mustard oil, result, and finds that the ferment (myrosine) and the glucoside (potassium myronate), from the mutual reaction of which the mustard oil results, are contained in distinct and easily recognisable cells in the various parts of the plants.

—C. A. K.

*The Purple produced by Purpurea lapillus.* A. Letellier. *Compt. Rend.* **111**, 307—309.

FROM the moment when the colouration appears, an alliaceous odour is noticeable, and chemists have been inclined to attribute this to the formation of allyl sulphide. The odour is obtained not only by exposing the substance of *Purpurea lapillus* itself to light, but also from the crystallised substance extracted from the same; the odour is, therefore, not due to putrefaction, but has for its origin true chemical change.

The author has succeeded in proving directly that the odour is due to allyl sulphide, though only very small quantities are formed.

Presence of a cyanide or sulphocyanide is also assured.

—T. L. B.

*Otto of Rose.* R. Blondel. *Les produits Odorants des Rosières*, Paris, 1889, and *Pharm. J.* 1890, July 5, 3—5.

THE most important centre of the production of otto of rose is in the region of the Balkan mountains, and includes about 150 villages situated in the two valleys of Toundja and Strema, affluents of the river Maritza. These two valleys run from west to east and lie between the Great Balkan chain on the north and the Svedna Gora mountains on the south, the extreme points where rose culture is carried on being Koprivchitza on the west and Twarditza on the east. The best otto is obtained on the southern slope of the Balkans. Here the climate is temperate, but the variations in temperature are sudden and frequent. The ground is sandy and porous, as a rule; where this is not the case, and water accumulates, roses are killed by the frost, or by fungi which attack the roots. Although the rose cultivation extends over a large tract of country, it is nowhere conducted on a large scale by any one individual, but is divided into a large number of small plots belonging to small proprietors. The dealers live in the towns, and although they sometimes buy the flowers and distil the oil, they rarely cultivate the roses, and then only to a small extent.

The roses are grown in long borders or plots 100 or 200 metres long, the plants attaining the height of a man. Between the plots a space of  $1\frac{1}{2}$  to 2 metres is left, so as to allow of a cart passing along between them.

Two roses are usually cultivated, a red and a white one, specimens of which, gathered in the Kejanlik plantations, have been identified by the author. The red one proved to be a branching form of *Rosa damascena*, and the white one *Rosa alba*.

The *Rosa alba* is employed to form a dividing line between the different plantations, and is also grown at the end of each plot, so as to present only roses of less value to the peasant, who would otherwise steal the more valuable red rose; it is not used, as a rule, for distillation. Dishonest cultivators, however, take advantage of the fact that when distilled it yields a product rich in stearoptene (although of poor fragrance), to mix it with the red rose, and thus obtain an otto which will bear dilution with Turkish oil of geranium (oil of *Andropogon schenanthus*, L.).

In May of the second year the rose trees are about 2 feet high and yield sufficient flowers to pay the expense of their cultivation up to that date. The next year they are in full bearing, and the maximum product is obtained when they are five years old, although the plants will live more than

20 years. At 10 years of age they are, however, usually cut level with the ground, new branches, and even flowers, appearing the next year.

The flowers commence to expand between May 20th and 28th, and they are collected daily till June 15th and 20th. The collection commences every day at dawn, for an opening flower-bud left till next day will then have lost its fragrance and its colour.

The contents of the baskets are weighed in the field, paid for and emptied into sacks, which are carried at once to the distillery. If the white roses are present in too large proportion they are paid for separately at a lower price. The stills are usually placed in sheds near the river, for the convenience of obtaining a good supply of water, and in some cases are let by the proprietors at the rate of 4 francs per still. The stills are of copper, and are made to contain 75 litres of water and 10 kilos. of flowers. The latter are introduced by means of an osier basket, and the stills are heated with long logs of wood lighted at one end, placed under the still. After a time, if steam comes out of the refrigerating tube, the fire is moderated, and when 10 kilos. (two flasks) of rose-water have distilled over, the fire is removed by withdrawing the burning logs. The still when cool is opened, the contents strained through the osier basket, and the water remaining in the stills saved for the next distillation. If the operation is continued until 15 kilos. of rose-water is obtained the otto is of inferior quality, and contains more stearoptene.

The rose-water is then submitted to distillation a second time, 40 litres being distilled at a time, and only 5 litres collected. The vessels in which it is received have the shape of a long necked flask, and the water is at first milky, like an emulsion, the oil floating after a time in the neck of the flask. It is removed by inserting a conical tin funnel about two centimetres long, furnished with a slender handle, and having a capillary orifice. It is dipped below the surface of the otto, and when a drop of oil on raising the funnel appears at the orifice it is emptied quickly into a special flask.

About 3,000 kilos. of flowers are calculated to yield a kilo. of otto, and this quantity of flowers is the usual yield per hectare, so that it may be considered that a hectare of roses yields a kilo. of otto. The adulteration of the otto takes place chiefly by the addition of oil of rose geranium (*Andropogon schenanthus*), whereby the congealing point is lowered from 15° or 16° R. to 14°, 13°, or 12°, and even lower. The purchaser of otto generally brings with him a basin and thermometer, and having obtained the desired temperature by mixing warm and cold water, and testing the temperature with the thermometer, inserts a flask containing 15 grms. of the essence to be tested. In about three minutes crystalline needles appear, and in 10 minutes the otto is solid enough for the uncorked flask to be reversed without spilling a drop. The price varies according to the degree of congealing point, that below 12° R. being openly sold at a special rate. The average production of otto of rose in Bulgaria is about 3,000 kilos. in good years, and 1,500 kilos. in bad ones.

The peasants are paid according to the season at the rate of .32 to .23 fr. per gramme for otto of standard purity. The flattened circular metal bottles in which it is exported contain 400 grms., 1 kilo. or 2½ kilos., and the price of the pure essence is 800 to 900 frs. per kilo., according to the season. The bottles are generally enveloped in felt. When the essence is freshly distilled it has a slight empyreumatic odour, possibly due to the fact that the water in the stills at the end of the day, having served for several distillations, has dissolved vegetable salts and boils at a temperature distinctly above the boiling point of pure distilled water. Otto of rose is also said to be produced in notable quantities in Asia Minor, at Broussa, Bourgas, and elsewhere by emigrants from Roumelia.

In Provence the cultivation of the roses is at the present time a flourishing industry, and the otto prepared in that district is considered superior to the Bulgarian product. It is obtained from *Rosa centifolia* (a specimen of the plant used from Grasse having been determined by Dr. Blondel). This rose flowers there in April and May. Rose-water is sometimes distilled from the yellowish tea rose known as

Safrano, which flowers in autumn and winter, but the water is very inferior, and the otto obtained from it is still more so, and unfit to mix with that of *R. centifolia*.

The cultivation of *Rosa centifolia* is carried on chiefly around Grasse, Cannes, Nice, and Valois, by peasant proprietors, who sell the flowers to the distillers in the towns. The chief use of the roses is for making rose pomade, only a small quantity, comparatively speaking, being distilled for the otto. The flowers are distilled in large stills, such as are generally used for spirit, some holding as much as 150 kilos. of flowers. Often one refrigerator receives the condensing pipes of several stills.

Usually 50 kilos. of flowers and 300 litres of water are placed in the still at one time, and 100 litres are distilled over, the first 25 being the richest in oil, and known as eau double de rose; the next 30 are of medium, and the remaining 45 of inferior quality. The oil which floats is collected by means of a florentine receiver. The yield of otto is small, 100,000 kilos. of flowers giving only a kilo. of otto, and the price is about 1,000 francs per litre; the average price of the rose-water is 2-60 francs per litre.

In Germany roses have been distilled for about five years in the neighbourhood of Leipzig, about six hectares being at present under cultivation. The species employed is *Rosa centifolia*, and the amount produced in 1887 was 3,000 litres of rose-water and 2 kilos. of otto. The latter congeals at 20° C., and contains 32½ to 34 per cent. of stearoptene, which is in excess of that in Bulgarian otto. Indeed the proportion of stearoptene present in otto of rose is always greater in cold countries.

Formerly otto of rose used to be adulterated by mixing it direct with oil of ginger grass, but now the flowers are sprinkled with the oil when placed in the retort. The odours under these circumstances amalgamate better, and during the distillation the proportion of waxy (cireuse) matter that passes over is larger, so that otto thus obtained may solidify at 14°, even when containing 25 per cent. of oil of ginger grass. In this case, however, the otto appears muddy, and does not present the sparkling crystals characteristic of pure otto. The odour also is easily detected by an expert. Bromine turns pure otto of rose of a green colour, but is itself decolourised by oil of ginger grass, and if a few drops of liquor potassæ are subsequently added, an apple-green precipitate in viscous clots is formed in a colourless liquid, and the odour of roses is not altered; but if adulterated with ginger-grass oil a yellow milky precipitate is obtained in a reddish liquid, and a disagreeable odour is given off. This test, however, is qualitative rather than quantitative.

The presence of spermaceti or paraffin in otto is detected in Bulgaria by rubbing the small test bottle of otto between the hands, containing about 15 grms., when the essence rapidly liquefies, since a temperature of 18° C. suffices, whereas paraffin does not melt till 32°—50° and spermaceti at 46° C., and these remain undissolved at the bottom of the bottle.

Experiments made by the author show that pure otto of rose has some physiological effect. In the dose of five or even of two drops it produced cramp of the stomach in the absence of food. Taken after a meal it seemed to stimulate the digestive functions at first, but doses of 10 or 12 drops appeared to have an opposite action, and caused marked flatulence. The only general effect noticed was a marked tendency to sleep. A guinea pig into which a cubic centimetre of otto was subcutaneously injected died in four days after a state of profound torpor lasting 48 hours. The only effect noticed in a post-mortem examination was a slight congestion of the meninges. In some persons the odour of the rose produces headache, and in rare cases severe symptoms, which are probably due to idiosyncrasy. The oil of *Andropogon schenanthus*, or Turkish oil of geranium, used to adulterate oil of roses, produces similar symptoms, but in a higher degree.

With regard to the portion of the flower in which the otto is formed, observations made under the microscope indicate that it is secreted by the epidermal cells. Other experiments show that these cells also contain fixed fatty matter, since when the petals are distilled until wholly deprived of odour, these epidermal cells are still blackened by osmic acid, and yield to ether a fixed fatty substance. But the fresh petals treated directly with ether, and thus

deprived both of fixed oil and essential oil, still show the blackened epidermal cells when treated with osmic acid, but with a bluer tint in them. This is due to tannin, as may be proved on a section of the fresh petal by the use of acetate of iron. When the petals are deprived of their essential oil by distillation of their tannin by boiling water, and their oil by ether, the epidermal cells no longer become blackened by osmic acid. There is, therefore, every reason to suppose that the chemical changes by which the otto is produced take place in the epidermal cells, or in other words that they are not special receptacles of secretion but secreting cells. The epidermis of the anther does not contain otto, but it is contained in small quantity in that of the filament and on the stigmatic surface and groove, though not in that of the ovary or style.

*Camphor in Florida.* Chemist and Druggist, November 29, 1890, 750.

At the October pharmaceutical meeting of the Philadelphia College of Pharmacy Professor Maisch gave an account of the prospective camphor-industry in Florida. It seems that the camphor tree grows in almost any kind of Florida soil, and is not affected by cold weather. At four years old the trees are 10 ft. high, and the trunk is 4 in. in diameter; at 10 years one tree was 35 ft. high, the trunk being 10 ft. long, and 1 ft. in diameter. Some of the branches, weighing 13 lb., yielded on distillation a teacupful of camphor, which was separated by cold from the oil which distilled over along with the steam. It is thought that in 10 years more camphor trees will be growing in Florida than orange trees, and that the industry will prove to be more profitable than the production of sugar. At the present time the price for camphor tree yearlings is from 25 to 50 cents. It is recommended that at the age of four or five years the first cutting should be made for distillation by pruning from the ground to the height at which the head of the tree is to be retained. From that time on, the head has to be sheared in a suitable manner, without neglecting the pruning from the ground up, with the view of making the head larger. With this treatment distillation would be carried on every year, and in 12 or 15 years the trunk of the tree will have attained a sufficient size to be sawn into timber, so that the valuable camphor-wood may also be utilised. The trees being set 15 ft. apart, the stumps may be allowed to produce shoots until young trees have been raised and are sufficiently advanced to take the place of the old ones, when the stumps are uprooted and subjected to distillation. According to Maisch, the camphor produced is of good quality, but the statement that it is stronger than Japanese camphor is erroneous. It is gratifying to know that a new source of camphor will thus be available, for at present it does not seem to be possible to get enough of it from China and Japan.

*The Apocynaceæ.* Chemist and Druggist, November 29, 1890, 750.

THE apocynaceæ have, during the last few years, yielded several new drugs to *matéria medica*, and naturally, therefore, physiologists give particular attention to this class of plants. But it must not be supposed that they are all poisons; some of them have comparatively mild physiological action. Quebracho, for instance, is used as a febrifuge and anti-asthmatic; *Alstonia scholaris* is the source of Dita bark employed in India, and recently in the United States, in chronic diarrhoea and dysentery. Some members of the order, such as *Alstonia* and *Vakea gumifera*, yield a substance resembling india-rubber, and used as such (especially the product of the latter-named plant) in France.

*Apocynum cannabinum*, which is a powerful emetic and purgative, takes precedence of the above-mentioned in point of intensity of therapeutical activity. Then we have *Wrightia antidysenterica*, containing "wrightine," identical with conessine; this has repute and is used as a cardiac. Further in the first rank there is *Nerium oleander*, and, of

course, *Strophanthus*, so well known in some respects, and yet—at least, as regards its botany and source—so little known.

Although only three years have passed since *strophanthus* became available for use in medicine, a number of apocynaceæ have been investigated, their active principles separated and physiologically examined. After *Strophanthus*, *Carissa schimperi* was worked on and found to contain ouabaine, which is now prepared from the so-called glabrous *strophanthus* seeds, and believed by some pharmacologists to be identical with strophanthidine.

Boehm then investigated *Adenium Boehmianum*, an ever-green shrub belonging to the same order; the milky juice of this plant is also used by the natives as an arrow-poison. Echujin was separated by Boehm and found to be a powerful heart-poison.

Recently a further link in the chain of apocynaceous drugs has been forged by Professor Kobert and his pupil, Minkiewicz, in the complete chemical and physiological investigation of *Urechites suberecta*. This West Indian climbing plant was first known under the name of *Echites neriantra*; the generic term (from the Greek *echis*=viper) was employed by Pliny for a twining plant. The plant, now termed as stated, *Urechites suberecta*, is known in South America, in the West Indies, and in Jamaica; a living plant was shown by Mr. Holmes at the recent *conferentia* of the Pharmaceutical Society, the exhibitor observing, with reference to its poisonous properties, that a native of Java would not accompany one of his brethren if he knew that the latter had some *urechites* leaves in his pocket.

The leaves of *Urechites suberecta* are dark green, oval somewhat acute, entire, petiolate, and opposite; the plant has large yellow flowers, the so-called savana flowers. The latter and all green parts of the plant have an intensely bitter taste, and the dry powder of the leaves affects the mucous membrane of the nose, producing sneezing. The milky juice of the plant is said to be used by some Indians to prepare the woorara poison.

A recent chemical examination by Kobert resulted in the separation of a glucoside, soluble in benzene, and of urechitic acid, insoluble in benzene, but taken up by amyl alcohol. This distinction was the basis of a process for separating the two principles. Urechitic acid, which seems to be a resin, is yielded in the proportion of 2 per cent., but only  $\frac{1}{2}$  per cent. of the glucoside is obtainable.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

### PATENTS.

*An Improved Trough for Use in Washing Photographic Prints, Photographic Negatives, and the like.* P. Ellis, Wallington. Eng. Pat. 16,492, October 18, 1889. 8d.

A box of thin sheet metal is provided with a vertical rack for holding plates and a horizontal rack for holding prints, and is constructed of such a shape that when filled with water to a certain height, it tips over and empties itself, returning to its original position under the supply tap as soon as the greater part of its contents has been discharged. —C. H. B.

*A New or Improved Compound or Developer for Use in Photography.* B. Jumeaux, Manchester. Eng. Pat. 18,547, November 20, 1889. 4d.

Eikonogen, potassium chlorate, and sodium sulphite are slightly damped, mixed together and dissolved in hot water. The proportions given are eikonogen 2 drms., potassium chlorate  $1\frac{1}{2}$  drms., sodium sulphite 3 oz., water 20 oz., but

other proportions may be used, and other suitable potassium salts, such as the bromide, or sodium salts, such as the carbonate may also be added.

It is claimed that this developer is self-contained, requires no accelerator, develops negatives, lantern slides, opals, and paper prints, and, except after repeated use, does not stain either the photograph or the hands of the operator.

—C. H. B.

*An Improved Process or Means for giving a Matt Surface to Albumenised Silver, Paper, Photographs, or Prints.*  
J. B. Payne, London. From C. F. Binns, Wellington, New Zealand. Eng. Pat. 19,205, November 29, 1889. 6d.

A SHEET of finely ground glass, or a slab of marble, slate, or other material with a finely ground or matt surface is coated with a waxing compound prepared by dissolving 3 drms. of best bees' wax and 3 drms. of yellow resin in 1 pint of turpentine. In warm weather a greater proportion of resin must be used. The waxing compound is poured on the plate, which is then carefully polished, and allowed to dry for 24 hours in a place free from dust. The plate is next coated with a gelatin-zinc compound made by dissolving 2 oz. of Coigners or some similar gelatin in 20 oz. of water and adding zinc oxide and glycerol in proportion depending on the character of the zinc oxide. With the ordinary commercial zinc oxide, 1 oz. of glycerol and  $\frac{1}{2}$  oz. of the oxide are good proportions, but 50 grs. of Hubbard's oxide and  $\frac{1}{4}$  oz. of glycerol will be found sufficient. The glycerol and zinc oxide are carefully mixed in a mortar and are then incorporated with the gelatin solution, which is afterwards strained.

The waxed and polished plates are coated with the gelatin-zinc compound at a temperature not exceeding 130° F. if the plate is dipped into the solution, or 108° F. if the solution is poured on the plate. As soon as the gelatin has set the plate is placed face upwards on a stand, flooded with cold water, and carefully brushed with a camel's hair brush. It is again flooded with water, and the silver print, previously soaked in water, is placed upon it face downwards and carefully squeezed into perfect contact, the superfluous moisture being afterwards removed with a sponge. A backing of paper or other suitable material may be applied to the print, and the plate is set up to dry, which requires from 18 to 24 hours in summer weather. When perfectly dry, the print is removed by inserting a penknife between it and the plate. Gelatin alone may be used in place of the gelatin-zinc compound, but the zinc oxide gives a desirable "bloom" to the picture.—C. H. B.

## XXII.—EXPLOSIVES, MATCHES, Etc.

*On the Absorption of Carbonic Oxide by the Earth.*  
Berthelot. *Compt. Rend.* 111, 469—471.

DOUBTLESS carbonic oxide is the cause of many deaths after coal-mine explosions. It is produced in considerable quantities from certain explosives which are now extensively used, such as mellinite, gun-cotton, &c., and the author has investigated the question whether this gas (CO) is more readily retained by the soil than ordinary atmospheric air. Contrary to the assumption of certain witnesses this is not the case, the earth being able to absorb exactly the same amount of carbonic oxide as it does of air.—A. R.

## XXIII.—ANALYTICAL CHEMISTRY.

### APPARATUS.

*Metallic "Scrated Tubes."* H. Schiff. *Chem. Zeit.* 14, 1407—1408.

ENAMELLED iron tubes, closed by a screw-plug, have been recommended, for laboratory purposes, to take the place of sealed glass tubes. In practice it is found that the enamel soon cracks off, and that the thread of the screw-plug rapidly wears out. The author has devised an apparatus in which these difficulties are avoided. It consists of a cast-bronze tube, open at one end, having an internal diameter of 36 mm. and a length of 330 mm. The walls of the tube are 8 mm. and the bottom 10 mm. in thickness. The lid—10 mm. in thickness—fits on the open end of the tube, and is held down by a screw clamp, the screw of which works in a slight depression in the centre of the lid. The jaws of the clamp are slipped under the projecting rim (14 mm. wide) of the tube. The tube is hermetically sealed by placing between the top of the tube and its lid, a disc of paper soaked in water or other suitable liquid. A glass tube sealed at one end and contracted at the other to a diameter of 12—14 mm., fits tightly inside the bronze digester, and is of such length that its open end reaches within 3—4 mm. of the lid when screwed down, the intervening space being filled by a disc of asbestos fitting moderately tight.

According to the author's experience, these tubes are but slightly attacked by the vapours of hydrochloric and hydrobromic acids, and of ammonia. Hydriodic acid gas attacks them to a greater extent; but operations with alkaline iodides may be safely carried on in such tubes. Sulphuretted hydrogen and ammonium sulphide strongly corrode the metal.

The author also describes a larger form of digester of  $1\frac{1}{2}$  litres capacity, similar in principle to the above.

—H. T. P.

### INORGANIC CHEMISTRY.

#### QUANTITATIVE.

*Experiments on the Quantitative Determination of Arsenic by Marsh's Method.* B. Kühn and O. Saeger. *Ber.* 23, 1798—1803.

POLENSKE showed that small quantities of arsenious acid can be almost completely converted into arseniuretted hydrogen, and this latter into metallic arsenic. But Polenske could not convert more than 0.005 grm. of arsenious acid into metallic arsenic. He now shows that larger quantities can also be converted into metallic arsenic. The experiments were made as follows:—150 grms. of pure granulated zinc were put into an Erlenmeyer flask of 400 cc. capacity, and the flask closed by a doubly-perforated india-rubber stopper. Through one perforation passes a funnel tube reaching nearly to the bottom of the flask, through the other an outlet tube, bent at right angles. The latter is connected with a wash-bottle, containing 50 cc. of a 5 per cent. lead acetate solution, to absorb sulphuretted hydrogen, if present. There is next arranged a system of drying tubes containing altogether at least 100 grms. of calcium chloride, and the last of these is connected with a combustion tube, open at both ends, 165 cm. in length and 10 to 12 mm. in diameter. The middle part of this tube rests in a combustion furnace 80 cm. long, both ends of 25 and 60 cm. length respectively are drawn out to a diameter of 5 mm. inside. The shorter end (of 25 cm.) is connected with the calcium chloride tubes, and the longer one with a silver nitrate solution of 3 per cent. strength. The furnace is heated by 24 Bunsen burners. Two burettes are placed above the funnel tube of the Erlenmeyer flask, one of them containing dilute sulphuric acid (1 part of sulphuric acid of 1.84 sp. gr. to 3 of water), and the other the liquid to be tested.

After the development of hydrogen has commenced, and after all air is expelled from the apparatus, the tube is heated to redness. If after half an hour's heating no arsenic mirror is obtained at the end of the tube, the reagents can be considered as pure. Then this last part of

the tube is also heated by three Bunsen burners, 15 cm. distant from each other, and the liquid to be tested is introduced from the burette into the Erlenmeyer flask drop by drop. Care must be taken that the liberation of hydrogen is always regular. Such regular liberation is effected by adding the sulphuric acid drop by drop (about 0.75 cc. per minute). After some minutes an arsenic mirror can be observed at that part of the tube away from the furnace. Should traces of mirrors appear also between the Bunsen burners placed there, or at the end of the tube, the hydrogen current is too strong, and must be diminished. After one hour all the liquid to be tested (25 cc.) will have been added, and the reaction with the course of operations are continued for two hours more, after which time the presence of arsenic is tested for by heating the tube just where it enters the furnace. If no arsenic mirror is observed the process is complete; in the other case, it has to be continued until no more arsenic is discoverable. When different small arsenic mirrors have been obtained, they are united to the large one, and the latter is cut out, dried, and weighed. Then the arsenic is dissolved in nitric acid, the empty tube weighed again, the difference indicating the amount of arsenic. When it is of importance to retain the arsenic mirror, the small tube is put into a larger combustion tube of about 50 cm. length, one end of which is drawn out. By heating in a current of hydrogen, the mirror is driven forwards to the larger tube, and the small tube is weighed when empty. Nos. I. and II. of the following tests were made with pure arsenious acid, obtained by sublimation. 25 cc. of the solution contained 0.1105 gm. of arsenious acid, equivalent to 0.0837 gm. of arsenic. Nos. III. and IV. were obtained by using potassium arsenate, which was converted into magnesium arsenate (pyro salt), and thus weighed.

From 25 cc. of potassium arsenate were obtained 0.1907 gm. of  $Mg_2As_2O_7$ , equivalent to 0.09227 gm. of As. The different tests gave the following results:—

Nos.	Arsenic employed.	Arsenic obtained.	Difference.
	Gm.	Gm.	Gm.
I.	0.0837	0.081	0.0027
II.	0.0837	0.0832	0.0005
III.	0.09227	0.0911	0.00117
IV.	0.09227	0.0907	0.00157

The authors have further found that arseniuretted hydrogen is decomposed by caustic potash, a fact not generally known. This was ascertained by introducing into the apparatus described, a tube containing dry caustic potash between the last of the calcium chloride tubes and the glass tube which was to be heated. The following results were thus obtained:—

Nos.	KHO used.	Arsenic employed.	Arsenic obtained.	Difference.
	Grms.	Gm.	Gm.	Gm.
I.	25	0.0837	0.0760	0.0077
II.	100	0.0837	0.0732	0.0105

In a subsequent test in which 225 grms. of caustic potash were used, 0.0573 gm. of the arsenic mirror obtained in the last experiment (No. II.) were converted into potassium arsenate and dissolved in 25 cc. of water. The result of the test was as follows:—

Arsenic employed.	Arsenic obtained.	Difference.
Gm. 0.0573	Gm. 0.0316	Gm. 0.0257

The presence of arsenic in the caustic potash tube was easily ascertained by the blowpipe reaction.—H. S.

*Quantitative Determination of Antimony by Marsh's Method.* A van. Bylet. Ber. **23**, 2968—2971.

The method is adapted to the estimation of small quantities of antimony in antimony alloys. The alloy is dissolved in mercury, and to the solution sodium amalgam is added to form the sodium antimonide. This is then decomposed by means of dilute sulphuric acid. The apparatus necessary consists of a three-necked Woulff's bottle, one neck of which is connected with a carbon dioxide generator; through the middle neck passes a feed tube, which should be as wide as is compatible with the size of the neck, and the third neck has attached to it a calcium chloride tube and a piece of glass combustion tubing. 0.5 gm. of the alloy is dissolved in 20 cc. of mercury by warming in a test tube to 60° C. After cooling the amalgam is introduced into the Woulff's bottle and the central tube is made to dip under its surface. Through one of the side necks 100 cc. of sulphuric acid (1 to 10) are added. The air is now displaced by carbon dioxide, the combustion tube heated to redness, and sodium amalgam (made by dissolving 5.5 grms. of sodium in 25 cc. of mercury) added drop by drop down the central tube. The apparatus is carefully shaken, care being taken that no possibility of an escape from the central tube shall occur. When the evolution of gas has ceased, carbon dioxide is passed for some time, and then the heating of the tube is discontinued. Evolution of gas lasts usually about an hour. After this the supernatant liquid is removed and the mercurial solution is poured off and dried by filter paper. After the apparatus has been thoroughly cleansed and dried, one half of the mercurial solution is re-introduced, with 75 cc. of sulphuric acid (1 to 10); to the other half 3 grms. of sodium are added and this mixture is then poured drop by drop through the central tube to the first half. The antimony from this second experiment is collected in a fresh tube; it should not exceed 1 per cent. of the total antimony. The weight of antimony is ascertained by weighing the tubes.

To prevent the possibility of a deposit of carbon near the heated portion of the tube, a current of hydrogen may be employed instead of carbon dioxide.—T. L. B.

*The Estimation of Metallic Zinc in Zinc-Dust.* W. Minor. Chem. Zeit. **14**, 1142.

DREWSEN (Zeits. Anal. Chem. **19**, 50; this Journal, 1887, 147) has described a method which depends on the fact that finely divided zinc reduces a solution of chromic acid without evolution of hydrogen. The following modification of his process will be found to give exact results:—

The following solutions are required:—About 30 grms. of pure potassium bichromate dissolved in water, and made up to 1 litre. A solution of ammonio-ferrous sulphate, made by dissolving 150 grms. of ferrous sulphate, and 75 grms. of ammonium sulphate in water, adding 100 cc. of strong sulphuric acid and diluting to 1 litre. The bichromate solution is standardised by means of chemically pure zinc dust, and since this is difficult to obtain, even in the case of the electrolytic product, it is advisable to prepare it by filing down a rod of the pure metal by means of a hard file. The iron in the zinc so obtained should be estimated, but it will probably not exceed a few thousandths per cent., and may consequently be neglected. The ferrous solution is compared with the bichromate, using potassium ferrieyanide as the indicator. 1 gm. of the pure zinc is then treated with 60 cc. of bichromate, diluted with water, and about 20 cc. of dilute sulphuric acid added drop by drop from a burette, the addition being so regulated as to occupy about 20 minutes. More acid is then added and the excess of bichromate estimated by the ferrous solution. For the analysis of a sample of zinc-dust, the operation is conducted in the same way, the metal present being in proportion to the amount of bichromate reduced, compared with the quantity used in standardising on the pure metal.

The figure so obtained includes the amount of cadmium and iron reckoned as zinc, and when, as is usually the case, the zinc-dust is to be used as a reducing agent, this is immaterial, those metals acting like the zinc. A method of



calculation is given with a suitable practical illustration, showing how the exact amount of zinc may be determined. (See also this Journal, 1890, 109, 417, 554, 769, and 968.)

—F. H. L.

*The Dry Assay of Tin Ores.* Part II. H. O. Hofman. Technology (quarterly), 3, 261—280.

THE author describes further test assays of black tin-stone by other methods (this Journal, 1890, 899). Winkler's method gave results averaging 65·96 per cent. or 1·88 per cent. too low, the figures disagreeing amongst themselves to over 3 per cent. The process consists of reducing together 5 grms. each of cassiterite and cupric oxide with 15 grms. of black flux, 1·25 grms. of borax, and a little salt as a cover. A similar experiment is conducted without the cassiterite, and the copper obtained in the one case deducted from the alloy in the other gives the amount of tin. The following modifications of this method were tried: increasing the amount of charcoal; using chalk-lined crucibles; omitting the salt and increasing the potassium carbonate; and Ricketts' modification of adding argol; but they all gave worse, and in some instances very variable, results.

The iron method—reduction with hematite, charcoal, and potassium cyanide or fluorspar—gave unsatisfactory results. The same may be said when a mixture of ferrocyanide and cyanide was used, and also when any of the Cornish methods were employed. So far, with fairly pure cassiterite, only two methods have proved satisfactory, viz., the German and the pure cyanide methods. The author now attempted to prove which of these two methods gives the best results when the cassiterite is mixed with those minerals likely to be naturally associated with it. The minerals used were quartz, felspar, mica, tourmaline, garnet, and columbite. Mixtures of the purified cassiterite ore with varying proportions of each of these minerals were assayed by both methods, the percentage of tin being calculated on the cassiterite ore present. The charges were so regulated that the flux in the German assay was always equal to three times the quantity of ore plus the mineral, and in the cyanide assay to six times the quantity. In the latter case four parts of cyanide were mixed with the ore, one part was used to coat the crucible bottom, and one part as a cover. In all cases the cyanide method gave more concordant and less prejudicially affected results than the other. The results of the German assays fluctuated greatly as the amount of mineral present increased. Thus, in the case of quartz and albite the results were lower as the quantity of mineral was increased, the respective figures being, with 37·5 per cent. of each mineral, 60·7 and 60·2 per cent.; the corresponding cyanide figures being 63·90 and 65·30 per cent. (the real percentage of tin was 67·84). The tin buttons, moreover, in the German assay with albite contained iron, while those by the cyanide process were free, the temperature of fusion being too low to cause decomposition of the albite.

With muscovite, tourmaline, garnet, and columbite, the results with the German method were very unreliable, being sometimes too high and very ferruginous, and sometimes too low, varying from 82 per cent. with 50 per cent. of garnet to 46 per cent. with 44 per cent. of tourmaline. The cyanide method gave fairly good results in all the above cases, very little iron getting into the buttons except in the case of garnet, when the result was 69·89 per cent., with 37·5 per cent. of mineral present. With the same quantity of muscovite, tourmaline, and columbite the assays were respectively 63·3, 64·7, and 66·0 per cent. Consequently where anything like a true result is required the cyanide assay is by far the most to be relied upon and should be employed in cases of buying and selling, although in concentration works, where comparative results and large numbers of assays are required, the question of cost must be considered, and the cheaper German method might satisfactorily be adopted.

The author emphasises the importance of properly preparing the ore for assay, and states that the ordinary method of grinding the whole sample to a uniform size is not suited to the tin ores of the Black Hills, chiefly on

account of the preponderance of mica. Hence a preliminary sizing is advantageous. The ore while being pulverised is screened through a series of sieves, Nos. 20, 40, 60, and 80, the different sized screenings being then washed separately and the concentrates afterwards mixed, dried, passed through a 60-mesh sieve and again washed. This method gives purer concentrates and necessitates less loss of cassiterite as the material has been washed without excessive grinding, which in the present process reduces the brittle cassiterite to a slime, while some of the harder associated minerals are only converted into sands. The subsequent treatment of the concentrates mentioned in the first part of the paper is recommended, as the roasting renders the cassiterite more friable and more readily reducible, the assays in some cases showing a difference of 0·5 per cent. between the raw and roasted ores in favour of the latter.

—A. W.

*The Estimation of Nitrates in Manures by Reduction of the Nitric Acid to Ammonia.* T. F. Schmitt. Chem. Zeit. 14, 1410—1411.

THE reduction of nitrates to ammonia is rapidly effected in the following manner: 40 cc. of glacial acetic acid are placed in an Erlenmeyer flask of 750 cc. capacity, and 15 grms. of a mixture of equal parts of zinc dust and iron filings are added. 50 cc. of the nitrate solution, containing 0·5 gm. substance are next added, and the mixture is thoroughly shaken. The evolution of hydrogen, which at once commences, continues for about 15 minutes, when another 15 grms. of the metallic mixture must be added. At this stage the mixture sometimes becomes pasty; if so, an addition of 30 cc. of water must be made. After 30—40 minutes the reduction is complete. The mixture is rendered alkaline by adding 200 cc. of a sodium hydrate solution of 1·25 sp. gr., and the ammonia is distilled off in the usual way. The test analyses are exceedingly satisfactory. This method probably might be substituted for Jodlbauer's modification of Kjeldahl's process, the substance under examination being first reduced and then boiled with strong sulphuric acid.—H. T. P.

*The Division of Sulphuretted Hydrogen between the Metals of two Soluble Salts.* G. Chesneau. Compt. Rend. 111, 269—271.

WHEN sulphuretted hydrogen is added to a mixture of the nitrates of lead and copper in molecular proportions, in insufficient quantity to precipitate the whole of the two metals, the two metals are precipitated approximately in the ratio represented by the heats of formation of their monosulphides. This ratio decreases with an increase in the number of equivalents of hydrogen sulphide present; it also varies with the length of time the precipitate is allowed to stand. The ratio Cu:Pb after immediate precipitation was found in two cases to be 1·74 and 2·20 respectively; this decreased a little at first, being 1·61 and 1·88 after allowing the precipitate to stand for 5 minutes before filtering, but after 15 minutes standing in the first case and after 10 minutes in the second, the ratio began to increase again, and after three days had reached 20·20, and after 38 days 23·10. It appears from this that the nitric acid liberated by the sulphuretted hydrogen acts gradually upon the sulphides and redissolves them.

—C. A. K.

*Direct Determination of Total Acidity in Aluminium Salts.* H. Heidenhain. Pharm. Rundschau, 8, 189.

AS is well known alumina is not precipitated by alkalis in presence of tartrates. The question is, whether in such solutions the alumina still plays the part of a base, or whether it is indifferent, or acts as an acid. By using phenolphthalein as indicator it was found that alumina in presence of small quantities of a tartrate has a weak alkaline reaction, whilst in presence of a comparatively large quantity of a tartrate it was almost without action, and that further,



the alkaline character was more noticeable in the hot than in the cold. The following method thus serves for determination of total acidity in aluminium salts: The salt is dissolved in water, and for every part of  $Al_2O_3$ , 100–200 times the quantity of sodium potassium tartrate is added; after addition of phenolphthalein solution so much of a standard solution is added that a portion of the acid remains unsaturated. The whole is boiled to drive off any carbon dioxide, and after cooling titration is completed. The end reaction is not quite sharp, but is sufficiently so, since the error is not over 0.1–0.2 per cent. of the volume of standard solution used.—T. L. B.

### ORGANIC CHEMISTRY.—QUALITATIVE.

*Detection of Impurities in Alcohol.* E. Mohler. Compt. Rend, **111**, 187–190.

A NUMBER of reagents have been proposed at various times for testing the purity of alcohol; the action of such reagents has been examined in the present memoir.

Solutions of alcohol, 50°, were made up containing 0.1 per cent. of the various impurities contained in alcohol, and on such solutions the action of the reagents was tried.

**Sulphuric Acid.**—The maximum proportion of acid that should be used is 10 cc. of acid to 10 cc. of alcohol. With larger quantities, even pure alcohol becomes coloured; and with smaller quantities the delicacy of the reaction diminishes very rapidly. The solutions containing butyl aldehyde, acetone, propyl, isopropyl, normal butyl and methyl alcohols, ethyl acetate, and many other substances, showed no colouration.

**Rosaniline Bisulphite.**—The relative proportion of sulphuric acid to sodium bisulphite in the reagent exercises a great influence on the delicacy of the reaction. With little acid the reagent is coloured by pure alcohol, whilst if much acid be present, 0.1 per cent. of ethyl aldehyde produces no colouration. If the reagent be made up as follows, it is not affected by pure alcohol, and 0.001 per cent. of aldehyde may be detected by its means:—

	Cc.
Solution of magenta, $\frac{1}{1000}$ .....	30
Bisulphite of soda of 34° B.....	20
Sulphuric acid .....	3
Distilled water .....	200

The reagent should be used immediately after making up, and in the proportion of 4 cc. to 10 cc. of alcohol.

**Aniline Acetate.**—This is the reagent for furfural. The colouration produced is not largely influenced by the relation between the acetic acid and the aniline. The proportion of 10 drops of aniline to 2 cc. of glacial acetic acid gives the best results; the maximum of colouration is attained after about half an hour. The reaction is extremely delicate.

**Potassium Permanganate.**—In acid solution decolourisation was effected by paraldehyde, isobutyl alcohol, and isobutyl aldehyde. The reduction is in proportion to the amount of aldehyde present, and is independent of the concentration of the alcohol or the degree of acidity of the liquid.—T. L. B.

*Reactions of Salts of the Alkaloids.* A. Colson. Compt. Rend, **111**, 266–268.

PIPERIDINE precipitates the soluble salts of calcium, although the heat of formation of piperidine hydrochloride in aqueous solution is 13 calories, whilst that of calcium chloride is 14 calories. Similarly aniline and hydrochloric acid combine in aqueous solution with the evolution of 7.4 calories, whilst pyridine under the same conditions evolves 5.2 calories. The heats of formation of aniline and pyridine hydrochlorides (taking the base as liquid, the hydrochloric acid as gas, and the resulting salt as solid) are respectively 27.3 and 24.9 calories, but still pyridine

decomposes aniline hydrochloride in solution. The toluidines and other aniline bases furnish similar instances of such endothermic decompositions.

These apparently abnormal reactions are quite in accordance with Berthollet's laws, their occurrence being due to the differences in the heats of solution of the various salts referred to.

Piperidine hydrochloride absorbs 0.98–1.02 calories; aniline hydrochloride absorbs 2.66–2.73 calories; whilst pyridine hydrochloride evolves 0.1–0.15 calories, when dissolved in water.—C. A. K.

*The Presence of Furfuraldehyde (Furfural) in Commercial Alcohols.* L. Lindet. Compt. Rend. **111**, 236–238.

FURFURALDEHYDE forms an accidental impurity in commercial alcohols, and is not a normal constituent of the same.

Whenever the alcohol has been distilled over a naked fire or whenever the wort has been treated with acid, furfuraldehyde is found to be present, but when this latter condition has not been fulfilled, or when the alcohol has been distilled by steam, there is no furfuraldehyde formed. A sample of alcohol distilled over a naked flame was found to contain 0.2 cc. per litre of furfuraldehyde and various liqueurs prepared under like conditions from 0.02–0.40 cc. per litre, of this impurity. Its formation is probably to be traced to the decomposition of vegetable debris contained in suspension in the liquid during the distillation.

Where the saccharification of the cereals had been effected by means of mineral acids, 0.06–0.10 cc. of furfuraldehyde per litre was found in the resulting alcohols; the spirit in these cases was distilled by steam. In this case the impurity was shown by direct experiment to result from the action of the acid on the entire of the grain.

A small quantity of furfuraldehyde (0.01 cc. per litre of alcohol) was also found when the saccharification had been effected by means of diastase; its production in this case is traced to a preliminary lactic fermentation, the lactic acid acting in a similar manner though to a less extent than the mineral acids. Spirit obtained from beetroot, potatoes, and Jerusalem artichokes prepared under conditions so as not to produce furfuraldehyde, as indicated above, were found not to contain a trace of the impurity. Four samples of spirit obtained from molasses were found to contain from 0.04–0.05 cc. of furfuraldehyde per litre of alcohol; in this case the latter does not result from the action of mineral acids upon the sugar, but by the employment of a wort for the fermentation of the molasses which had been prepared by means of mineral acids.

In these experiments the acetate of aniline reaction with furfuraldehyde was employed both for the qualitative and quantitative determination of this substance (see also this Journal, 1889, 734).—C. A. K.

*A Characteristic Reaction of Cocaine.* F. da Silva. Compt. Rend. **111**, 348–349.

A SMALL portion of cocaine or of one of its salts is treated in the solid state with a few drops of fuming nitric acid.

After evaporation to dryness one or two drops of a concentrated alcoholic solution of potash are added, and the whole is well stirred. Presence of cocaine is evidenced by a distinctive odour reminding of peppermint. The test is said to be very delicate.—T. L. B.

*Examination of Olein for Resin.* G. Meyer. Chem. Zeit. **14**, 1202.

THE resin may best be qualitatively recognised by saponifying and salting out; resin soap is incompletely precipitated, or not precipitated at all. The amount of resin may be approximately estimated by a determination of the specific gravity, by polarisation, and by means of the acid value determination. Such means as these may also be adopted for recognising the presence of resin in soap.—T. L. B.

*Examination of Fats, Meats, &c.* G. Meyer. Chem. Zeit. **14**, 1202.

HORSE fat absorbs much more bromine than beef-fat and lard. Determination of bromine absorption may therefore lead in many cases to detection of the use of horse-fat in preparations.—T. L. B.

*Distinction between Genuine and Imitation Parchment Paper.* E. Muth. Dingl. Polyt. J. **277**, 360–361.

STEEPED in strong lime water, the material retains its white colour if made of cotton fibre, but turns yellow or brownish yellow if made of sulphite cellulose. Since mechanical wood-pulp also shows the latter change of colour, its absence should first be ascertained by the aniline sulphate or phloroglucinol reaction. The test is thus only applicable when mechanical wood-pulp is absent.—H. S.

## ORGANIC CHEMISTRY.—QUANTITATIVE.

*Percentage of Cellulose in Wood.* Papier Zeit.; and Chem. Trade J. 1890, **7**, 335.

THE method of analysis employed is as follows:—The wood under investigation is broken or cut into small pieces and then ground by rubbing in a mill. From 3 to 5 grms. of the ground wood are weighed off, dried at 100° C. till the weight is constant, and the percentage of water calculated from the loss in weight. The dry ground wood is then boiled in water, and afterwards digested in ether, the residue separated from the ethereal solution and dried. It is now digested with 3 to 5 grms. of caustic alkali, dissolved in 50 cc. of water in a small pressure flask placed in a paraffin bath to 130°–150° C. (preferably 135° C.), for two hours, or in some cases longer. If properly screwed up the small flask stands the pressure completely. It is allowed to cool in the paraffin bath, then opened, water added to its contents, and the brown lye separated from the insoluble matter by filtration and subsequent washing with water. The wood, or more properly speaking the cellulose powder, is now heated with an aqueous solution of bromine, and washed. Instead of this, an alkaline solution of bromine may also be used. The bromine acts upon the ligneous residue in the same way as chlorine. The cellulose is heated with ammonia water, again filtered and washed. If its colour be changed to brown on the addition of the ammonia, incrusting materials are still present, and the cellulose must again be repeatedly treated with bromine water and ammonia till it is pure white, and the ammoniacal filtrate is perfectly clear and colourless. In this way completely pure white cellulose is obtained, which is washed many times with water and with alcohol, and finally dried at 100° C. and weighed. By this method, in the preliminary treatment with water, colouring matters and mineral substances are removed. In the treatment with potash lye the greater part of the incrusting substances are removed and the bulk of the colouring matter, and finally, by first oxidising the incrusting substances remaining with the pure cellulose, with bromine, these are removed in the soluble form by ammonia.

*Prunus Padus (Bird Cherry Tree)*—The charcoal made from this tree is used for the manufacture of gunpowder. The raw wood is coloured yellowish and reddish. When boiled in water it assumes a grey colour, whilst the aqueous solution is reddish. The ethereal extract was yellow, the alkaline fluid red, while the remaining cellulose still retained a considerable reddish hue. Bromine water and ammonia at first turned the cellulose a light yellow, finally, however, a pure white. The yield on the air-dried wood was 40.98 per cent. of cellulose.

*Peruvia Wood.*—Dark red chips. The aqueous solution was red, the alkaline lye black red. With bromine water the cellulose appeared yellowish white, with ammonia colourless. On the air-dried wood it yielded 39.63 per cent. of cellulose.

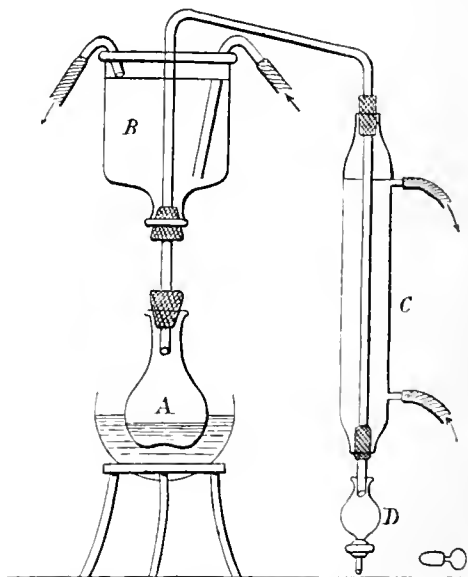
Recent investigations seem to show that the sulphite method of estimating the amount of cellulose in wood is more correct than the caustic alkali methods. By digesting wood in caustic alkalis at high temperatures some of the cellulose ( $C_6H_{10}O_5$ ) passes into solution, or is dissolved, and is, of course, lost in the determination, and consequently a low result is obtained. Instead of using caustic alkalis, bisulphite of soda or potash may be employed for all those kinds of wood that are rendered soft by the bisulphite liquors, and whose incrusting and resinous materials are completely dissolved by them. On the other hand for oak and similar hard timbers the bisulphite process is useless.

*Ebony.*—The quantitative determination of the cellulose in this wood was impossible. It is, however, interesting to know that by the method just described, the deep black ebony wood yielded a pure white cellulose.

*A Quantitative Reaction for Lignin.* R. Benedikt and M. Bamberger. Monatsh. **11**, 260–267.

THE authors describe a number of experiments by which they have determined the methyl numbers of several kinds of wood, the experiments being a continuation of those on the ethereal oils (Chem. Zeit. **13**, 872 and 1087; this Journal, 1890, 735 and 925) and resins (Monatsh. **11**, 84; this Journal, 1890, 659).

Forty grms. of the wood shavings dried at 100° were put into the flask A together with 500 cc. of hydriodic acid of specific gravity 1.7, and containing 8 per cent. of acetic anhydride. Heat was applied and the distillation of the methyl iodide produced was regulated by adjusting the temperature of the water in B. The distillate which collected in D proved



be nearly pure methyl iodide; it amounted to 4 grms., i.e., 10 per cent. of the weight of the dry wood, but this does not represent the whole of the methyl iodide produced, as the apparatus remained full of vapour and part was no doubt lost. In the methoxyl determination (this Journal, 1889, 735) the methyl number obtained was 26.2, corresponding to 24.8 per cent. of methyl iodide.

Pure cellulose yielded no methyl iodide when boiled with hydriodic acid. On the other hand various woods which were boiled with water, dried, and then extracted for days first with alcohol and then with ether, were found to give the same methyl number as the same woods when not so treated. From these results it is concluded that *lignin* is the constituent of the wood to which the methyl number is to be ascribed.

Assuming the correctness of Schulze's determination of the percentage of lignin in oak, namely 54.4 (Chem. Centr. 1857, 321), the authors are able to calculate from the methyl number of the same wood, namely, 28.6, that lignin should have the methyl number 52.9, and from this they also calculate the percentage of lignin in various woods:—

	Methyl Number.	Percentage Lignin.	
		Benedikt and Bamberger.	Schulze.
Walnut shell .....	37.4	70.0	65.9
Holm-oak .....	28.6	54.1	54.1
Alder.....	28.9	54.6	52.0
White beech.....	26.4	49.9	51.6
Aescia.....	24.2	45.9	47.0
Pine.....	21.3	40.3	42.0

The paper concludes with a table showing the methyl numbers obtained with a number of different woods and other substances. The woods examined gave methyl numbers varying from 20 to 31. The older wood near the centre of a stem is shown to contain more lignin than the later rings, and the wood of a branch is richer in lignin than that of the stem. A comparison of the results obtained from lignite, peat and coal shows that the first has a methyl number corresponding with the woods, namely, 24.4, whilst that given by peat is 2.7, coal, on the other hand, yielding no methyl iodide when boiled with hydriodic acid.

—A. K. M.

*Determination of Tannin in Barks, &c.* G. Meyer. Chem. Zeit. 14, 1202.

Precipitation with acetate of copper gives good results, if the precipitation be performed in the hot, and if filtration and washing with hot water follow immediately; the precipitate is dried at 110°, weighed and ashed. From the total precipitate four-fifths of the weight of the copper oxide is deducted. Total tannin is thus ascertained. All bark extracts, however, contain both tannic acid and gallic acid with hydration products of the latter, and the value of the material is judged from the tannin matter precipitable by gelatin. This precipitable tannin may be determined by the method of Davy and Müller as follows:—The extract is prepared by digesting 20 grms. of bark once with 200 cc. of hot water, and five times with 160 cc. of hot water in a water-bath, each time for a quarter of an hour. The whole decoction is filtered and the filtrate is diluted to 1 litre. A solution containing 1 gm. of tannin per 500 cc. is required, and also a gelatin solution containing 10 grms. of gelatin, 5 grms. of alum, 10 grms. of boric acid per litre, which keeps better if shaken up with about 20 cc. of ether and a little chloroform.

100 cc. of the bark extract are taken as soon as possible after preparation, and to them are added about 5 grms. of sodium chloride; the mixture is gently warmed in a water-bath at about 40°, and gradually 50 cc. of the solution of gelatin are added. The whole is filtered, washed with water at 50°–60°, and dried at 110°. 100 cc. of tannin solution are treated in a similar manner. By comparing the weights of the two precipitates obtained, and knowing the strength of the prepared tannin solution, of course the strength of the extract is directly found. The method showed in medium oak bark 8–10 per cent. of tannin substance. The probable error is given at less than 5 per cent. of the value found. If the solution be allowed to stand the error becomes great.—T. L. B.

*New Method of Determining Tannin by Means of Iodine.* A. Moullade. J. Pharm. Chim. [5], 22, 153.

This is a modification of the method of Jean, whereby the tannin is determined by iodine in presence of sodium

bicarbonate. Instead of starch paste, carbon bisulphide is used as indicator. The iodine solution contains 5.2 grms. of iodine and 7.6 grms. of potassium iodide per litre; the sodium bicarbonate solution is 1 to 10. To determine the tannin value of the iodine solution, 10 cc. of an aqueous tannin solution (1 to 1000) are introduced into a stoppered bottle, then 20 cc. of the sodium bicarbonate solution and about 10 cc. of water, with also 2–3 cc. of carbon bisulphide. The iodine solution is run in from a burette, the flask being kept well shaken. Suppose 10.5 cc. of iodine solution have been run in before blue colouration of the carbon bisulphide manifested itself, a fresh experiment is then performed, running in this time 10 cc. of iodine solution all at once, shaking well; if no colouration appears, another trial should be made, running in this time 10.3 cc. If now blue colouration appear, a fourth trial must be made, this time running in 10.2 cc. of iodine solution. Supposing now the carbon bisulphide to remain colourless 10.3 is taken as the correct equivalent of 10 cc. of tannin solution = 0.01 gm. of tannin. In examining unknown tannin solutions with the iodine solution thus prepared, exactly the same plan is adopted; the amount of tannin in the solutions under examination should not exceed 1–1.5 gm. per litre, otherwise the amount of bicarbonate must be increased. To 10 cc. of solution 30 cc. of bicarbonate solution are added, and titration is immediately proceeded with in spite of any turbidity, which may be brought about by the bicarbonate. To ascertain the true quantity of tannin in all cases, when substances behaving in the same way as tannin are present, it is necessary to make two experiments, in one of which the tannic acid is precipitated by gelatin; the difference between the two titrations is the iodine corresponding to the tannin present.—T. L. B.

*Analysis of Butter and Margarin.* C. Violette. Compt. Rend. 111, 345–347.

By means of a current of steam the volatile fatty acids are distilled from about 50 grms. of butter, saponified by aqueous potash; not less than 10 litres of distillate are collected. By titration then the butyric and caproic acids may be ascertained; the solid volatile acids are washed, dried in vacuo and melted, and are weighed, as are likewise the non-volatile acids. The equivalents of these groups may be determined and calculated to the glycerides, so that in the end the composition of the butter is arrived at. To ascertain the respective amounts of butyric and caproic acid, suppose A to represent the quantity of butyric acid equivalent to the volatile acids, then the true quantities, B and C, of butyric and caproic acid respectively are given by the following:—

$$B = A \times 0.68469$$

$$C = A \times 0.41565$$

A number of analyses are given of samples ranging from good butter to margarin.—T. L. B.

*Examination of Gantter's Method for Estimating Tannin.* Von Schroeder and J. Paessler. Dingl. Polyt. J. 277, 361–377.

GANTTER'S new volumetric method (see Zeits. f. angew. Chem. 1889, 377–380; this Journal, 1889, 1015) is to some extent based upon the well-known method of Loewenthal (see this Journal, 1882, 509–510; 1884, 82, 524–526; 1885, 263–266; 1886, 79, 210; 1887, 51, 94, 388; 1888, 593, 647, 779; 1890, 260, 607), since in both methods the tannin is oxidised by potassium permanganate. But whilst in Loewenthal's method only a very incomplete oxidation of tannin takes place, and very dilute solutions of the substances to be tested are used, Gantter uses more concentrated solutions and employs heat. He claims for his method a complete oxidation of the tannin, and consequently greater accuracy. Moreover, Gantter's results with oak bark are almost identical with the estimations of tannin obtained by the indirect gravimetric method, whilst it is well known that Loewenthal's method gives only relative results, from

which the actual quantities have to be found by calculation. It is evident that the method would be of much higher utility than that of Loewenthal, if for all tanning materials the results were not only more uniform, but also in direct accord with the gravimetric method. But the authors remark at the outset that Gantter's method is to be preferred only to a limited extent to that of Loewenthal, and that it does not possess all the expected advantages.

To become completely oxidised to carbon dioxide and water, 1 part of tannin requires 1.71 parts of potassium permanganate (this Journal, 1890, 261). But in working with Loewenthal's process according to the method proposed by the commission for fixing upon a uniform method of tannin estimation (see Bericht der Commission, Cassel, 1885; this Journal, 1886, 79) only 0.85 parts of potassium permanganate are used for the incomplete oxidation of 1 part of tannin (until the colouration of indigo disappears). Although in Gantter's method the oxidation of tannin is carried much further, yet it is not complete, for 3.988 parts of potassium permanganate are in this case necessary for the oxidation of 1 part of tannin. It is clear from this that the oxidation in the case of Loewenthal's method is very incomplete, but it has been proved by many experiments that the method is nevertheless accurate, if the details be carefully followed. Gantter's idea in the use of heat and concentrated solutions, implies errors which are skilfully avoided by Loewenthal, for the great dilution of the liquid to be tested by the latter method is based on the fact that tanning substances are much more easily destroyed by potassium permanganate than many other organic substances which are often found associated with them. Soluble carbohydrates and certain organic acids have little or no influence on the accuracy of Loewenthal's test, and it is of no consequence whether they are absorbed along with tannin by hide powder or not. But in Gantter's method all such substances are more or less oxidised together with tannin by the permanganate solution, and errors in this respect are therefore unavoidable. Thus, it becomes clear that in all cases where such non-tanning substances are absorbed by hide powder along with the tannin, Gantter's method will not be applicable. The authors have found that Gantter's statement, that 3.988 parts of potassium permanganate are necessary for the oxidation of one part of tannin, is not correct under all circumstances, and this is quite natural, since the oxidation is not a complete one. Gantter has not defined the strength of the dilute sulphuric acid which he used in his tests, and it is probable that changes in the proportion of the acid will produce variations in the degree of oxidation. The authors have found that it is necessary to keep constant the proportions of tannin and sulphuric acid for accurate work. They propose to use 0.50 cc. of the above-mentioned dilute sulphuric acid for every milligramme of tannin. The results thus obtained were very satisfactory.

The total average of potassium permanganate necessary for the oxidation of 1 part of tannin is 3.999 parts, a figure which does not differ much from that recommended by Gantter. The following method of procedure is therefore recommended by the authors in employing Gantter's method:—A tannin solution of 1:500 is to be used. An equal quantity of dilute sulphuric acid (1:5) is to be added to from 10 to 25 cc. of the tannin solution, and the resulting liquid is titrated with potassium permanganate (4 grms. in 1,000 cc.), 1 cc. of which is equivalent to 1 mgrm. of tannin. The results prove that Gantter's method is superior to Loewenthal's for the volumetric determination of tannin, but in making experiments with different tanning materials, the oxidations were by no means so uniform as with tannin. If the details of the tests were not always strictly uniform (e.g., duration of times of boiling, of titration, &c.), the results varied considerably. Procter has made the same observation (see this Journal, 1890, 261). After having ascertained this, the authors successfully tried to make the method more accurate by carefully working uniformly in all the tests. At the close of the reaction a brown precipitate of manganese hydrate (compare this Journal, 1889, 1015) is formed, and since it is essential to boil the solution always the same length of time, the authors add the permanganate solution in single

cubic centimetres at a time, and boil in each case not longer than one minute. The test is finished at the moment when, after a minute's boiling, a thick precipitate of manganese hydrate has been formed. It is not difficult to ascertain this point. The determinations were made in the following way:—The liquid to be tested, mixed with dilute sulphuric acid as above described, is introduced into a flask of about 350 cc. capacity, and heated to the boiling point. Then potassium permanganate is added in single cc. and the mixture shaken for about 5 seconds after each addition. As soon as the red colouration no longer disappears when the liquid is shaken as described, the contents of the flask are heated once more to boiling, and the titration continued as before. As soon as the precipitate of manganese hydrate begins to appear the flask must be heated for one minute after each addition of potassium permanganate. Then about 5 cc. of permanganate solution are added, and the titration is finished by alternately adding oxalic acid and potassium permanganate (compare this Journal, 1889, 1015). For the sake of uniformity in all the tests, tanning materials were so extracted as to furnish a solution containing in 1,000 cc. about 2 grms. of tanning substances. For this purpose the following quantities were dissolved in 1,000 cc.:—20 grms. of oak bark and of pine bark; 7 to 10 grms. of mimosa bark, sumach, and quebracho; 5 to 7 grms. of valonia, Hungarian galls and myrabolans; 5 grms. of divi-divi and algarobilla; 3 to 4 grms. of solid quebracho extract (for further experiments it is recommended to take a little more of the last-named substance, and to increase the quantity of sulphuric acid proportionately). 10 cc. of these extracts as well as 10 cc. of the hide powder filtrates were in each case mixed with 10 cc. of dilute sulphuric acid and then titrated in the described way. It is difficult to understand how Gantter arrived at the conclusion that the hide powder filtrate is of no practical importance for the tests, as great differences occur by neglecting the latter, on account of the oxidation of the non-tanning materials present (compare Procter's remarks, this Journal, 1890, 261). Hide powder has been used as recommended by the commission for determining a uniform method of tannin examination (1885), viz., 3 grms. of hide powder, gently heated with 50 cc. of the solution for 18 to 24 hours. For comparison the same substances were analysed by the indirect gravimetric method (this Journal, 1888, 647). Seven different samples of oak bark were tested in this way, with good results.

Gantter's method and the gravimetric tests accord fairly well with each other, but it has to be noticed that Gantter's results are on an average lower by 1.031. They are, like Loewenthal's, only relative results for tannin, useful merely for comparing different samples of the same tanning material. But whilst Loewenthal's figures are always less than those obtained by the gravimetric method, it is a disadvantage of Gantter's method that the results vary, being sometimes more than the gravimetric results. But if (in Gantter's method) by comparison with the gravimetric method, reduction factors have been found, then the real results can be ascertained from the relative results obtained by titration. Gantter's method may be more limited in this respect than Loewenthal's, but its application for tanning materials is easy. This is shown by the small differences between the gravimetric and Gantter's method, if the results of the latter be multiplied by 1.031, which is the average figure by which Gantter's results are smaller than those obtained by the gravimetric method. Even the greatest difference, viz., that of 1.30 per cent., is not bad, compared with the differences which are often obtained in Loewenthal's method when the latter is not always performed in a thoroughly uniform manner. The authors state that the average differences with Loewenthal's method amount to 1.36 per cent. in their own laboratory, but even if the tests be made in exactly the same way, the differences may amount to more than 2 per cent. In working with Gantter's method such variations are much smaller, and the accuracy is therefore greater, its chief advantage being that it does not require so much experience, skill and care as Loewenthal's method does. It is therefore to be recommended for analysts who only occasionally have to make determinations of tannin. But an analyst who is constantly practising and well experienced in Loewenthal's process,

will derive no great advantage from Gautter's method. Hence each method has its proper sphere. In doubtful cases the indirect gravimetric method is always to be preferred.

The result of the thoroughly critical examination to which the authors have put the methods of Gautter and Loewenthal, is a conclusion that it would be best to admit only the gravimetric results as absolute, and to leave it to every analyst to arrive, by the best means he can adopt, at these results, either by the gravimetric method itself, or by calculations from Loewenthal's or Gautter's methods. In cases of dispute the gravimetric method should always be accepted as decisive. The results obtained from other tanning materials than oak bark by Gautter's method are contained in the following table:—

	1.	2.	3.	4.	5.	6.
	Percentage of Tannin.		Factor 1:2.		2 mul- tiplied by 4.	Diffe- rence be- tween 5 and 1.
	Gravi- metric Method.	Gautter's Method.	—	Mean.		
Pine barks.....	{ 9.74 18.42 21.75 24.50 25.64	{ 9.55 18.45 17.75 18.80 20.00	{ 1.020 0.998 1.225 1.303 1.282	{ 1.009	{ 9.64 18.62 22.51 23.84 25.36	{ -0.10 +0.20 +0.76 -0.66 -0.28
Valonias .....	{ 30.78 30.85 32.36 34.54	{ 23.93 25.14 25.43 26.71	{ 1.286 1.227 1.259 1.293	{ 1.268	{ 30.34 31.88 32.25 33.87	{ -0.44 +1.03 -0.11 -0.67
Oak bark ex- tracts .....	{ 22.83 25.58 40.07	{ 17.97 20.45 33.64	{ 1.276 1.251 1.191	{ 1.261	{ 22.66 25.78 40.57	{ -0.17 +0.20 +0.50
Divi-divi.....	{ 42.21 49.21	{ 35.21 40.09	{ 1.199 1.227	{ 1.206	{ 42.56 48.35	{ +0.35 -0.86
Hungarian galls	{ 24.08 32.78 35.29	{ 20.86 28.21 30.64	{ 1.154 1.162 1.152	{ 1.156	{ 21.11 32.61 35.42	{ +0.03 -0.40 +0.13
Algarobilla.....	{ 42.78 43.64	{ 37.29 37.71	{ 1.147 1.157	{ 1.154	{ 43.03 43.52	{ +0.25 -0.12
Myrabolans ....	{ 31.15 39.00 28.78	{ 27.86 33.43 25.36	{ 1.118 1.167 1.135	{ 1.143	{ 31.84 38.21 28.89	{ +0.69 -0.79 +0.11
Sumach.....	{ 29.15 29.86	{ 25.42 26.29	{ 1.146 1.136	{ 1.139	{ 28.96 29.94	{ -0.19 +0.08
Mimosa barks..	{ 23.90 40.90	{ 29.20 49.57	{ 0.818 0.825	{ 0.822	{ 24.00 40.75	{ +0.10 -0.15
Quebracho wood	{ 25.30 47.80	{ 33.20 62.77	{ 0.762 0.762		{ 25.93 49.02	{ +0.63 +1.22
Paste-like Que- bracho ex- tracts .....	{ 48.57 49.77 68.80	{ 61.05 62.23 88.58	{ 0.797 0.800 0.778	{ 0.781	{ 47.68 48.60 69.02	{ -0.89 -1.17 +0.22
Solid Quebra- cho extracts..	{ 69.83 72.75	{ 86.33 96.00	{ 0.809 0.758		{ 67.42 74.98	{ -2.41 +2.23

These results differ considerably from the gravimetric method, but the authors have no doubt that by using special reduction factors for each substance, the method will be well adapted for the estimation of many tanning materials. It is added that the reduction factors given in the last table should first be confirmed by other tests before being used for technical purposes.—H. S.

#### Optical Analysis of Butter. C. Viollette. Compt. Rend. **111**, 348.

The following conclusions are drawn:—

(1.) That butter and margarin have different indices of refraction, varying within certain limits.

(2.) The indications of the oleo-refractometer are sufficient, provided the deviations for the various constituents of the mixture are known.

(3.) It is necessary to fix a limit of deviation below which all samples are considered margarin.

(4.) The oleo-refractometer is capable of being used in the examination of the commercial product, qualitatively only.—T. L. B.

#### A Method for the Quantitative Estimation of Camphor.

F. Foerster. Ber. **23**, 2981—2989.

The following method has been worked out especially in reference to the determination of camphor in ivorine, celluloid, and other solid products consisting of a mixture of camphor and nitrocellulose. When such a body is treated with caustic soda and the resulting solution distilled with steam, the camphor volatilises and may be readily dissolved out from the distillate by benzene. By determining the rotatory power of the resulting benzene solution, the quantity of camphor present can be found.

The author has in the first place investigated the relation of the rotatory power of a benzene solution of camphor to the quantity of camphor in solution. From Landolt's observations this ratio is expressed by the formula—

$$[\alpha]_D = (39.185 + 0.17084 C)^\circ$$

where "C" represents the degree of concentration, or the weight of substance contained in 100 cc. of the solution. Experimental results were not found to agree with this formula, and therefore fresh determinations were undertaken. The camphor used was purified by crystallisation from 50 per cent. alcohol, instead of by sublimation, as it was found that a certain amount of decomposition always takes place when the latter process is adopted; two recrystallisations were found to render it sufficiently pure. The purified product melts at  $176.3^\circ$ — $176.5^\circ$  when heated in a wide capillary tube; its point of solidification determined by Landolt's method was  $178.7^\circ$ ; it boils at  $209.1^\circ$  under a pressure of 759 mm. The formula deduced from the experiments, which are given in detail, is:—

$$[\alpha]_D = (39.755 + 0.17254 C)^\circ$$

Since—

$$C = \frac{100 a}{l [\alpha]_D}$$

where "l" is the length of the tube in decimetres it follows that—

$$C = 115.2052 \left( -1 + \sqrt{1 + 0.0436683 \cdot \frac{a}{l}} \right).$$

The determinations were made with a Laurent's half-shadow apparatus, using a water-jacketed tube 400 mm. long. The temperature was kept constant at  $20^\circ$ , to which temperature all the volumes employed were reduced, whilst all the weighings were reduced to their real value (i.e., in vacuum).

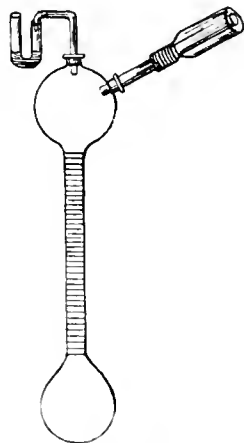
The following table of results shows the accuracy of the above formula:—

C found.	C calculated according to Formula.	Difference.
5	5.001	+ 0.0001
10	9.9824	- 0.0106
15	14.9982	- 0.0018
20	19.9915	- 0.0085
30	30.0037	+ 0.0037
40	39.9700	- 0.0300

To correct the above values for "C" to standard volume and a vacuum they can be multiplied by the factor 0.9966. For practical purposes this correction may be neglected.

The author has also studied the effect of temperature on the rotatory power of camphor dissolved in benzene, and finds that the latter increases with a rise in temperature, but that there is no simple relation between this increase and the temperature or degree of concentration of the solution. Tabulated results are given.

The actual determination of camphor in one of the already-mentioned substances is carried out as follows:—So much of the substance as corresponds to from 2—3 grms. of camphor are weighed out into a litre flask which is connected with a condenser and also provided with a dropping funnel. The receiver consists of a specially designed graduated vessel (see Figure), the lower bulb of which has a capacity of 150 cc., and the overlying, 1 cm. wide, graduated tube, a capacity of 30 cc. The decomposition is started by adding a 10 per cent. solution of caustic soda from the dropping funnel in sufficient quantity to saponify the product, the flask being heated on the water-bath to 80°. When the saponification is effected, a concentrated solution of caustic soda is then run in till the residue in the flask contains 10 per cent. of caustic soda. The contents of the flask are diluted, if necessary, to 250 cc. and then boiled, when the camphor distils over. The distillation is stopped when the lower bulb of the receiver is nearly full (120—150 cc.) and water added to the distillate up to the zero of the scale, which is at the bottom of the receiver tube. The condenser tube is then washed well with benzene and so much benzene is added to the contents of the receiver that the total volume (of benzene) is 25—30 cc. The small U-tube attached to the receiver (see Figure) is filled with benzene



during the experiment, to prevent any loss of camphor. The camphor is dissolved in the benzene by shaking the contents of the receiver, and the resulting benzene solution

is then ready for polarisation, after it has been brought to a temperature of 20° C. The method is accurate within 0.7—1.0 per cent. when pure camphor is employed. Two determinations of the camphor in celluloid gave 22.43 per cent. and 22.53 per cent.

For the determination of camphor in pharmaceutical preparations a slight modification of the process is desirable. Many of these preparations consist of a solution of camphor in fatty oils, in which cases the camphor is simply driven off by passing a current of steam through the oil, without the addition of any alkali. When the distillate amounts to about 300 cc. (a large flask is used as a receiver) it is treated with a little caustic soda and the distillation repeated under the conditions described above. The contents of the flask are apt to bump when heated, but a small platinum spiral prevents this. A trial of this method, using a 10 per cent. solution in camphor in olive oil, gave 98 per cent. of the total camphor contained.—C. A. K.

#### *Determinations of Alkaloids and their Molecular Weights.*

A. Christensen. Chem. Zeit. **14**, 1346—1352.

For determining ammonia, Kjeldahl has recommended a method of titrating the acid iodometrically. The author has applied this process with advantage for estimating alkaloids. He proceeds as follows: A weighed quantity of the alkaloid is dissolved in an excess of decinormal sulphuric acid and dilute alcohol made up with water to 50 cc. and 50 cc. of alcohol added. An excess of potassium iodide and iodate is added, which causes any excess of acid to react on these salts, setting iodine free. This is then titrated back with thiosulphate until the yellow colour of the solution disappears. If A cc. decinormal sulphuric acid were used, and a cc. decinormal thiosulphate solution, the molecular weight of the alkaloid being V, the quantity of the latter is  $\frac{V(A-a)}{10,000}$ , or the quantity of the alkaloid being found to be p, its molecular weight  $\frac{p \cdot 10,000}{(A-a)}$ .

Almost all alkaloids can be determined according to this method, the only exceptions being pilocarpine, narcotine, thiobromine, and caffeine, which is due to the ready dissociation of their sulphates. In the case of pilocarpine this may be prevented by titrating in aqueous solution; or in the case of narcotine the solutions should be thoroughly cooled.

Details of the carrying out of this process are given for the quinine, opium, and strychnine alkaloids, as well as for using it in the case of mixtures of different alkaloids.—A. R.

#### *The Estimation of the Sugars by Potassium Copper Carbonate Solution.* Part II. H. Ost. Ber. **23**, 3003—3011.

THE solution of potassium copper carbonate, the preparation of which has been previously described (this Journal, 1890, 825), has the following advantages over Fehling's solution:—It is very much more stable; it is much less reduced by cane sugar; slight departures from a particular mode of working cause less variation in the result, and consequently it is more easily managed; the amount of copper reduced by a given weight of sugar is from 1½ to 2 times as great; the different glucoses each reduce a different amount of copper; in using the solution volumetrically, the completeness of the reaction is sharply marked by the solution becoming completely colourless, and consequently no indicator is necessary.

In performing a gravimetric analysis, 50 cc. of the copper solution containing 298.7 mgrms. of copper are heated to boiling for 10 minutes with 25 cc. of the sugar solution. In a volumetric estimation, 50 cc. of the copper solution are heated with the amount of sugar solution needed to just decolourise the solution after 10 minutes' boiling. The volume of the mixed solutions should be about 75 cc.

Determinations have been made gravimetrically with the different glucoses. The following table contains a condensed synopsis of the results:—

Copper.	Invert Sugar.	Dextrose.	Levulose.	Galactose.	Arabinose.
Mgrms.	Mgrms.	Mgrms.	Mgrms.	Mgrms.	Mgrms.
50	15.2	15.6	14.7	17.4	17.0
75	22.3	22.9	21.7	25.9	25.1
100	29.5	30.3	28.5	34.5	33.1
125	36.8	37.8	35.4	43.1	41.1
150	44.0	45.3	42.5	51.8	49.3
175	51.5	52.9	49.5	60.8	57.5
200	59.1	60.8	57.0	70.3	66.2
225	67.5	69.2	65.2	80.3	75.4
250	76.7	78.4	74.4	91.2	85.5
275	87.1	89.9	85.9	103.3	97.1
298.7	100.0	102.5	99.0	117.0	109.5

The numbers in the last line show the amount of sugar required to completely precipitate 50 cc. of the copper solution, *i.e.*, the volumetric factors.

Lactose does not give such concordant numbers as the above glucoses, 198 mgrms. of lactose (hydrated) completely reduce 50 cc. of copper solution, that is reduce 298.7 mgrms. of copper, and 150 mgrms. of lactose reduce 231.2 mgrms. of copper.

The author has applied this mode of estimating sugar to the determination of the reducing power of hydrolysed lactose and raffinose.

The greatest reducing power of hydrolysed lactose is obtained by boiling 1 gm. of sugar with 80—90 times its weight of 0.6 per cent. hydrochloric acid for 5—8 hours. 50 mgrms. of the glucoses contained in such a solution when neutralised reduce 152—153 mgrms. of copper. If 25 mgrms. of dextrose and 25 mgrms. of galactose be submitted to the same treatment they reduce 149.7 mgrms. of copper.

Raffinose does not reduce potassium copper carbonate solution; when hydrolysed the highest reduction is obtained with 1.1 gm. of crystallised raffinose (containing 1.0 gm. of glucose) is boiled for 5 hours with 80—90 times its weight of 0.3 per cent. hydrochloric acid, 50 mgrms. reduce 150 mgrms. of copper. 50 mgrms. of a mixture containing equal quantities of dextrose, galactose and levulose submitted to the same treatment reduce 152.0 mgrms. of copper.—A. L. S.

*The Estimation of Cane Sugar.* C. O'Sullivan and F. W. Tompson. *Proc. Chem. Soc.* 1890, [88], 160.

THE authors describe a new method of estimating sucrose which they claim is applicable to all solutions containing cane sugar, including natural juices and other preparations in which the use of acids is not possible. The process is a very simple one, and it is shown, by comparing it with estimations in which invertase is used, that it is very accurate. It is based on the one used by Kjeldahl in 1881, and is as follows:—

The neutral solution containing cane sugar is placed in a constant-temperature bath at 55°. A little pressed brewer's yeast is added, and complete admixture secured by gentle stirring. At the end of four hours inversion is nearly always finished. The solution is cooled, made up to double its original bulk, and filtered. During the process there is a decrease in the optical activity of the solution, and an increase in the cupric reducing power. Each of these factors is an accurate measure of the amount of cane sugar originally present. The authors quote experiments showing that good results are thus obtained in solutions which would not admit of the use of acids.

*Determination of Mineral Matter in Sugar by Means of Benzoic Acid.* E. Boyer. *Compt. Rend.* 111, 190—192.

DIRECT incineration is a long and delicate process for which the process of Scheibler (incineration in presence of sulphuric acid) has been substituted. Correction is, however,

needed in this case, and it is a disputed point what this correction really ought to be. The author, therefore, proposes, instead of sulphuric acid, to use benzoic acid dissolved in strong alcohol. 5 grms. of sugar are moistened with 1 cc. of distilled water in a platinum crucible; the crucible is gently warmed, but not so that the sugar is carbonised, a little alcoholic benzoic acid solution is added, and the heating is continued, at first carefully, until the alcohol is all gone, then more strongly. The benzoic acid gives off a large amount of vapours, which swell out the sugar. When the benzoic acid has all volatilised there remains a voluminous residue of carbon. The crucible is then put into a muffle at a dull red heat for, say, half an hour, at the end of which time incineration should be complete, and a perfectly white ash should remain. The weight of ash represents the mineral matter contained in 5 grms. of the sugar.

Ammonium benzoate may be used instead of benzoic acid.  
—T. L. B.

*On the Estimation of Mineral Oil or Unsaponifiable Matter.* Fairley and Burrell. *Pharm. J.* October 18, 1890, 315.

IN the Yorkshire district large quantities of oil and grease are used in the woollen and leather manufactures and other purposes. Much of this oil is recovered and used again for the same or other operations. It is frequently found that these recovered oils and greases contain large proportions of mineral oil or unsaponifiable or resinous matter, which are not always readily estimated by the process given in text-books. The following process has, however, been found to succeed in these cases:—

Five grms., equivalent to nearly 80 grains of the oil, are weighed out, saponified with alcoholic potash (about 80 grms. of KHO in 1,000 cc. of alcohol), evaporated in a basin on the water-bath to pastiness, then dissolved in from 45 to 50 cc. of warm water and treated in a separating funnel with an equal volume of ether and 2 to 3 cc. of alcohol and shaken 3 to 4 minutes. On standing the ethereal solution which comes to the surface is decanted and evaporated. A second treatment with ether is unnecessary, because, as a rule, the results with a single extraction are correct within 0.1 and 0.2 per cent.

The points in this process are—1. Keeping the aqueous solution of the soap within a volume not exceeding (for 5 grms. of oil) 50 cc. 2. Treating with an equal volume of ether in one operation, at a temperature of near 90° F. Then the ethereal solution of the mineral oil separates, and, in all successful experiments, is very nearly equal to the volume of ether employed. 3. Washing this ethereal solution with warm water in the separator, which can be done very quickly. 4. When the residue, left after evaporation of the ether and drying, has been weighed, it should always be distilled in a small tube retort. If it distils unchanged, without blackening or formation of acrolein, it is free from soap or ordinary fats.

The peculiar bitter after-taste and fluorescence of oils containing mineral oil are also of importance.

The following results were carried out by the above process, and by different operators:—

No.	Mineral Oil.	Per Cent.
1	A.....	16.40
	B.....	16.24
2	A.....	17.87
	B.....	17.60
3	A.....	18.85
	B.....	18.60
4	A.....	32.8
	B.....	31.4
5	A.....	35.50
	B.....	35.36



*On the Estimation of Cotton-Seed Oil in Lard.* Fairley and Cooke. Pharm. J. October 18, 1890, 315—316.

For this purpose Beech's test, when carefully worked, gives useful results, which can be confirmed by the observation of the heat given out in mixing the samples with a definite proportion of sulphuric acid.

Attempts have been made to apply the specific gravity test; and Bockairy, in a recent number of the *Bulletin de la Société Chimique* (3rd series, ii., 310), takes the density of the lard at 50° C. = 122° F. He finds that unadulterated lard varies little in density at this temperature, and that there is a sufficient difference between the density of lard and cotton-seed oil to give a means of approximate quantitative estimation.

Bockairy gives the following numbers:—

	Density at 50° C.
Lard, highest.....	*8915
Lard, lowest.....	*889
Lard, mean.....	*890
Very rancid lard.....	*8895
Oleo-stearine.....	*8885
Rend fat of the ox.....	*8895
New cotton-seed oil.....	*897
Old cotton-seed oil.....	*896

#### *New Cotton-Seed Oil and Lard.*

Cotton-Seed Oil.	Lard.	Density at 50° C.
0	100	*890
10	90	*8915
20	80	*892
30	70	*8925
50	50	*894
75	25	*8953
100	0	*897

Careful experiments fully confirm the utility of the method and prove that it may give a most valuable confirmatory test.

The following is a list of results:—

#### EXPERIMENTS WITH LARD.

*Sp. Gr. of Mixtures at 50° C. (Water at 50° C. = 1,000).*

<i>Lard.</i>	
900°42 } .....	Mean = 900°38
900°35 } .....	

#### *Lard + 10 per cent. Cotton-Seed Oil.*

901°19 } .....	Mean = 901°16
901°13 } .....	

#### *Lard + 20 per cent. Cotton-Seed Oil.*

902°15 } .....	Mean = 902°09
902°03 } .....	

#### *Lard + 30 per cent. Cotton-Seed Oil.*

903°11 } .....	Mean = 903°02
902°93 } .....	

#### *Lard + 50 per cent. Cotton-Seed Oil.*

905°13 } .....	Mean = 904°94
904°85 } .....	

#### *Lard + 75 per cent. Cotton-Seed Oil.*

907°33 } .....	Mean = 907°36
907°39 } .....	

#### *Cotton-Seed Oil.*

908°72 } .....	Mean = 908°79
908°86 } .....	

*Sp. Gr. of Mixtures at 50° C. (Water at 15·5° C. = 1,000).*

#### *Lard.*

891°63 } .....	Mean = 891°59
891°56 } .....	

#### *Lard + 10 per cent. Cotton-Seed Oil.*

892°30 } .....	Mean = 892°46
892°53 } .....	

#### *Lard + 20 per cent. Cotton-Seed Oil.*

893°34 } .....	Mean = 893°28
893°22 } .....	

#### *Lard + 30 per cent. Cotton-Seed Oil.*

894°39 } .....	Mean = 894°21
894°12 } .....	

#### *Lard + 50 per cent. Cotton-Seed Oil.*

896°29 } .....	Mean = 896°17
896°06 } .....	

#### *Lard + 75 per cent. Cotton-Seed Oil.*

898°18 } .....	Mean = 898°50
898°53 } .....	

#### *Cotton-Seed Oil.*

899°87 } .....	Mean = 899°92
899°98 } .....	

*Examination of Olive Oil.* G. Meyer. Chem. Zeit. **14**, 1202.

DETERMINATIONS are made of the specific gravity, of polarisation, of bromine absorption, and of saponification value.—T. L. B.

### ANALYTICAL AND SCIENTIFIC NOTES.

*The Diffusion of Carbonic Anhydride through Caoutchouc.* E. Obach. Chem. Zeit. **14**, 1142.

On emptying one of Kipp's carbonic acid apparatus for the purpose of recharging, which had been fitted with a disc of brown unvulcanised rubber, to prevent small particles of marble from falling into the acid in the lowest bulb, the author noticed some large blisters, one of which was egg-shaped, with a diameter of 2 cm., and projected only on one side of the disc; another extending equally on both sides. When exposed to the air, these blisters gradually became smaller, the former diminishing to a diameter of 16 mm. after an exposure of 25 minutes. The rubber sheet from which the disc was cut had evidently been made by rolling together two thinner sheets, and the adhesion not being perfect in some places, air had entered, to be afterwards replaced by carbonic acid. (See also this Journal, 1890, 1072.)—F. H. L.

*Relative Stability of Salts, whether isolated or in presence of Water.* Berthelot. Compt. Rend. **111**, 135—144.

STRONG acids and strong bases are characterised by the fact that their union gives rise to a much greater development of heat than the union of weak acids and bases; by reason of this circumstance the salts of strong acids and strong bases are the more stable; that is to say, in the isolated condition they, as a rule, resist the action of heat and the decomposing action of water better than salts from weak acids and weak bases.

In comparing the heats of formation of various series of salts, it is found that the differences between the heats of formation of solid salts of the same base, united with two distinct acids, is approximately constant for equally strong acids; whilst the difference is more uncertain where salts of

weak acids are concerned. This points to instability in these salts. Like phenomena occur in the case of bases. Similar differences are observable with solutions of salts, and they show the dissociation existing amongst salts of weak acids and bases, compared with salts of strong acids and bases.

In the present memoir salts of aniline are examined in solution. In sulphate of aniline, ammonia, it is shown, displaces the aniline completely; soda replaces equally ammonia, and there is here a "series," confirmed by the respective heat reactions.

Acetate of aniline shows a much greater degree of dissociation than ammonium acetate does.—T. L. B.

*On Silk Secretion by Bombyx Mori.* R. Dubois. *Compt. Rend.* **111**, 206—207.

THE name *fibroin* has been given to the central portion of the thread of the cocoon and also to the viscous contents of the centre of the silk gland. Both, however, differ materially; the latter, for instance, being soluble in potassium carbonate (15 to 100), the former not. It has been suggested that the fibroin of the gland undergoes on exit a coagulation similar to that which the white of an egg undergoes when heated; it has further been considered, that desiccation or oxidation may have something to do with the phenomenon in question. The author reduces the probability to the consideration of coagulation and then shows that the coagulation is not to be compared to the coagulation of albumen by heat, but rather to the coagulation of blood. A kind of serum is obtained by macerating silk glands for two or three days in a cool place with distilled water, or with salt solution (4 to 100), or with potassium carbonate solution (15 to 100). This serum is filtered, and is then agitated; a clot forms without addition of any reagent. The clot presents the ductility and elasticity of silk on exit from the duct, but it quickly loses the property whereby it admits of being spun. It fixes a portion of the yellow colouring matter contained in solution in the artificial plasma, and resists the action of reagents which dissolve the contents of the gland before coagulation. As is the case with blood, oxygen aids the formation of the clot, for when the air in the experimenting vessel is replaced by carbon dioxide or hydrogen a clot is certainly still obtained on agitation, but if this be filtered off and the filtrate shaken again, this time in contact with air, a further clot will be obtained.—T. L. B.

*The Colouration of Silk by means of Coloured Food given to Silkworms.* L. Blanc. *Compt. Rend.* **111**, 280—282.

See under V., page 1125.

## New Books.

**ASBESTOS.** Its Properties, Occurrence, and Uses, with some Account of the Mines of Italy and Canada. By ROBERT H. JONES. London: Crosby, Lockwood, and Son, 7, Stationers Hall Court, Ludgate Hill. 1890.

OCTAVO volume, bound in cloth, price 12s. 6d., containing Frontispiece, Preface, Table of Contents, List of Illustrations, and Text. The Text covers 226 pages and concludes with an Alphabetical Index. The pages of this volume are exceedingly well illustrated by eight colotype plates and 39 engravings.

The matter is sub-divided into Chapters treating of the following details, and prefaced by an Introductory Chapter. Chap. I. Varieties and Qualities of Asbestos. II. Present Sources of Supply. III. Italian Asbestos and the Italian Mines. IV. Canadian Asbestos. V. The Canadian Mines

or Quarries. VI. Output, Cost of Production, Wages, &c. VII. Newfoundland and Norway. VIII. The Uses of Asbestos for Engineering Purposes. IX. Applications of Asbestos to Military and Fire Preventive Purposes. X. Miscellaneous Applications of Asbestos. XI. Substitutes and Similarities. XII. Fibre Spinning.

In the preface the author states his object in writing this book as being the preparation of "a synopsis of all that is known on the subject of asbestos generally, so that those who, like himself, are interested in this singular mineral, may find all the details ready to hand without the expenditure of the labour and time he has had to give in looking them up."

**SUGAR ANALYSIS.** For Refineries, Sugar Houses, Experimental Stations, &c., and as a Handbook of Instruction in Schools of Chemical Technology. By FERDINAND G. WIECHMANN, Ph.D. New York: John Wiley and Sons, 53, East Tenth Street. London: Kegan Paul, Trench, Trübner and Co., Ltd. 1890.

OCTAVO volume bound in cloth, price 10s. 6d. The work is dedicated to the author's teacher, Professor Ch. F. Chandler, of Columbia College, and consists of a systematic treatise on the physical and chemical methods for determining the strength and constituents of sugars and sugar juices, &c. It commences with a Preface, followed by Table of Contents, and Subject-Matter covering 181 pages. An Alphabetical Index concludes the work. Pages 116 to 181 are entirely devoted to tables of strengths, constants, &c. There are five wood engravings of a simple character.

The contents are sub-divided as follows:—Chap. I. Physical Apparatus required in Sugar Determinations. II. Sampling Sugars and Molasses. Determination of Colour of Sugar and Sugar Solutions. Determination of Densities, Alkalinity, and Acidity. Test for Sulphurous Oxide in Sugar. III. Determination of Sucrose in absence of other Optically Active Substances. Optical Analysis. Quotient of Purity or Exponent. Gravimetric Analysis. IV. Determination of Sucrose in the Presence of other Optically Active Substances. Clerget's Inversion Method. Sucrose in the Presence of Raffinose and Dextrose. Sieben's Process for Destruction of Lævulose. Determination of Sucrose, Dextrose, and Lævulose. V. Invert Sugar. Qualitative and Quantitative Determination. Volumetric and Gravimetric Methods. Soldani's Solution. VI. Water. Ash. Quantitative Analysis of Sugar Ash. Suspended Impurities. Determination of Woody Fibre. Detection of the Sugar Mite. VII. Organic Non-sugar. Classification of Organic Bodies accompanying Sucrose. Schemes for the Analysis of the Organic Acids. Determination of Total Nitrogen. Non-nitrogenous Organic Substances. Determination of Pure Cellulose. VIII. Notes on the Reporting of Sugar Analyses. Rendement. Calculation of Rendement. Duty. Calculation of the Weight of Solids and Liquids from their Specific Gravity. Chapter IX. contains a valuable list of References to Literature on Sugar Analysis.

**CHEMISCH-TECHNISCHES REPERTORIUM,** Uebersichtlich Geordnete Mittheilungen der neuesten Erfindungen, Fortschritte und Verbesserungen auf dem Gebiete der technischen und industriellen Chemie mit Hinweisen auf Maschinen, Apparate, und Literatur. Herausgegeben von Dr. EMIL JACOBSEN. 1889. *Erstes Halbjahr, Zweite Hälfte.* Berlin: R. Gaertner's Verlagshandlung, Hermann Heyfelder, S.W. Schönebergerstrasse, 26. London: H. Grevel & Co., 33, King Street, Covent Garden.

THE first issue for the second half-year of 1889. It contains 107 pages of subject-matter, copiously illustrated with wood engravings. In the text the following subjects are treated of:—Building Materials, Cements, Artificial Stone. Colours, Dyeing and Calico Printing. Fats, Oils, Illuminating and Heating Materials. Fermented Liquors. Tanning, Leather and Glue Manufacture. Textiles, Glass, and Earthenware. Wood and Horn. India-rubber. Cements, Plasters, Artificial Stone. Lakes, Varnishes and Paints. Metals.

GRUNDZÜGE DER THEORETISCHEN CHEMIE. VON LOTHAR MEYER. Leipzig: Druck und Verlag von Breitkopf und Härtel. 1890.

OCTAVO volume in paper cover, dedicated to Professor Franz Neumann. Dedication. Preface. Table of Contents and 200 pages of subject-matter. The work concludes with an Alphabetical Index and a table exhibiting the Natural System of the Elements. This abridged form of a work so well-known as Lothar Meyer's "Modern Theories of Chemistry" will be very welcome to students or those who desire a more condensed account of the subject. Although Ostwald's "Grundriss der allgemeinen Chemie" might appear to cover precisely the same ground, in reality it does no such thing, as the author points out, and there is consequently room for the abridgments of both well-known and complete works.

#### ERRATUM.

In the November number of this Journal, 1890, page 1074, second column, "*Report on Analyses and Research conducted in 1886-1889 in the Central Chemical Laboratory of Italian Customs*," &c., for the first paragraph substitute the following:—This report is addressed to Senator Cannizzaro.

## Trade Report.

### BOARD OF TRADE NOTICE.

#### TENDERS FOR THE PURCHASE OF ANTIMONY ORE FROM THE SERBIAN GOVERNMENT.

The subjoined translation of a note issued by the Serbian Minister of Commerce, inviting tenders for the purchase of certain antimony ore, has been received from Her Majesty's Minister at Belgrade, and was issued from the Foreign Office on the 2nd December.

On the 28th December next (9th January 1891) there will be publicly sold at the Mining Section of the Royal Department of Commerce and Agriculture, to the highest bidder, 100,000 kilos. of antimony ore from the Government mines of Podrinje. The conditions of sale, as well as the analysis of the above-mentioned ore, will be on view every day from 11 to 12 at the said office for (the convenience of) those concerned. Ten thousand francs (400*l.*) in cash must be deposited as caution money. Tenders must be sent in by noon on the 28th December (9th January 1891) at the latest, with all particulars in accordance with prescribed conditions.

### TARIFF CHANGES AND CUSTOMS REGULATIONS.

#### ROUMANIA.

##### *The Importation of Vegetable Oils.*

Sir F. C. Lascelles, Her Majesty's Minister at Bucharest, in a despatch to the Foreign Office, dated the 22nd November, encloses translation of a regulation published in the official *Monitor*, stating that the importation of vegetable oils can only take place at the following Custom houses in Roumania:—Jassy, Galatz, Braila, Giurgevo, Ploesci, Bucharest, Severin, and Craiova. The reason given for the issue of this regulation is that the greater part of the

vegetable oils which are imported into Roumania are adulterated; that it is not easy to detect the adulteration; and that it is only at the Custom houses named that a proper analysis can be made.

#### ARGENTINE REPUBLIC.

##### *Tariff Changes.*

*Note.*—Dollar = 4*s.* (nominal). Litre = 0.22 Imp. gallon.

The Hon. F. J. Pakenham, Her Majesty's Minister at Buenos Ayres, in a despatch to the Foreign Office, dated the 27th October, forwards a statement of amendments in the Argentine Customs tariff, which had been put to the vote in the Senate, and passed. The following chemicals are mentioned in the statement in question:—

Imports to pay, *ad valorem*:—

50 per cent.—Gunpowder, perfumery.

45 per cent.—Wooden matches, rockets.

40 per cent.—Tanned leather.

15 per cent.—Dynamite.

10 per cent.—Paper, cement.

5 per cent.—Sulphuric acid, sulphate of lime, zinc, lead.

25 per cent.—All articles not specified as above.

Special import duties are as follows:—

Starch, 90 dollars per ton, refined.

Sugar, 90 dollars per ton; kerosine, 5 cents. per litre; candles, 18 cents. per kilo.

Free of duty.—Quicksilver, sulphur, benzene for combustion, and sheep dips.

#### EQUADOR.

##### *New Customs Law.*

*Note.*—Kilogramme = 2.204 lb. avoirdupois. Sucre = 4*s.* 2*d.* (nominal value).

The following are from a translation of a new law passed by the Government of Ecuador, making various modifications in the Customs tariff and regulations forwarded in the report of Mr. G. Chambers, Her Majesty's Consul at Guayaquil, dated the 3rd October last:—

New articles free of duty.—Powdered sulphur, carbolic acid, and chloride of lime.

New articles at 1 cent. per kilo.—Dynamite or gunpowder for mines, wood charcoal, common sheet glass.

New articles at 2 cents. per kilo.—Tubes of iron, lead, clay or porcelain, carbonate of potash, caustic soda.

New article at 5 cents. per kilo.—Machine grease.

New articles at 50 cents. per kilo.—Powder made up into artificial fireworks, slippers and other rubber articles.

New export dues.—The export dues upon each 100 kilos. of the following will be:—

Rubber, 5 suere; mangrove bark, 0.64 cents.; hides, 0.64 cents.; orchilla, 0.64 cents.; tanned hides, 1 suere.

### EXTRACTS FROM DIPLOMATIC AND CONSULAR REPORTS.

#### FRANCE.

##### *Tartaric Acid.*

The manufacture of tartaric acid in Marseilles has received a considerable impetus from the establishment of a new factory. In 1888 the output in that city was about 400 tons; in 1889 it reached 800 tons.

#### JAPAN.

##### *Fine Chemicals.*

The Japanese are now trying to manufacture iodide of potassium themselves. Among new remedies sulphonal and chloralamide have been introduced as hypnotics in 1889.

Of the former small quantities have been sold; the latter has not yet been able to gain a footing. Among antipyretics, antipyrin and antifebrin have maintained their position; phenacetin and exalgine have also been tried, but have not as yet found any sale whatever. In santonin very little business was done during 1889, as the Japanese, owing to the high quotations for that preparation, use the raw worm-seed, which can be bought at 7 dols. per picul. Of amorphous phosphorus, French and Russian makes have been introduced in competition with the English, but thus far without the same success as the first named. The excessively stringent inspection of all imported chemicals in the Government laboratories still continues, and from all those not strictly in conformity with the Japanese Pharmacopœia the official stamp, without which they may not be sold in Japan, is withheld.

The following were among the quantities imported into Yokohama in 1889 (the figures representing cetties of 1½ lb.):—Potassium bromide, 32,170; potassium iodide, 21,206; glycerin, 58,821; gum arabic, 116,444; carbolic acid, 25,900; caustic soda, 1,289,903; cinchona, 34,524; phosphorus, 11,626; salicylic acid, 15,224; santonin, 5,029; tartaric acid, 15,199. In addition to these the imports of quinine were 47,332 oz., and of morphia, 6,736 oz. Of unenumerated raw drugs, 83,514 yen; of medicines and fine chemicals (unenumerated), 402,587 yen; and of dye-stuffs, 745,802 yen value were also imported.

#### MEXICO.

##### *Drugs and Duties.*

The following figures represent the imports of drugs and chemical products into Mexico during the first half of the fiscal year 1888—89, from the four principal markets of supply:—

Imported from	United States.	United Kingdom.	France.	Germany.
Drugs, value, dols. ..	476,756	40,949	117,652	50,304
Duties payable on these, dols. ....	318,498	24,408	56,878	29,208
Or per cent. ....	66½	59½	47½	58½

#### RUSSIA.

##### *Customs Regulations.*

Stringent, and in many cases vexatious, Customs regulations continue to be strictly enforced. The attention of shippers cannot too urgently be called to the necessity of conforming to the Russian Customs regulations in every particular, to avoid "accidence" fines and penalties connected with incorrect description of weight and quantity of goods in bills of lading. A translation of the Russian regulations has been published by the Board of Trade, and all firms interested in the trade with this country are recommended to provide themselves with a copy. During the last navigation season, 10 British vessels were obliged to pay fines varying from 10*l.* to 20*l.* for contravention alone of the rule which makes the production by shipmasters of bills of lading imperative on the arrival in a Russian port.

##### *Drugs at Nijni-Novgorod.*

The great fair of Nijni-Novgorod, in 1889, was, on the whole, a successful one. Among other goods a good business is reported to have been done in drugs, indigo, and olive oil. Of drugs, dyestuffs, and paints, 5,565,750 roubles' worth were brought to the fair, against 5,479,850 roubles in 1888.

#### BRAZIL.

##### *Drugs for Bahia.*

The following is the value of pharmaceutical imports (in lbs.) into Bahia during the last five years:—

	1885.	1886.	1887.	1888.	1889.
Plants and herbs. ....	24,369	20,215	44,739	32,689	48,175
Perfumery, dyes, &c. ...	\$9,563	54,108	114,479	99,391	85,494
Chemical products. ....	14,664	30,033	69,509	41,824	38,199
Surgical instruments. ...	1,973	1,177	2,344	1,932	1,375

#### ITALY.

##### *The Naples Oil Industry.*

There are in the province of Naples 140 oil mills, all working on very antiquated principles—not a single one being a steam mill. A total of 780 workmen are employed in them. They work on an average 38 days every year, and produce an aggregate of 4,200 hectolitres of olive oil.

##### *Chemicals.*

In the same province there are 31 chemical works, employing 373 hands. Sulphuric and nitric acids, matches, soap, and blacking are the principal products.

#### PERU.

##### *Coca.*

Two establishments exist in Peru for preparing cocaine from the coca-leaf, one in Lima and one in Callao. During 1889, 1,730 kilos. of cocaine were exported to Europe, principally for Germany. No statistical data are forthcoming concerning the amount of production of leaves, but in the year 1888, 28,660 kilos. were exported through the port of Callao.

#### MISCELLANEOUS TRADE NOTICES.

##### REPORT FROM BAKU.

##### *Chem. Zeit.* 14, 1393.

An unusual naphtha spring has been discovered at Bina-gadi, near Baku, oil being struck at a depth of only 55 m. From St. Petersburg new laws are announced regulating the naphtha industry. Foreigners and Jews are only allowed to engage in the work by special permission of the Court. Trial borings must be covered in, and sufficient tank accommodation for the crude naphtha provided, to avoid waste of material.—F. H. L.

#### THE MINERAL RESOURCES OF INDIA.

The following interesting information respecting the mineral resources of India is extracted from the *Times of India* for the 25th October:—

Though less interesting than iron, in that they do not affect the greatness of the country so much, the baser metals of India are of especial interest from the antiquity of their development, and the fact that to a large proportion of the inhabitants they have, practically, been the only useful metals known. The noble metals, as gold and silver are called, have been used almost entirely for ornament and coinage. Platinum, really a precious metal, more valuable even than gold, is generally found with the latter, though rejected by the native gold washers of the Indus Valley as *safed sona* and valueless. It has never yet been discovered in large quantities in India, and except in the Indus Valley and the Kolar gold-fields, we have no evidence of the discovery of more than traces of the metal. Mercury,

though thoroughly understood in this country, does not seem to have been discovered in any large quantities, though the persistent rumour that asserts its plentiful presence in the Andaman Islands can hardly be without some basis. The probable explanation may be in a discovery of cinnabar in the Little Andaman, which has, as yet, never been thoroughly searched, as its rocks of the early tertiary period interspersed with volcanic "faults" closely assimilated to those of the great mercury beds of California. The author of "Hamilton's East Indies" saw in 1694 a native who had brought much mercury from the Little Andaman (of which he was a native) to the Nicobars. It has been discovered in Cannanore in spots of pure metal in a bed of laterite, but never seems to have been much developed. Pure mercury is brought from Afghanistan, said to be obtained from Pir Kisee, some 250 miles beyond Kandahar. The trade, however, has never reached any importance. Of the baser metals proper, by far the most important and valuable is copper, as it is also, perhaps, the oldest known in Asia. There are mines now unused that appear to be of great antiquity, showing in this as in many other things how greatly the people of India have deteriorated in scientific knowledge. Where formerly the extraction and smelting of copper ores was carried out on a large scale, we now see petty workings that yield before every reverse of nature. The largest copper workings in India are those of the Singhbhum district, extending as they do for over 76 miles from Midnapore to Lohardugga. It is believed that the Jains started mining in this district over two thousand years ago, since which they have never quite ceased work.

In the Goomacunda Valley, in the Karnaul district, there exists a deserted copper mine, so old that the very legend of its workers has been lost to the people living there. In the Hazaribagh district there are also deserted copper mines and large mounds of copper slag, showing that the ore was smelted on the spot. Ore is found here, which would probably repay any attempt at systematic working. The chief secrets of success in Indian mining seems to be: 1st, working on a scientific basis so as to waste neither metal nor labour; and 2nd, utilising native labour wherever possible, and obtaining European foremen who can teach their men the practical use of their tools.

The Khetri mines, as they are called, including those of Kullari and Singhana, are still worked, and of great antiquity. They resemble nothing so much as a rabbit-warren, the galleries, such as they are, winding about after the metal and being little bigger than absolutely required to admit the bodies of the miners. Large quantities of blue vitriol and coppers are extracted from the refuse of the mines, and these "refuse products" are at least as valuable as the actual ore itself as at present extracted; but this is becoming a not uncommon feature of modern manufacturing industries. Flooding is the great danger of the Kullari galleries, as the only means of clearing them from water consists of a string of men with *ghurras*. The Singhana mine must have paid well in former days, for the miners paid 14,000 rupees a year to the Khetri rajah as the rent of the mines, in addition to one-sixth of the copper mined. It is thought that copper mining in the Hazara district would be profitable; but the question of carriage would be a serious one, though the ore is extremely rich, being in this respect far superior to that found in Kumaon. The Nepalese do a good deal of copper mining in a small way, their ore yielding about  $4\frac{1}{2}$  per cent. of copper. The Hazara ores are said to have yielded from 60 to 90 per cent. of copper from the picked ore, but we feel utterly disinclined to accept these figures. They probably were obtained, if obtained at all, from small and excessively rich selected pieces, and are by no means a fair sample of the whole. Possibly by "copper" is meant "regulus," which in itself only yields 50 per cent. of copper. Even the smallest pieces of ore could not yield 90 per cent. of pure metal. Next to copper, its common alloys, zinc and tin, are the most interesting of the baser metals. Zinc is only found to any extent in Oodeypore, where the Jawar mines were formerly worked on a large scale, and yielded a yearly revenue of nearly 2½ lakhs of rupees. At present no extensive zinc workings exist in India, though possibly the

indications of the metal at both Sirmur and Tavoy might yield profitable results to scientific development. Tin, on the contrary, is found on a large scale in several places, though the fact that the natives of India have used brass universally for centuries seems to point to the presence of zinc in some form or another possibly imported. Tin occurs chiefly as a dioxide, the tin stone containing as much as from 75 to 80 per cent. of metallic tin. In 1867 the native iron smelters of Raniganj smelted some of the tin stone, supposing it to be iron ore, and when they saw white metal, mistook it to be silver. Burmah is the greatest Indian source of tin. It is worked by the Karens in the hill, near the Salwin, and sold by them in Tounghu. In the Tenasserim division, tin stone is very plentiful, every stream bed near Malceewoon in Mergui yielding the metal when washed. Dr. Oldham states that the main source of all the Tenasserim tin is the granite range separating that province from Siam, where "it exists as an essential ingredient of the mass of rock, occurring disseminated through the granite in small crystals, and being similarly arranged to the quartz and felspar. . . . The degradation of this granite by weathering through an enormous period of time has supplied the sand which is now so abundantly impregnated with stream tin."

At Mergui it used to be worked in the very gardens of the town, and in the Thawhawleek River there have been extensive stream washings for years, the fine sand being sorted out with a cane shovel that acts like a large sieve, and finally washed in wooden dishes, in which the tin sinks by its own weight on the water being revolved. The only European attempt to work Burmese tin on a considerable scale was made between 1873 and 1877, when Messrs. Steel and Sons leased the township of Malceewoon, where they not only washed the stream tin but opened out veins of ore in the hills. During the cold weather of 1874—75 some seven tons of metal and 14 tons of cleaned picked ore were exported. The works were, however, closed in 1877. It is worthy of note, however, that since the European workers failed, the Chinese have found the mines remunerative, and are still at work there, though they pay a ground rent and a royalty of 5 per cent.

From a manufacturing point of view, tin naturally suggests lead, and no mining industry, except iron, shows such a falling off as that of the working of lead mines. Galena, the sulphide of lead, is common in many parts of India, but Cuddapah and Karnaul are probably richest in the ore. In Cuddapah, at the village of Judgmrazpilly, close to the Nallamallay Hills, the old lead workings are conspicuous.

A sample of ore from these regions was analysed in 1879, and yielded 78 per cent. of lead and 22 oz. 7 dwt. of silver to the ton of lead. Both these and the Karnaul mines would probably repay scientific exploitation handsomely. Three analyses of ore from the latter yielded, respectively, 374 oz., 175 oz., and 165 oz. of silver to the ton, which would render them well worth working. In Bengal galena has been found in the Southall Peggannahs and also in Bhagalpore. An analysis of ore found in the latter district showed 78 per cent. of lead and 103 oz. of silver to the ton. Hazaribagh, so rich in metals, yields also galena, and in 1880 a company was formed in Bombay to work this district; but it came to nothing. Ajmere, Sirmur, and Tenasserim would also yield profitable results.

#### SOLVAY AND CO.'S ALKALI WORKS.

According to the Journal of the Dutch Society for the Promotion of Industry, the production of soda in the combined works of Messrs. Solvay & Co. has risen from 3 ewt. in 1864—when it was first commenced—to 400,000 tons in 1889. The latter figure represents about one-half of the world's consumption. The Société Solvay & Cie. owns, in addition to the immense works at Conillet and Vaugeoisville-Dombasle in France, three factories in Germany (Sarralbe, Wyhlen, and Bernburg), one in Russia (Beresniki), one in Austria (Ebensee), and one in the United States (Syracuse).

**SULPHURIC ACID AND MANGANATE OF SODA  
CONTRACTS.**

At its meeting on Tuesday the London County Council accepted the tender of Messrs. C. Tennant, Sons, & Co. for the supply of 1,000 tons of manganate of soda at 16*l.* per ton, and of the Widnes Alkali Company (Limited) for the supply of 600 tons at 15*l.* 15*s.* per ton. The Council also accepted the tender of Messrs. Sadler & Co. for the supply of 1,000 tons of sulphuric acid at 1*l.* 14*s.* per ton.

**DISCOVERY OF NICKEL ORE.**

A rich vein of nickel ore has been discovered at Frankenstein, in the Kosenmetz Hills. Herr Krupp, the ironfounder, has made an offer of one and a half million marks to the finders and owners of the ore, but they refuse to sell the whole vein. They have, however, undertaken to supply Herr Krupp with 2,000 cwt. of it.—*Ironmonger.*

**BOARD OF TRADE RETURNS**

**SUMMARY OF IMPORTS.**

	Month ended 30th November	
	1889.	1890.
	£	£
Metals.....	1,898,436	1,802,300
Chemicals and dyestuffs.....	492,902	458,182
Oils.....	664,123	601,428
Raw materials for non-textile industries.....	3,853,895	3,536,514
Total value of all imports ....	41,337,091	37,152,273

**SUMMARY OF EXPORTS.**

	Month ended 30th November	
	1889.	1890.
	£	£
Metals (other than machinery) ....	4,032,840	3,783,090
Chemicals and medicines .....	709,350	754,401
Miscellaneous articles.....	3,082,569	2,915,877
Total value of all exports.....	22,270,038	21,025,553

**IMPORTS OF OILS FOR MONTH ENDED 30TH NOVEMBER.**

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Cocoa-nut..... Cwt.	18,344	14,387	£ 22,383	£ 22,081
Olive .....	1,546	519	53,949	20,282
Palm .....	78,385	94,859	84,894	117,485
Petroleum .....	11,084,733	10,701,400	272,657	232,691
Seed .....	1,514	2,149	41,105	59,356
Train, &c..... Tuns	1,313	1,364	29,528	27,270
Turpentine .....	42,373	42,952	72,461	61,083
Other articles .. Value £	..	..	87,116	60,280
Total value of oils ...	..	..	664,123	601,428

**IMPORTS OF METALS FOR MONTH ENDED 30TH NOVEMBER.**

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Copper:—			£	£
Ore .....	9,395	10,513	73,294	89,034
Regulus .....	8,861	5,991	238,861	182,060
Unwrought ....	3,082	4,758	143,619	278,469
Iron and steel:—				
Iron ore .....	290,790	267,660	225,810	211,037
Iron bolt, bar, &c. ..	14,640	7,633	137,393	74,054
Steel, unwrought..	644	614	8,023	7,611
Lead, pig and sheet ..	13,555	11,383	175,046	158,613
Pyrites .....	37,915	52,956	66,241	97,501
Quicksilver..... Lb.	127,906	30,431	15,435	5,080
Tin .....	61,408	52,865	280,122	253,014
Zinc .....	5,272	5,925	111,858	139,639
Other articles ... Value £	..	..	122,131	305,188
Total value of metals	..	..	1,898,436	1,802,300

**IMPORTS OF RAW MATERIALS FOR NON-TEXTILE  
INDUSTRIES FOR MONTH ENDED 30TH NOVEMBER.**

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
Bark, Peruvian .. Cwt.	8,788	8,764	£ 28,506	£ 26,757
Bristles..... Lb.	241,718	292,455	34,651	50,060
Caoutchouc..... Cwt.	17,409	28,324	188,724	350,072
Gum:—				
Arabic.....	3,023	7,725	11,853	23,565
Lac, &c.....	8,176	9,755	31,398	44,862
Gutta-percha ....	2,320	5,477	21,652	66,389
Hides, raw:—				
Dry.....	29,482	38,659	77,953	91,420
Wet .....	55,874	41,787	127,835	95,956
Ivory.....	1,114	1,022	61,724	55,585
Manures:—				
Guano..... Tons	1,832	1,828	16,248	6,687
Bones.....	6,942	9,377	35,925	51,799
Paraffin..... Cwt.	36,486	46,854	41,068	59,674
Linen rags..... Tons	2,907	2,247	31,379	23,782
Esparto.....	15,635	18,344	74,169	90,378
Pulp of wood ....	8,544	13,655	53,571	70,132
Rosin..... Cwt.	114,600	92,369	24,221	22,356
Tallow and stearin ..	74,471	64,828	99,785	83,180
Tar .....	2,660	8,127	2,026	7,455
Wood:—				
Hewn .....	178,707	182,350	521,098	398,201
Sawn .....	453,980	430,876	1,175,095	940,621
Staves .....	11,952	13,899	55,206	51,726
Mahogany .... Tons	3,420	3,892	30,167	34,938
Other articles ... Value £	..	..	1,109,641	881,919
Total value ...	..	..	3,853,895	3,536,514

Besides the above, drugs to the value of 53,877*l.* were imported, as against 65,347*l.* in November 1889.

IMPORTS OF CHEMICALS AND DYE-STUFFS FOR MONTH  
ENDED 30TH NOVEMBER.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
			£	£
Alkali..... Cwt.	4,950	4,275	3,615	3,327
Bark (tanners. &c.) ..	20,832	22,764	6,804	6,481
Brimstone..... "	72,224	49,254	15,146	12,837
Chemicals..... Value £	..	..	147,303	119,476
Cochineal..... Cwt.	1,034	437	6,619	2,984
Cutch and gambier Tons	1,715	3,059	47,553	73,239
Dyes:—				
Aniline..... Value £	..	..	31,037	23,862
Alizarine..... "	..	..	13,985	20,942
Other..... "	..	..	950	727
Indigo..... Cwt.	1,137	957	13,227	14,602
Madder..... "	836	1,648	852	2,271
Nitrate of soda.... "	74,603	77,780	32,393	31,682
Nitrate of potash. "	25,437	22,210	23,305	20,677
Valonia..... Tons	939	1,028	14,846	19,008
Other articles... Value £	..	..	131,307	97,064
Total value of chemicals	..	..	492,902	458,182

EXPORTS OF METALS (OTHER THAN MACHINERY) FOR  
MONTH ENDED 30TH NOVEMBER.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
			£	£
Brass..... Cwt.	12,762	8,699	50,342	41,780
Copper:—				
Unwrought..... "	80,416	76,926	184,078	243,360
Wrought..... "	46,491	20,137	130,681	74,937
Mixed metal.... "	38,941	23,159	99,408	71,600
Hardware..... Value £	..	..	200,995	218,557
Implements..... "	..	..	107,627	110,817
Iron and steel.... Tons	375,199	299,477	2,843,218	2,519,518
Lead..... "	3,428	4,174	50,658	61,992
Plate-ware... Value £	..	..	46,354	39,688
Telegraph wires... "	..	..	113,306	246,378
Tin..... Cwt.	8,106	10,510	39,561	52,423
Zinc..... "	8,539	10,180	8,566	11,143
Other articles.. Value £	..	..	98,016	91,737
Total value.....	..	..	4,032,840	3,783,990

EXPORTS OF DRUGS AND CHEMICALS FOR MONTH ENDED  
30TH NOVEMBER.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
			£	£
Alkali..... Cwt.	594,722	641,393	156,795	225,517
Bleaching materials ..	131,834	161,858	48,122	47,680
Chemical manures. Tons	24,922	16,336	159,253	117,859
Medicines..... Value £	..	..	90,015	84,273
Other articles... "	..	..	255,135	279,072
Total value.....	..	..	769,350	754,401

EXPORTS OF MISCELLANEOUS ARTICLES FOR MONTH  
ENDED 30TH NOVEMBER.

Articles.	Quantities.		Values.	
	1889.	1890.	1889.	1890.
			£	£
Gunpowder..... Lb.	1,017,800	875,800	28,879	17,779
Military stores.. Value £	..	..	84,336	94,813
Candles..... Lb.	1,283,300	1,276,800	23,927	25,881
Caoutchouc..... Value £	..	..	99,741	102,627
Cement..... Tons	49,862	47,295	99,023	96,992
Products of coal Value £	..	..	133,956	113,719
Earthenware... "	..	..	195,890	177,318
Stoneware..... "	..	..	19,833	18,358
Glass:—				
Plate..... Sq. Ft.	388,212	221,735	21,239	12,668
Flint..... Cwt.	10,759	8,738	26,855	23,165
Bottles..... "	83,021	73,611	39,067	33,852
Other kinds.... "	24,231	15,262	18,078	12,990
Leather:—				
Unwrought.... "	15,209	12,456	127,633	115,902
Wrought..... Value £	..	..	33,952	30,815
Seed oil..... Tons	5,911	6,395	133,133	141,907
Floor cloth..... Sq. Yds.	1,326,300	1,120,600	63,630	53,483
Painters' materials Val. £	..	..	135,797	123,683
Paper..... Cwt.	92,389	78,440	164,045	132,326
Rags..... Tons	5,213	4,401	41,948	33,236
Soap..... Cwt.	11,951	39,328	47,270	46,205
Total value.....	..	..	3,082,569	2,915,877



## Monthly Patent List.

\* The dates given are the dates of the Official Journals in which acceptances of the Complete Specifications are advertised. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the said dates.

### I.—GENERAL PLANT, APPARATUS, AND MACHINERY.

#### APPLICATIONS.

18,596. E. J. Hardy. Improvements in the production of cold and ice, and in apparatus therefor. November 18.

18,751. O. Schulz. Improvements in continuous evaporating apparatus. Complete Specification. November 20.

19,071. O. Imray.—From A. Bergh, Denmark. A method and apparatus for subjecting liquids having substances suspended in them to centrifugal action, whereby they are brought into contact only with sterilised or with other air, with which they are to be saturated. November 24.

19,110. J. A. Morrell. Improved methods of and apparatus for evaporating and concentrating solutions. Complete Specification. November 25.

19,199. J. Kent. An improvement in thermometers. November 25.

19,550. J. Kirkaldy. Improvements in apparatus for heating and evaporating liquids. December 1.

19,587. F. Soxhlet. A method of closing bottles suitable for sterilised liquids. Complete Specification. December 1.

19,667. C. C. Leathers.—From W. Radam, United States. New or improved retort for the manufacture of patent medicines. Complete Specification. December 2.

19,736. A. Hart. Improvements in or relating to vessels and appliances for boiling, infusing, steaming, washing, and bleaching purposes. Complete Specification. December 3.

19,835. H. J. Allison.—From The National Salt and Chemical Co., United States. Improved apparatus for the vapourisation of liquids. Complete Specification. December 5.

19,979. J. Cox. An improved apparatus for storing and discharging liquid and compressed gases. December 8.

19,981. H. Cotton. Improvements in the construction of mills for grinding flint stone, bone, glaze, and other like substances. December 8.

20,095. H. Roeske. Improvements in apparatus for the filtration of liquids. Complete Specification. December 9.

20,133. S. Rushton, E. Blades, and W. Holden. An improved grinding or crushing mill for pulverising or reducing salt. December 10.

20,166. F. M. Robertson. Improvements in the process of and apparatus for evaporating and drying. December 10.

20,309. E. C. C. Baly. An improvement in mercury vacuum pumps. December 12.

20,318. E. Cohn. Improvements in and relating to sterilising apparatus. Complete Specification. December 13.

20,351. W. Dewar. An improved condenser. December 13.

#### COMPLETE SPECIFICATIONS ACCEPTED.\*

1889.

19,096. T. Williams. Means and appliances for production of pure carbonic oxide gas. December 3.

20,049. D. Herman. Apparatus for concentrating solutions and drying substances. December 10.

1890.

606. J. Brock and T. Minton. Filter presses. December 3.  
1242. R. A. Kemp. Apparatus for containing material for absorbing moisture. November 26.

1948. H. W. Deacon and V. C. Driffield. Apparatus for separating liquids from solids. December 10.

2055. B. J. B. Mills.—From A. Siegle. Apparatus for heating liquid and gaseous bodies. December 10.

2207. W. Scollay. Means for regulating admission of gas and air to Bunsen burners. December 17.

7886. T. Norman and H. T. Simpson. Improvements in furnaces to promote combustion. December 17.

8295. B. Versen. Production of refractory linings for converter bottoms, and apparatus therefor. December 10.

17,718. H. H. Lake.—From E. Bolton. Distilling and condensing apparatus. December 10.

### II.—FUEL, GAS, AND LIGHT.

#### APPLICATIONS.

18,488. R. E. Slade. Improvements in fire-lighters. November 17.

18,618. T. A. Greene and C. M. Walker. Improvements in gas-lighting. November 18.

18,639. P. S. Hyslop. Apparatus for utilising liquid fuel for the production of motive power for heat motors. November 18.

18,640. P. S. Hyslop. Apparatus for producing and storing under pressure combustion gases from solid fuel for use as motive power in heat motors. November 18.

18,681. E. J. Duff. Improvements in the manufacture of gas from oils and water vapour. November 19.

18,825. J. Staincliffe. Improved means for charging and discharging retorts employed in the manufacture of gas. November 21.

18,871. H. Williams, of The Illuminating, Heating, and Power Gas Company, Limited. Improvements in the method of and in apparatus for burning gas for heating and steam generating purposes. November 21.

18,872. H. Williams, of The Illuminating, Heating, and Power Gas Company, Limited. Improvements in gas generating apparatus. November 21.

18,949. J. Raines, jun., and G. Raines. An improved fire-lighter. November 22.

18,991. R. F. Strong. An improved smokeless fuel. November 22.

19,067. S. Clarke. Improvements in the manufacture of night-lights. November 24.

19,219. H. C. Bull. Improvements in and connected with the manufacture of combustible gases from coal and analogous substances, and in and connected with the production of sulphate of ammonia from the product of the process of manufacture. November 26.

19,501. F. J. Jones. Improvements in the manufacture of coke, in obtaining by-products therefrom, and in apparatus used therefor. November 29.

19,543. J. Wilson, jun. Improvements in the consumption of smoke. December 1.

19,553. V. B. Lewes and W. W. Staveley. Improvements in the manufacture or production of illuminating gases. December 1.

19,732. W. C. Owston. Improvements relating to the purification of smoke, and the collection of by-products therefrom. December 3.

19,761. T. Proud. An improvement in wooden grids for gas purifiers. December 4.

19,888. J. Love. Improvements in and relating to the manufacture of gas, and to apparatus therefor. December 5.

19,963. A. Vinaard. Producing and supplying gas for lighting and motive power, and apparatus therefor. December 6.

20,002. A. Dauber. A gas generator. Complete Specification. December 8.

20,030. L. Van Vestraut. Improvements in apparatus for charging inclined gas retorts. December 9.

20,033. T. S. Forster. The economising of fuel. December 9.

20,658. J. Turner. An improved method of conversion or treatment of small coal, smudge, shale, and other organic matter and refuse, and the apparatus therefor. December 9.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1890.

1349. W. L. Wise.—From E. Solvay and L. Semet. Gasification of fuels and apparatus therefor. December 3.

9956. R. Kennedy. Heating by electricity. December 3.

17,000. J. J. Thomas. Coal-gas generating apparatus. December 3.

17,151. B. F. Field. An artificial fuel and the method of manufacturing the same. December 3.

17,466. H. H. Lake.—From The Standard Coal and Fuel Co. A compound for treating fuel. December 10.

17,470. H. H. Lake.—From The Standard Coal and Fuel Co. A compound for treating fuel. December 10.

17,484. H. H. Lake.—From The Standard Coal and Fuel Co. A compound for treating fuel. December 10.

#### III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

##### APPLICATIONS.

19,502. A. M. Clark.—From The Gewerkschaft Messel, Germany. Production from mineral oils of sulphonic acids and sulphones, and the manufacture of a new product for treating gelatinous matters with sulphonic acid. November 29.

19,943. W. W. Staveley. Improvements in the production of hydrocarbons of the aromatic series from blast-furnace, coke-oven, or coal-tar oils, or from crude phenols or phenoloid oils or compounds (such as are obtained from these oils). December 6.

#### IV.—COLOURING MATTERS AND DYES.

##### APPLICATIONS.

18,526. J. Imray.—From La Société L. D. Huguenin and Cie., Switzerland. Manufacture of colouring matters obtained by the action of amines of the fatty series upon galloxy-anine. Complete Specification. November 17.

18,527. O. Imray.—From the Firm J. Curtius, Germany. Process and apparatus for the manufacture of ultramarine. November 17.

18,623. H. H. Lake.—From Wirth and Co., agents for A. Leonhardt and Co., Germany. Improvements in the manufacture of colouring matters. November 18.

18,637. B. Willecox.—From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture of new anthraquinone dyestuffs. November 18.

18,729. B. Willecox.—From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture of new derivatives of anthraquinone. November 19.

19,016. A. E. Delacroix. The improvement of lactic acid and its salts in the application of colouring matters. November 24.

19,065. S. Pitt.—From L. Cassella and Co., Germany. The production of sulphonated thionines, and dyestuffs therefrom. November 24.

19,167. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik, Germany. Improvements in the manufacture of sulpho-acids of a red basic naphthalene dyestuff. November 25.

19,330. S. Pitt.—From L. Cassella and Co., Germany. The preparation of colouring matters with gamma-amido-naphtholsulphonic acid. November 27.

20,252. H. H. Lake.—From Wirth and Co., agents for A. Leonhardt and Co., Germany. Improvements in the manufacture of chemical compounds. December 10.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1890.

569. A. Kern and E. Sandoz. Blue colouring matters. December 17.

1579. J. Imray.—From La Société Anonyme des Matières Colorantes et Produits Chimiques de St. Denis, A. F. Poirrier and D. A. Rosenstiehl. Manufacture of azo-amines by reduction of azo-colouring matters derived from nitranines. December 3.

1699. O. Imray.—From The Farbwerte vormals Meister, Lucius, and Brünig. Manufacture of colouring matter of the induline series, soluble in water. December 8.

1808. O. Imray.—From The Society of Chemical Industry, Basle. Production of yellow basic colouring matter. November 26.

1811. R. Holliday. Manufacture of colouring matters. December 10.

1812. R. Holliday. Manufacture of colouring matters, and dyeing or printing fibres therewith. December 10.

1874. J. Imray.—From La Société Anonyme des Matières Colorantes et Produits Chimiques de St. Denis, and Dr. Chapuis. Production of soluble blue colouring matters of the induline class. December 17.

2499. H. H. Lake.—From K. Oehler. Manufacture of soluble blue cotton dyestuff. December 3.

#### V.—TEXTILES, COTTON, WOOL, SILK, Etc.

##### APPLICATIONS.

18,585. J. Davidson. Improvements in the use or application of thistle down. November 18.

18,955. J. Palmer. Improvement in the treatment of resinous or gummy fibres. November 22.

19,252. C. D. Abel.—From the Firm of Philips and Mathée, Germany. Process for cleansing or purifying woollen fabrics. November 26.

20,257. A. H. Briggs. An improved method of and apparatus for scouring, drying, and stretching mohair, alpaca, and such like wools and yarns. December 12.

20,267. A. Ambler, S. Ambler, and F. Ambler. Improvements in cleansing, treating, or washing wool and like animal fibres, and in apparatus employed therein. December 12.

20,334. D. Barnett. Improvements in apparatus for treating textile vegetable substances to obtain fibres therefrom. December 13.

## COMPLETE SPECIFICATION ACCEPTED.

1890.

1461. J. Smith, I. Smith, and J. Smith. Method and apparatus for scouring wool and other fibres. November 26.

# VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

## APPLICATIONS.

18,700. J. B. Whiteley, E. Whiteley, A. Armitage, and W. Cleland. Improvements in the method of and apparatus for dyeing wool, cotton and other fibre, and also "tops" and slivers of fibre. November 19.

19,158. P. J. Grandsire. Improvements in machines for dyeing skeins or hanks. November 25.

20,274. E. Knecht. An improved method of mordanting mixed goods of cotton and wool. December 12.

## COMPLETE SPECIFICATIONS ACCEPTED.

1890.

1062. S. Knowles and J. Knowles. Production of azo colours upon cotton or other vegetable fibres in the piece, and apparatus therefor. November 26.

1607. J. Smith, I. Smith, and J. Smith. Method and apparatus for mordanting and dyeing wool, cotton, and other fibre, and for separating animal from vegetable fibre. December 17.

1812. R. Holliday. See Class IV.

1813. R. Holliday. Dyeing textile fibres. December 10.

2411. T. Ingham. Dyeing cotton. December 17.

17,872. T. Salzmänn. Production of dyed cotton yarns and threads. December 10.

# VII.—ACIDS, ALKALIS, AND SALTS.

## APPLICATIONS.

18,835. J. Simpson. Improvements in the manufacture of caustic soda, with the recovery of reagents employed for repeated use. November 21.

18,990. J. Greenwood. Improvements in the manufacture or production of chlorine and caustic soda, and in apparatus therefor. November 22.

19,089. G. E. Davis and A. R. Davis. Improvements in apparatus for the manufacture of sulphate of ammonia. November 25.

19,121. T. Needham. Improvements in the packing or putting up of chloride of lime and other substances. Complete Specification. November 25.

19,316. J. C. Richardson. Improvements in chloridising lime. November 27.

19,390. M. N. d'Andria. Improvements in the manufacture of ammonium chloride and calcium sulphate. November 28.

19,776. J. Simpson. Improvements in the manufacture of chlorine. December 4.

19,792. M. Netto. Improvements relating to the production of ferrate of sodium, and to the manufacture of caustic soda therefrom. December 4.

19,815. F. M. Lyte and O. J. Steinhart. Improvements in the production of chlorine. December 4.

20,012. F. Ellershausen. Improvements in the treatment of soda waste. December 8.

## COMPLETE SPECIFICATIONS ACCEPTED.

1890.

448. F. M. Spence, D. D. Spence, and A. Esilman. Manufacture of alum. December 10.

984. H. Grimshaw. Compound for generation of carbon dioxide. December 10.

1016. F. Ellershausen. Manufacture of soda and potash. December 10.

1524. G. Wischm. Method of vaporising solutions containing nitrates of manganese, and means for decomposing the said nitrates. December 10.

5058. E. Delplace and J. Delplace. Manufacture of sulphuric acid. December 10.

6758. J. McEwen. Process and apparatus for continuous generation of carbonic acid gas from a soluble carbonate and an acid in solution. December 17.

15,648. J. Leith. Production of sulphhydrate of calcium to be used in the manufacture of alkali and sulphuretted hydrogen, and apparatus therefor. December 3.

# VIII.—GLASS, POTTERY, AND EARTHENWARE.

## APPLICATIONS.

18,940. J. Hartley. Improvements in apparatus for annealing rolled plate glass. November 22.

19,340. H. R. Lumley. Improvements in the manufacture of sheet or plate glass and other glass-ware. November 27.

19,342. W. Ambler. An improved method and means to be used in the manufacture of glass bottles. November 27.

19,532. D. Rylands. Improvements in the manufacture of hollow glass and other ware, and of the caps for such ware. December 1.

19,594. G. Bosselier. Improved methods of preparing "Mosaic" and other slabs, tiles, medallions, and like articles. December 1.

19,717. J. P. Bayley.—From C. Ulfig, United States. Improved method of joining parts of glass-ware. December 3.

19,798. A. T. Wedelin. Improvements in furnaces and implements for annealing glass. December 4.

20,207. C. F. Bailey. Improvements in or relating to saggers used in firing pottery-ware. December 10.

20,240. F. E. Grant. A process relating to the application of coloured designs to vitreous surfaces, and machinery therefor. December 10.

## COMPLETE SPECIFICATIONS ACCEPTED.

1890.

1803. L. Appert. Moulding glass, and apparatus therefor. December 17.

2626. C. Huelser.—From J. Kemper. Producing opalescent glass or enamel from a glass mixture containing silico-fluorides of alkalis or compounds thereof. December 17.

13,436. F. C. Roberts. Pottery kilns. December 17.

14,983. J. T. King.—From E. Jones and J. A. Jones. Apparatus for manufacture of glass-ware. November 26.

16,112. W. Boulton. Machinery for manufacturing cups and other articles in pottery-ware. November 26.

17,952. A. J. Boulton.—From H. Thumler. Manufacture of glass chains. December 17.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

### APPLICATIONS.

18,518. J. Wilson. An improved plaster called "Terra-Tinta." November 17.

18,603. T. Arnold. Improvements in or relating to the manufacture of cement, and the means employed therefor. November 18.

18,732. A. Cajot. Improvements in apparatus for the enriching of phosphate of lime, and of phosphated chalks for the preparation of cements, marls, and starches, and for similar purposes. Complete Specification. November 19.

18,774. J. T. Welch and W. J. Owen. Improvements in the utilisation of granite and other kindred rocks in the manufacture of bricks, tiles, and other articles. November 20.

18,899. L. C. Delahaye. An improved method of enriching phosphated chalk and other calcareous phosphates. November 21.

18,924. A. F. E. de St. Dalmas. Improvements in and relating to paving blocks. Complete Specification. November 21.

18,992. W. Smartt. Improvements in the process of drying or burning bricks, pipes, tiles, and other goods in clamps, kilns, and other works, and in apparatus for effecting the same. November 22.

### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

20,303. C. Smith.—From G. P. V. Nielsen. Artificial asphalt. December 17.

1890.

2371 and 2371A. C. H. Edwards. Manufacture of certain bricks and fireclay substitutes. December 10.

7107. J. P. Bayly.—From P. Arnold. Paving blocks. December 10.

## X.—METALLURGY, MINING, ETC.

### APPLICATIONS.

18,508. J. Johnson.—From J. W. Langley, United States. Improvements in aluminium alloys, and in the manufacture thereof. November 17.

18,546. F. H. Molesworth. Improvements in the construction of rotary furnaces for the calcination of sulphide and other ores. Complete Specification. November 17.

18,604. L. L. Burdon. Improvements in compound gold wire. Complete Specification. November 18.

18,605. L. L. Burdon. Improvements in compound hollow metal ingots and wire. November 18.

18,636. R. Hadden.—From G. Conkling, United States. A new or improved process for concentrating iron ore. Complete Specification. November 18.

18,673. A. J. Spiller. Improvements in bronzing brass and other alloys. November 18.

18,718. D. Jenkins. Improvements in machinery or apparatus to be employed in coating metal sheets or plates with tin or other metal or alloy. November 19.

18,726. M. R. Garcia. Improvements in the method and process for the recovery of tin from tinned plate and other sources. Complete Specification. November 19.

18,869. A. Gutensohn. An improved method of and process for annealing metal plates and sheets, and apparatus for use in the same, and for other purposes. November 21.

18,898. T. D. Nicholls and The Cape Copper Company, Limited. An improved method of extracting copper from ores or compounds containing the same. November 21.

18,900. A. Coehn. An improved method of producing castings of aluminium or its alloys. November 21.

18,915. W. P. Thompson.—From C. H. Land, United States. Improvements in or appertaining to the manufacture of compound aluminium plates. Complete Specification. November 21.

18,932. R. Starke and W. Paterson. Improvements in the extraction of metals from ore. November 22.

18,933. R. Starke and W. Paterson. Improvements in the extraction of metals from ore. November 22.

18,934. W. Paterson and R. Starke. Improvements in the extraction of metals from ore. November 22.

19,163. H. W. Lash and J. Johnson. Improvements in the reduction of ores. Complete Specification. November 25.

19,164. J. Johnson. Improvements in the reduction of ores. Complete Specification. November 25.

19,179. J. C. Bayles. Improvements in the art of uniting iron or steel plates or sheets by welded joints. Complete Specification. November 25.

19,180. J. C. Bayles. Improvements in the method of and apparatus for welding together the meeting edges of iron or steel sheets or plates. Complete Specification. November 25.

19,181. J. B. Hannay. An improved process and apparatus for extracting gold from minerals containing it. November 25.

19,572. W. P. Hopkins. Improvements in manufacturing iron. Complete Specification. December 1.

19,646. T. Smith and J. B. Hamond. Improvements in the process and in apparatus for manufacturing metallic cement. December 2.

19,707. L. Joret. Improvements in means for stopping tapping holes of cupolas and blast furnaces, especially those used for "ferromanganese" and "spiegel." December 3.

19,734. J. Y. Johnson.—From A. Imbert and G. Juillien, France. Improvements in the manufacture of iron and other metals, and apparatus therefor. December 3.

19,756. G. W. Hart. Reducing haloid mineral ores, and obtaining metals, oxides, and carbonates therefrom. December 4.

19,875. R. Nahusen. Improvements in the extraction of gold, silver, copper, and other metals from ores or substances containing the same. December 5.

19,910. J. Heydon. Improvements in the means and apparatus for making the moulds in which metals are to be cast. December 6.

19,940. W. D. Bohm. Improvements in the extraction of gold and silver. December 6.

20,160. T. C. Sanderson. Improvements in smelting ores of antimony, and in furnaces and crucibles therefor. December 10.

20,393. H. C. S. Dyer. Improvements in the manufacture of steel. December 13.

### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

19,687. T. Teague. Treatment of tin refuse or slag. December 3.

20,181. T. Young. Treating ores to obtain precious metals, and apparatus therefor. December 10.

20,264. A. Gutensohn. Annealing metal plates and sheets. November 26.

20,380. J. E. Bott. Manufacture of compound armour plates. December 17.

20,587. J. Lysaght. Galvanising apparatus. November 26.

1890.

213. J. Mackintire. Manufacture of steel and iron. December 10.
316. J. E. Chaster. Extraction of metals from ore. December 10.
560. T. Twynam. Manufacture of copper. December 10.
859. W. White. Production of aluminium. November 26.
1053. H. Grimshaw. Recovery of zinc from galvanised iron and steel. December 3.
1443. J. T. King.—From C. Adams. Reduction of metallic ores, and apparatus therefor. December 3.
1522. T. H. Johns. Apparatus for coating hoop iron with tin or terne metal. December 17.
1624. A. H. Holdich. Stopper or valve for controlling the flow of steel from the ladle or reservoir used in steel works. December 3.
2390. J. Buchanan, jun. Precipitating gold or silver in the process of extraction. December 10.
5407. O. Lumaghi. Apparatus for reduction of argenteriferous or other zinc ores. November 26.
5847. C. Wood. Process and apparatus for manufacturing fibrous material from slag or other vitreous substances. November 26.
8295. B. Versen. *See Class I.*
8572. T. L. Thomas and J. B. Hillman. Process and appliances for galvanising metals.
- 14,053. F. A. Herbertz. Smelting furnaces. December 17.
- 16,060. L. Grabau. Obtaining metallic sodium. December 3.
- 17,613. P. M. Justice.—From J. Meyer. Manufacture of steel and ingot iron. December 10.

## XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

### APPLICATIONS.

- 18,491. R. Kennedy. Improvements in dynamo electric machines. Complete Specification. November 17.
- 18,523. H. Zerener. Improvements in apparatus for heating, welding, melting or gasifying solid bodies by electricity. November 17.
- 18,625. H. H. Lake.—From H. Lemp, United States. Improvements in and relating to apparatus for use in electric welding, forging, and other metal-working operations. Complete Specification. November 18.
- 18,631. H. H. Lake.—From E. Thomson, United States. Improvements relating to the welding of metals by electricity. Complete Specification. November 18.
- 18,661. J. T. Niblett. Improvements in the construction of plates for secondary batteries. November 18.
- 18,896. A. S. Elmore. Improvements in the process and apparatus for manufacturing copper tubes, sheets, strips, and wires by electrolysis. Complete Specification. November 21.
- 19,052. M. N. Mallison. Improved combination of ingredients constituting a compound as an illuminating medium for electric incandescent lamps. November 24.
- 19,155. H. H. Lake.—From G. E. Hatch, United States. Improvements in electrical storage batteries or accumulators. Complete Specification. November 25.
- 19,162. M. W. Dewey. Improvements in methods of electric welding and metal working. Complete Specification. November 25.
- 19,203. L. Hausmann. Improvements in the manufacture of thin metal tubes by electrolysis. November 25.
- 19,344. E. Placet and J. Bonnet. Improvements in electrolysis in general, and particularly in electrolysis of metals. November 27.

19,423. E. A. Ducretet. Improvements in apparatus for the electrolytic production of gases. November 28.

19,650. Siemens Bros. and Co., Lim.—From Siemens and Halske, Germany. Improvements in the distribution of electricity through accumulator batteries. December 2.

19,942. W. P. Thompson.—From F. Marse, Germany. Improvements in galvanic batteries, and in the electro-chemical formation of chlorine and chlorine-compounds applicable for such batteries and other purposes. Complete Specification. December 6.

20,102. A. S. Baxendale. An improvement in dynamo-electric and electro-dynamic machines. December 9.

20,111. H. H. Lake.—From L. L. Roberts and T. H. McGraw, United States. Improvements in apparatus for use in the electrolytic decomposition of metallic salts. Complete Specification. December 9.

20,126. C. E. Dutton, jun. Improvements in galvanic batteries. Complete Specification. December 10.

20,193. The Electrical Inventions Co., Lim., and E. Andreoli. Improvements in or relating to the electro-chemical extraction of gold, silver, and other precious metals from their ores. Complete Specification. December 10.

20,401. E. Andreoli. Improvements in the electrolysis of chloride and other solutions. Complete Specification. December 13.

### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

19,813. H. Wernsdley and H. Foster. Heating, melting, and working metals, alloys, and metalloids, by electricity; and apparatus therefor. November 26.

1890.

488. M. Sappey. Primary batteries. December 17.
8933. G. H. Felt. Method and apparatus for depositing metals electrically. December 10.
9956. R. Kennedy. *See Class H.*
- 16,522. G. D. Burton. Method and apparatus for working metals by electricity. November 26.
- 16,571. C. W. Kennedy and H. Groswith. Secondary batteries. November 26.

## XII.—FATS, OILS, AND SOAP MANUFACTURE.

### APPLICATIONS.

- 18,801. C. Billing. Improvements in or appertaining to the manufacture of soap. November 20.
- 19,066. C. F. Leake, H. W. Godfrey, and C. E. Lucas. Improved process for oxidising linseed and other oils. November 24.
- 19,070. O. Inray.—From the Firm of C. Spiegel, Russia. Process and apparatus for the production of castor oil. November 24.
- 19,382. J. Taylor. Improvements in the treatment of soapmakers' waste lyes for the recovery of glycerin and other substances. November 28.
- 19,960. P. Wild. Process and apparatus for smelting margarine, tallow, or other crude fatty substances by means of hot air. Complete Specification. December 6.
- 20,164. D. Clarke. Clarke's improved method for manufacturing chemical detergents. December 10.

### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

- 13,700. C. Billing. Manufacture of soap. December 3.

1890.

1109. H. Noerdlinger. Obtaining valuable products from press and extraction residues of oil manufacture. December 3.

1652. R. D. Barnett. Extracting oil and grease from cotton waste, &c. December 10.

2446. A. and E. des Cressonnières. Manufacture of kneaded or agglomerated soaps. December 3.

### XIII.—PAINTS, PIGMENTS, VARNISHES, AND RESINS.

#### APPLICATIONS.

18,609. J. Bond. Improvements in driers. Complete Specification. November 18.

19,072. O. C. Strecker. Improvements in the manufacture of sealing wax, blotting wax, and the like. November 24.

19,160. M. E. Dejonge. Improved anti-corrosive and anti-fouling compounds. Complete Specification. November 25.

19,275. R. C. Anderson. An improved manufacture of paints or varnish for coating leather and cloth. November 26.

19,323. W. Smith and W. Elmore. Improvements in the manufacture of basic carbonate of lead or white lead. November 27.

19,730. H. Meier. An improved compound applicable as paint, paste, or the like. December 3.

19,784. W. Smith and W. Elmore. Improvements in the manufacture of basic carbonate of lead or white lead. December 4.

20,145. A. F. St. George. An improved method of treatment of gum-resins and resins, and manufacturing varnishes. December 10.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1889.

19,910. J. E. Wood. Manufacture of white pigments, chiefly composed of sulphate of lead. November 26.

20,707. R. Mullard. Manufacture of paints and varnishes; treatment of gums and resins for same, and for preservative and decorative purposes; also for preserving plates and bottoms of ships from corrosion and fouling. December 3.

1890.

764. W. H. Barnes. Compositions to be used as paints. November 26.

13,719. E. Schmal. Medium for use with chemical and earth colours. December 3.

15,857. C. M. Higgins. Printing or stamping inks. December 3.

15,858. C. M. Higgins. Printing or stamping inks. December 3.

### XIV.—TANNING, LEATHER, GLUE, AND SIZE.

#### APPLICATIONS.

18,602. J. Broadhurst. An improved compound or size for fixing colours upon wall papers and the like. November 18.

19,216. C. L. Lawrence. Making leather boards, leather board soles, pulp board and soles, upper leather and sole leather, and soles waterproof. November 26.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1890.

981. H. Grimshaw. Recovery of tannin from waste leather. November 26.

17,853. T. O. Butler. A glue compound. December 10.

### XV.—AGRICULTURE AND MANURES.

#### APPLICATION.

20,310. R. Stone. Improvements in the manufacture of artificial manure. December 12.

#### COMPLETE SPECIFICATION ACCEPTED.

1890.

2265. J. Pipe and H. D. Andross. Compound for treating vines and plants for prevention and destruction of phylloxera, mildew, &c., and method of producing same. December 17.

### XVI.—SUGARS, STARCHES, GUMS, ETC.

#### APPLICATION.

20,181. H. Maenillan. Improvements in apparatus for evaporating, concentrating, or distilling saccharine liquids or such like. December 10.

### XVII.—BREWING, WINES, SPIRITS, ETC.

#### APPLICATIONS.

18,540. J. C. Fell.—From F. Tiedemann, Denmark. An improved apparatus for the continuous charging of liquids with carbonic acid gas. November 17.

18,652. A. J. Boulton.—From H. Lissagaray, France. Improvements in or relating to the manufacture of wine-spirit. November 18.

18,708. J. Cox. Improvements in brewing beer. November 19.

18,762. A. Blake. An improvement relating to fermentation of brewers' wort. November 20.

18,804. A. E. Favre and G. Savourat. Improvements in the treatment or application of Indian corn or maize for brewing purposes. November 20.

20,073. R. Genge and Sigmund Bros. Improvements relating to the production of dry yeast, cattle food, and spirit from vegetable products containing starch. Complete Specification. December 9.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1890.

1809. H. T. Brown, G. H. Morris, and E. R. Moritz. Manufacture of amyloins for the treatment of beer. December 10.

1996. J. F. Littleton. Method and apparatus for rousing beer and mixing or blending wort or gyle. December 10.

16,951. H. Bates, jun. Corn product for brewers' use, and manufacture thereof. December 3.

# XVIII.—CHEMISTRY OF FOODS, SANITARY CHEMISTRY, AND DISINFECTANTS.

## APPLICATIONS.

### A.—Chemistry of Foods.

18,627. J. W. Gordon.—From A. E. Wright, Australia. Improvements in the preparation of milk for dietetic purposes. November 18.

18,745. W. Cole. Improvements in the manufacture or production of butter. November 19.

19,168. E. Luck, R. Pott, and N. Pott. Improvements in the treatment of cereals for preventing or arresting decomposition, and for preserving or improving the condition thereof. November 25.

19,178. O. Imray.—From The Mosquera Julia Food Co., United States. Improvements in the manufacture of psin and peptonised food products. Complete Specification. November 25.

### B.—Sanitary Chemistry.

18,983. W. E. Adney. Improvements in the purification of sewage and other waste liquors, and in the recovery of ammonia therefrom. November 22.

19,628. R. S. Browldow. Improvements in apparatus for the treatment of sewage, parts of which are applicable to apparatus for purifying water for manufacturing and other purposes. December.

19,764. W. H. Mellor. Improvements in destructors or furnaces for burning towns' refuse. December 4.

19,967. R. Cunliffe and E. Vow. Improvements in the method of and apparatus for treating towns' slaughter-house, and other refuse or substance or material. December 8.

## COMPLETE SPECIFICATION ACCEPTED.

### A.—Chemistry of Foods.

1889.

19,882. H. Barotte. Process and apparatus for production of dry extract of coffee. December.

1890.

192. J. H. Hooker. An alimentary liquid. December 3.

982. H. Grimshaw. Producing infusions, &c., free from tannin. November 26.

2060. H. A. Snelling. Treating hops, blending same for infusion together with tea, cocoa, &c. December 10.

16,362. G. F. Redfern.—From E. Scott and A. Scheehong. Preservation of bread, &c., apparatus therefor. December 10.

17,245. B. Willeox.—From the Rumford Works. Baking powders, &c. December 3.

17,427. B. G. Hudnut. Corn products, &c., for manufacturing the same. December 17.

### B.—Sanitary Chemistry.

1890.

1170. C. A. Brown. Furnaces for burning refuse material. November 26.

16,972. F. P. Candy. Method and material of sewage and polluted water or liquids. December.

17,686. E. W. Cracknell. Furnace for garbage and refuse. December 10.

17,687. E. W. Cracknell. Process and apparatus for treatment of night-soil and noxious matters.

## C.—Disinfectants.

1890.

1589. C. T. Kingzett. Manufacture of disinfectants, insecticides, and sanitary reagents. December 3.

# XIX.—PAPER, PASTEBOARD, ETC.

## APPLICATIONS.

18,838. A. A. Haigh. Improved waterproof paper. November 21.

19,523. F. L. Leech and A. Horrobin. Improvements in means for waterproofing paper in the process of manufacture. December 1.

19,545. J. W. Bretherick. Improvements in machines or appliances used in the manufacture of paper. December 1.

## COMPLETE SPECIFICATIONS ACCEPTED.

1890.

1087. T. Goodall. Apparatus for incineration of black liquor and spent soda lyes used in the boiling of esparto grass and other fibres. November 26.

11,021. J. B. Atherton.—From L. D. Beck. Manufacture of Manila paper. December 10.

# XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES, AND EXTRACTS.

## APPLICATIONS.

19,074. J. Y. Johnson.—From F. von Heyden, Nachfolger, Germany. Improvements in the manufacture and production of medicinal compounds for ethereal oils. November 24.

18,576. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. Manufacture of a compound of antipyrine and iodine. December 1.

20,252. H. H. Lake.—From Wirth and Co., agents for A. Leonhardt and Co., Germany. See Class IV.

20,261. J. Bertram. Process or processes for obtaining the two isomeric monomethylethers of protocatechuic aldehyde. December 12.

# XXI.—PHOTOGRAPHIC PROCESSES AND MATERIALS.

## APPLICATIONS.

19,402. A. W. Scott. Improvements in the method of and apparatus for producing photographs, and in displaying them or the like. November 28.

19,658. A. J. Boulton.—From G. Eastmann, United States. Improvements in photographic films. Complete Specification. December 2.

## COMPLETE SPECIFICATION ACCEPTED.

1890.

16,238. H. H. Beale. A scarlet liquid for the treatment of negatives, by means of which clouds can be made, and other useful and artistic effects produced. December 17.



## XXII.—EXPLOSIVES, MATCHES, Etc.

## APPLICATIONS.

18,519. J. S. Walter. Improvements relating to explosive compounds. Complete Specification. November 17.

19,076. W. M. Mordey. Improvements in electric safety fuses. November 24.

19,125. W. M. Nix. Improvements in the manufacture of friction matches. Complete Specification. Filed November 25. Date applied for April 10, 1890, being date of application in United States.

19,259. G. G. André. Improvements in the manufacture of gunpowder, and in apparatus therefor. November 26.

## COMPLETE SPECIFICATIONS ACCEPTED.

1889.

20,286. E. W. Anderson. Apparatus for cutting smokeless explosives or plastic material into lengths. December 17.

1890.

1995. W. P. Thompson.—From J. Hess. Improvements in lucifer matches. November 26.

2547. J. Y. Johnson.—From C. Pieper. Manufacture of explosives. December 3.









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